Standard Operating Procedure – Hot Stage Setup, Panalytical Empyrean Diffractometer

X-ray Crystallography Laboratory

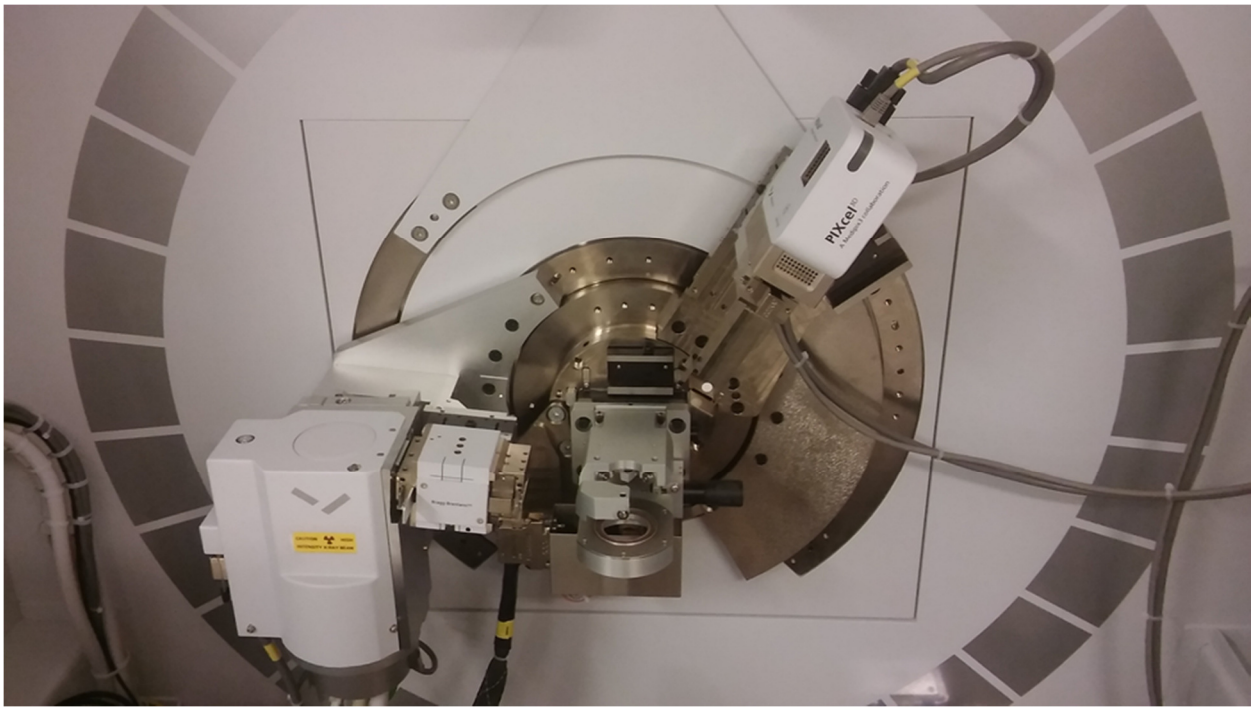
Purdue Department of Chemistry

Measurements above ambient temperature require the use of the Aton Parr TCU 1200 N hot stage for sample mounting. This SOP describes how to change the stage to the hot stage if it is not already installed, and how to collect basic data above ambient temperature.

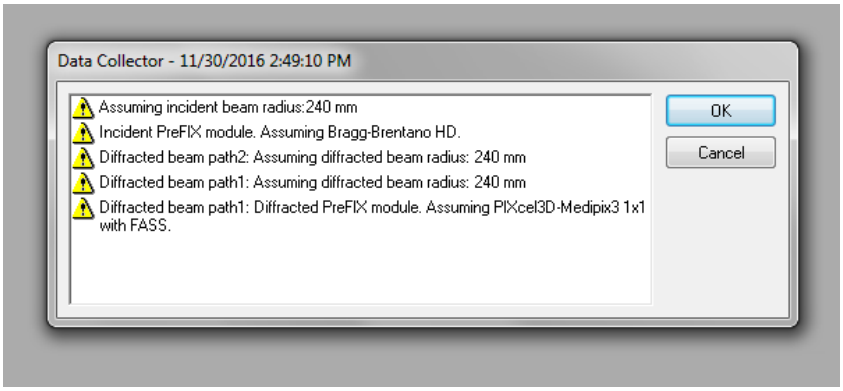
The stage is to be changed by fully trained users only! Please contact Dr. Zeller (zeller4@purdue.edu) for user training if you would like to collect data that involve using the hot stage.

Before getting started, inspect the instrument and check which stage and optics are mounted, and what slits and masks are used.

The image below shows the inside of the Empyrean enclosure with the reflection transmission spinner as the stage, the Bragg Brentano High Definition (BBHD) on the incident optics side (left), and the PIXel Medipix 3D detector with the "Fixed Anti Scatter Slit" (FASS) on the diffracted beam side (right). This is the most likely setup that you will encounter.

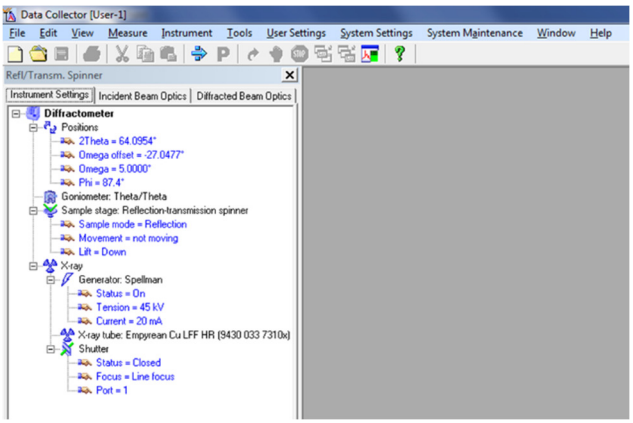


On the Empyrean control PC, check if the "Data Collector" software is running. Start it if necessary. Login in is "user-1", password is "galaxy". Once Data Collector is running, go to "Instrument", then "Connect". Wait for the software to finish establishing communication with the instrument. When prompted, select the stage that is installed. You will be prompted to confirm "assumed" hardware settings (settings the computer does not control electronically).



Carefully check that the assumed settings are correct (ass setups on the instrument use beam path 1 and a 240 mm radius). Click “ok”, regardless if the assumed settings are correct, but if any are not, make a note to change the settings in the next steps, below.

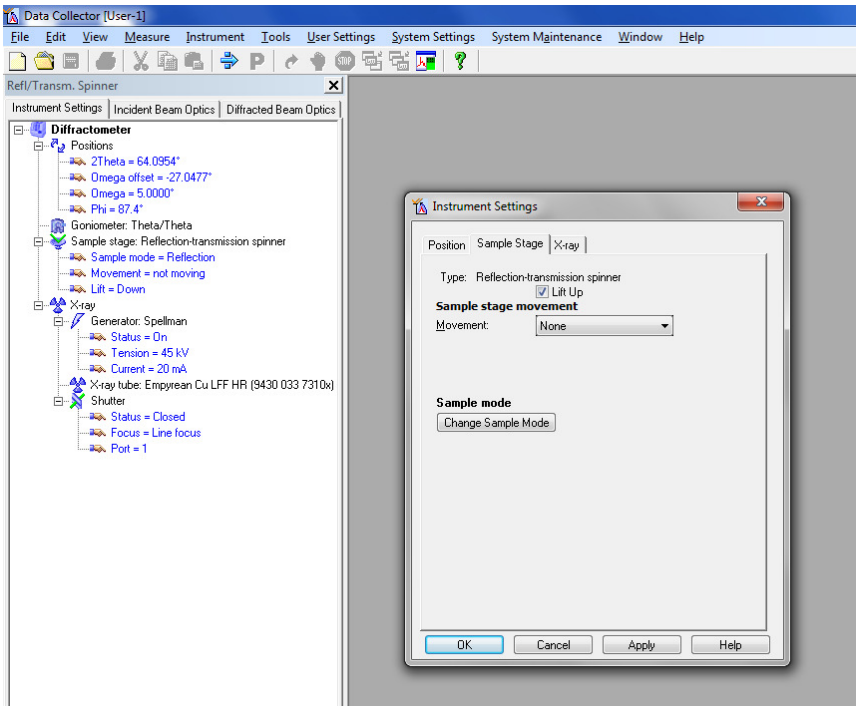
The image below shows the typical view of Data Collector after connection is established. Toggle between the tabs for “Instrument Settings”, “Incident Beam Optics” and “Diffracted Beam Optics” to check that the physically present stage, optics, slits and masks are identical to what is listed in Data Collector.



The hot stage is usually used in combination with the BBHD and with the PIXel Medipix 3D detector mounted on the “Fixed Anti Scatter Slit” (FASS). It can also be used in combination with the “Parallel Plate Collimator” (PCC). The following procedure describes how to change the configuration to the BBHD and PIXel Medipix with FASS. If you would like to use the PCC instead, please refer to the section of the “SOP Thin Films” that describes how to mount the PCC, or contact Dr. Zeller (zeller4@purdue.edu).

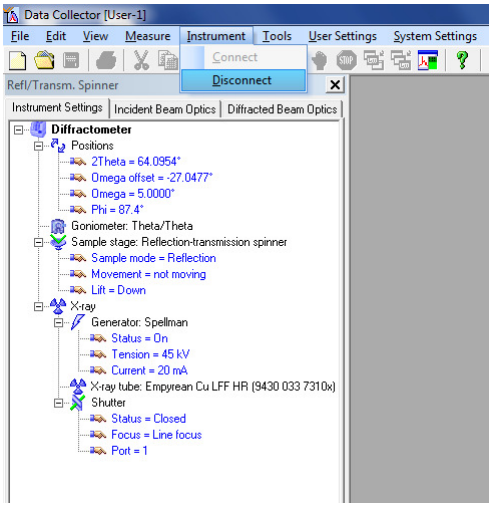
Changing the stage:

Check if a sample is present in the Reflection Transmission Spinner. If present, lower the stage: Double click on any of the “Generator” lines in the “Instruments Settings” tab. In the pop-up window uncheck “Lift up”, click “Apply”. Remove the sample.

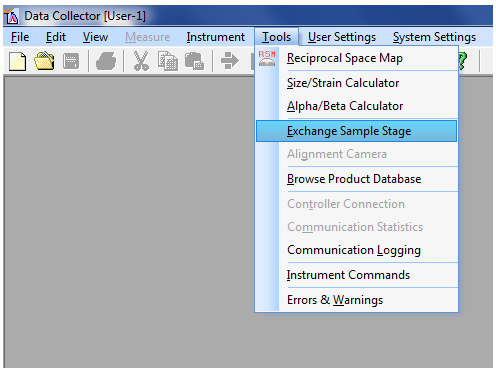


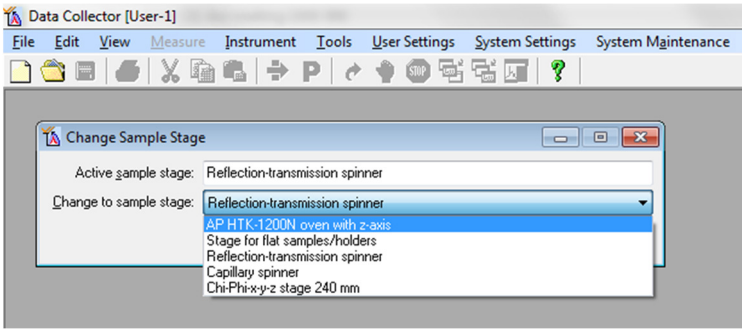
Check “Lift up”, Click “OK” to move the lift up again prior to changing the stage and close the pop-up window.

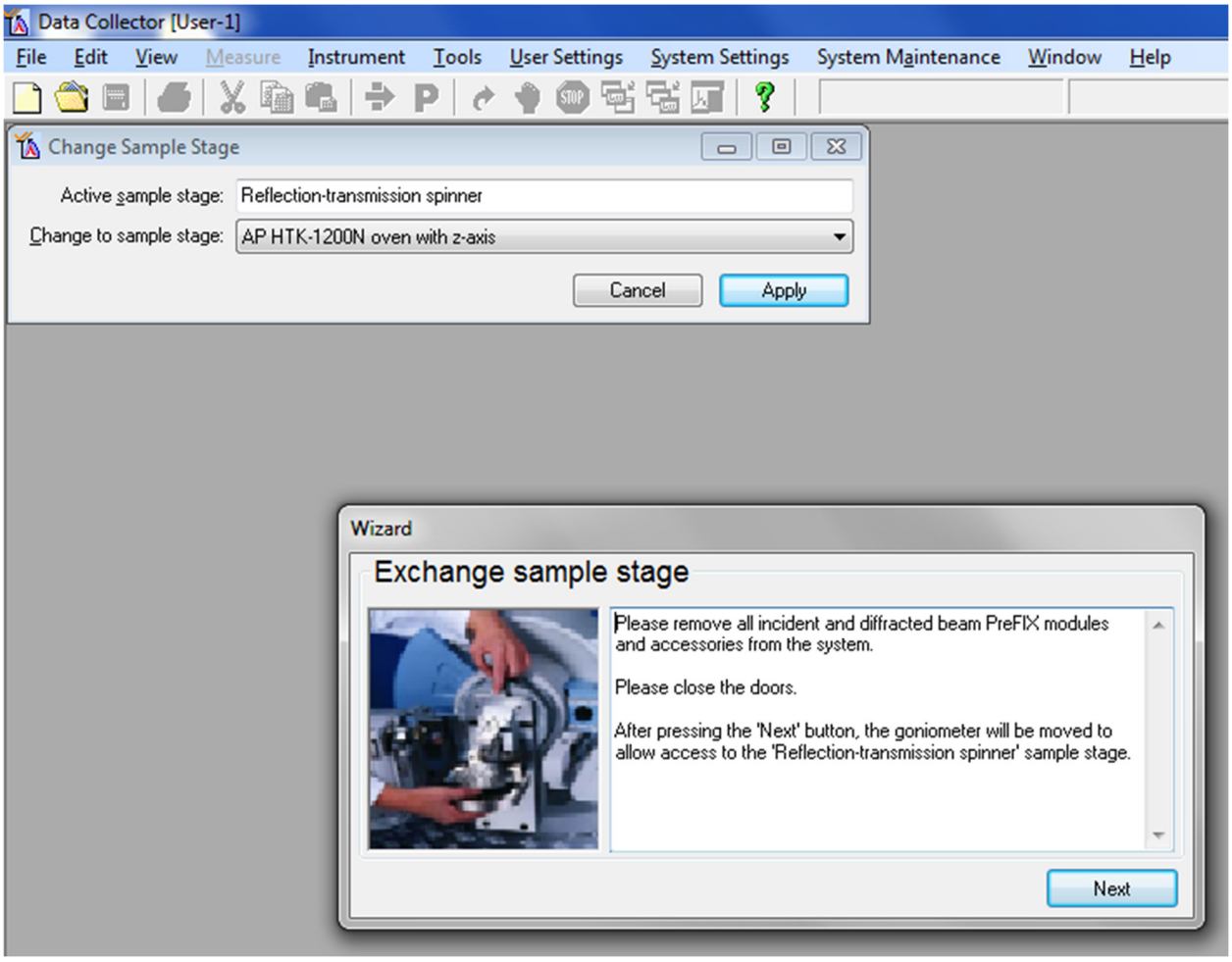
In Data Collector, go to the instrument tab. Disconnect the instrument. Follow the on screen instructions and wait for the Disconnect to finish.



Go to the Tools Tab, select “Change Sample Stage”. Follow the on screen instructions to change the stage to the “AP HTK-1200N oven with z-axis”.



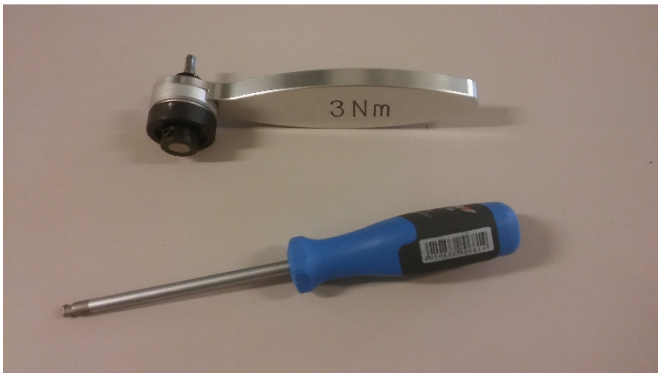




When prompted to remove the incident and diffracted beam Prefix modules unmount the BBHD and the detector as described below. Do not press the next button until the modules are removed.

Unmounting the BBHD:

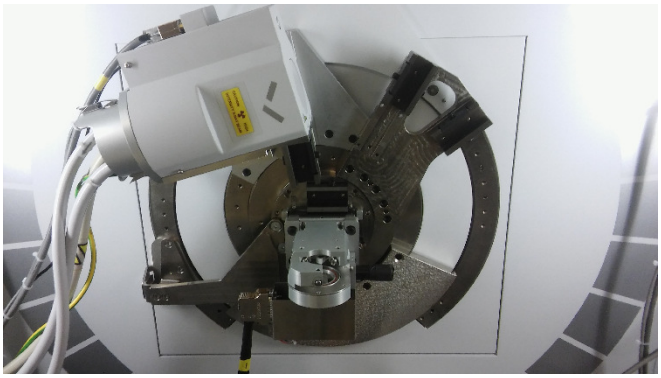
Always tightly hold the BBHD with one hand. With the other hand use first the small 3 Nm torque wrench to slightly loosen the top screw. Then use the small (blue) hex screw driver to completely loosen the top screw. Keep holding the BBDH, it can fall off! Remove the BBHD and carefully place it in a save corner of the instrument housing.



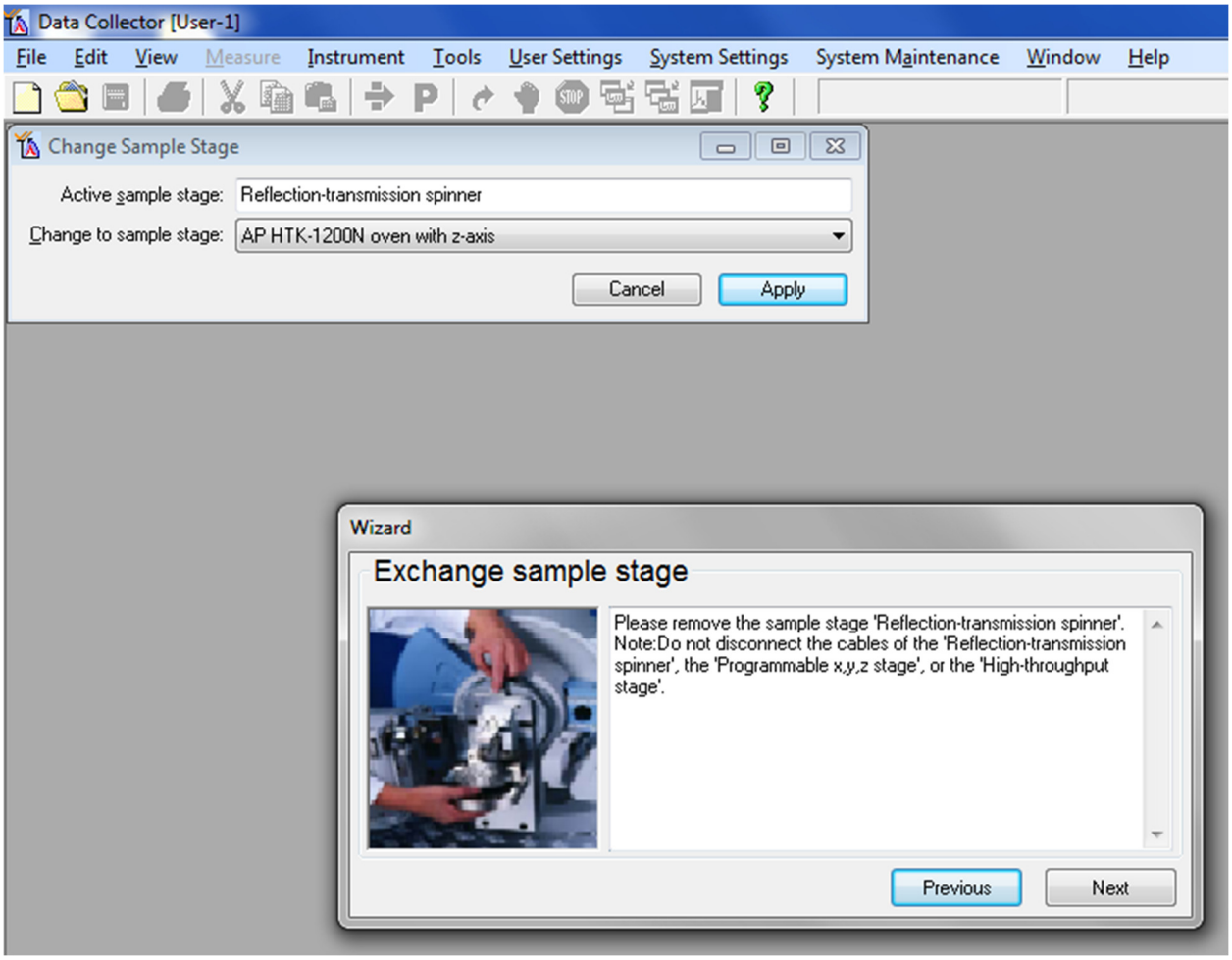
Unmounting the Detector:

Always tightly hold the detector with one hand (it can fall off!). With the other hand use first the small 3 Nm torque wrench to slightly loosen the top screw. Then use the small (blue) hex screw driver to completely loosen the top screw. Remove the detector and carefully place it on top of the slit assortment tray in the instrument housing. Do not disconnect any detector cables.

Once the modules are removed press the next button. The goniometer will drive the omega and theta arms up and out of the way to give you access to the stage.



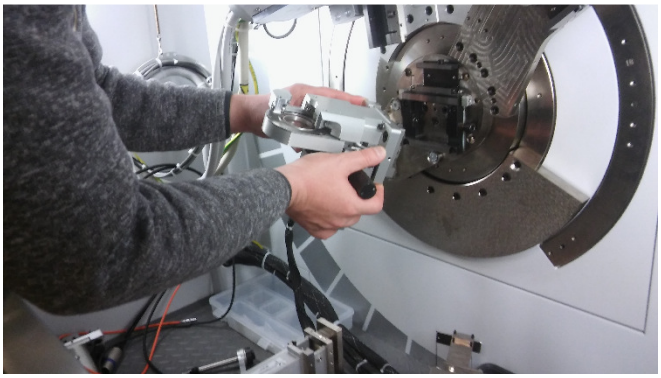
When prompted by the on screen messages remove the reflection transmission spinner stage as described below.



Use first the larger 10 Nm torque wrench to loosen the side screw (to the right of the stage). Then use the large (red) hex screw driver to completely loosen the side screw. The stage is securely locked even with the screw all the way out, it cannot fall off.

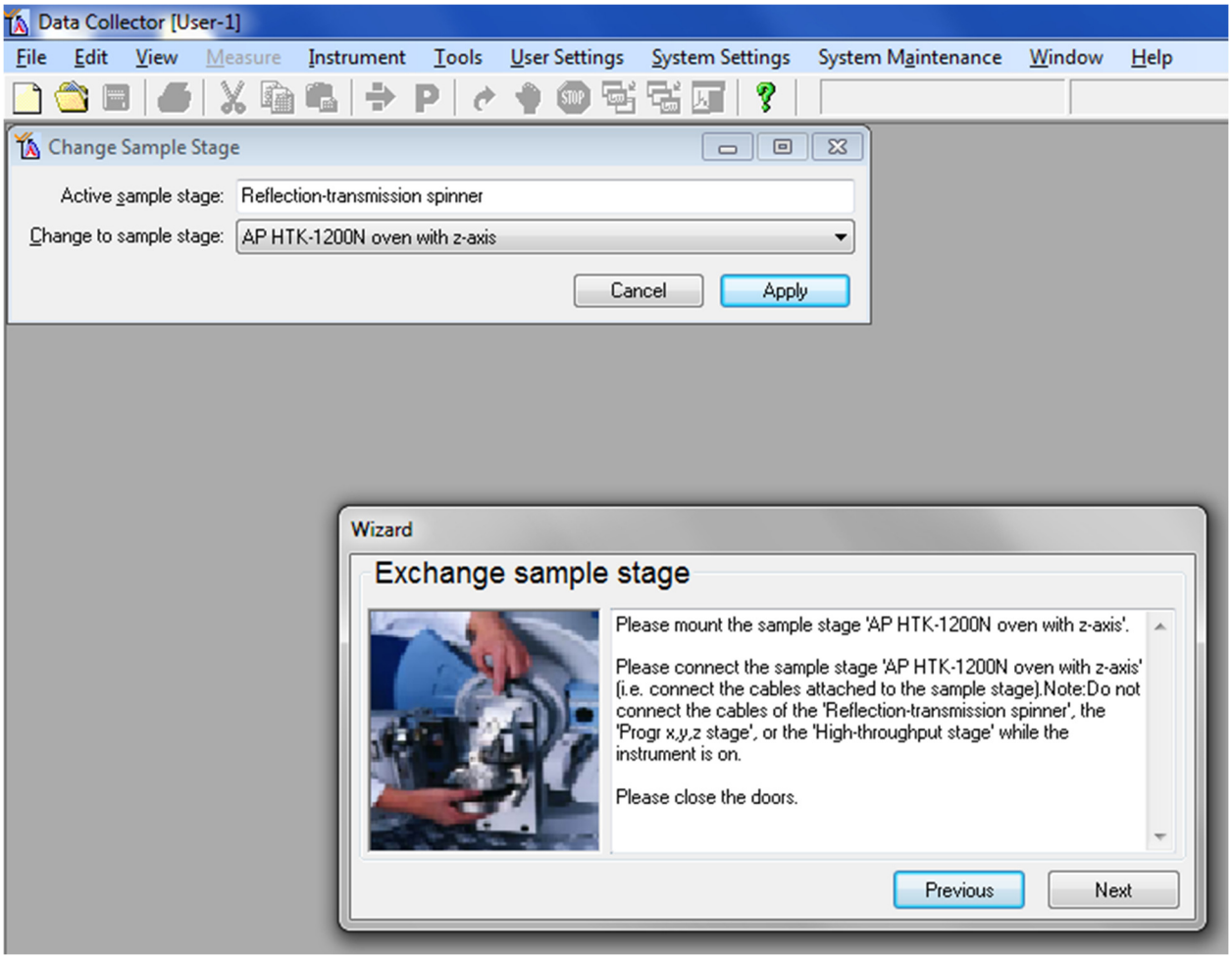
When the screw is far enough out the stage can be lifted out of its holder (it is secured with two pins in the back). Hold the stage securely with two hands at the bulky metal frame (not at any fragile parts or electronics, and do not grab into

the central sample holder area). Turn it towards you and up, and lift it out until the pins are completely off the sample stage mount. Place it carefully into a save corner inside the instrument housing. Do not disconnect any of its cables.

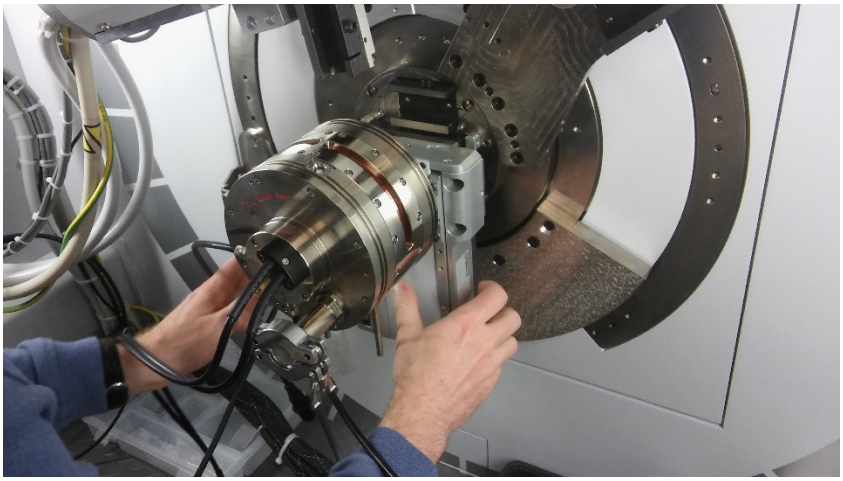


Press next in Data Collector.

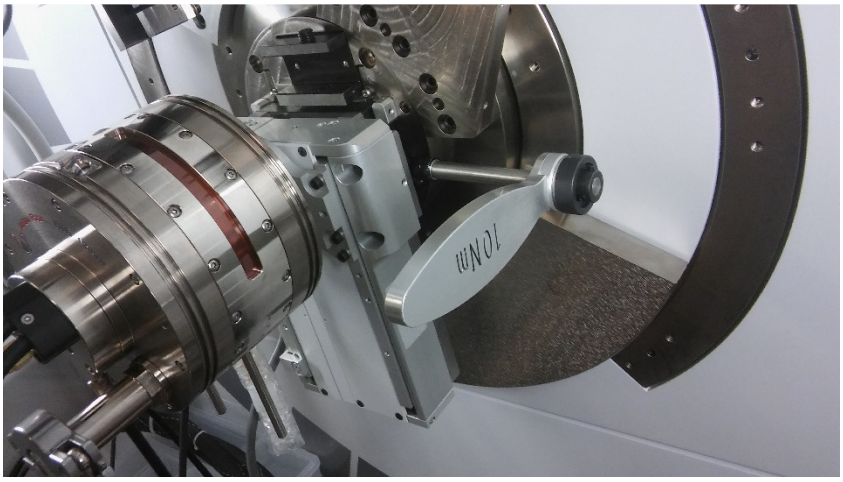
After removing the BBHD and detector, click “Next”. The following pop-up tells you that the AP HTK-1200N heated stage can be mounted. It is by default located inside the metal cabinet opposite the diffractometer.



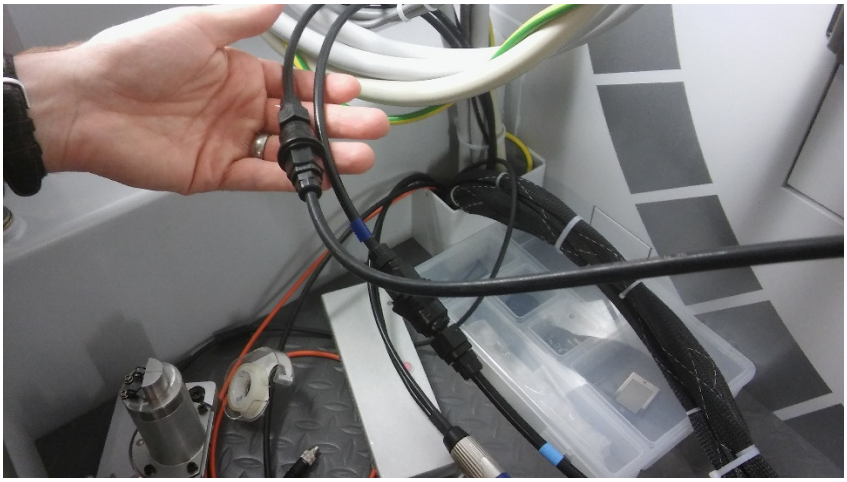
The heated stage is heavy; ask a second person to assist you with the next steps. To lift out the cradle, first securely wrap the cables and hoses around one of your arms so that they are not in the way. Then lift the cradle out of the cabinet, holding on to the bulky aluminum rectangular piece (NOT the circular pressure chamber.) Walk to the diffractometer and insert the pins of the mounting rack into the pin holes of the cradle using a forward/downward turning motion. Once the pins are securely inserted, slowly lower the cradle to check that it is indeed securely in place. Do not let go fast, the pins are difficult to see and might not be in place!



Once the cradle is securely affixed to the back of the diffractometer, tighten the screw to the right with the large (red) hex screw driver until hand tight. Then us the large 10 Nm torque wrench to tighten the screw until you hear one click of the wrench.

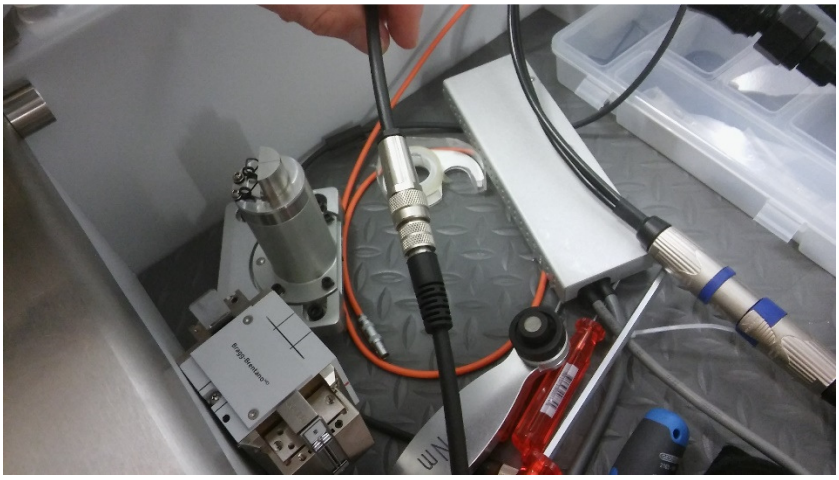


Connect the necessary cables to their mates. There are two black water hose connections with black plastic plugs. These are self-sealing, but place your hand underneath them while connecting as a few drops of water may come out.



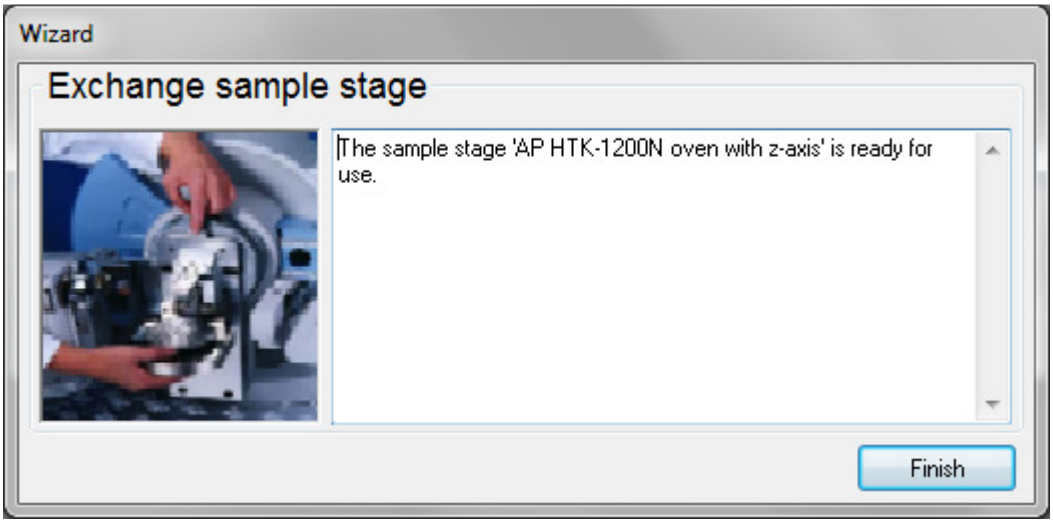
The large metal connector (with blue plastic bands) must be aligned prior to connection, after which they must be turned clockwise by about 10° to secure it.

The small metal connector (black wire) must be aligned prior to connection; once connected, screw the two halves together until tight. This will take multiple revolutions prior to achieving an acceptable connection.

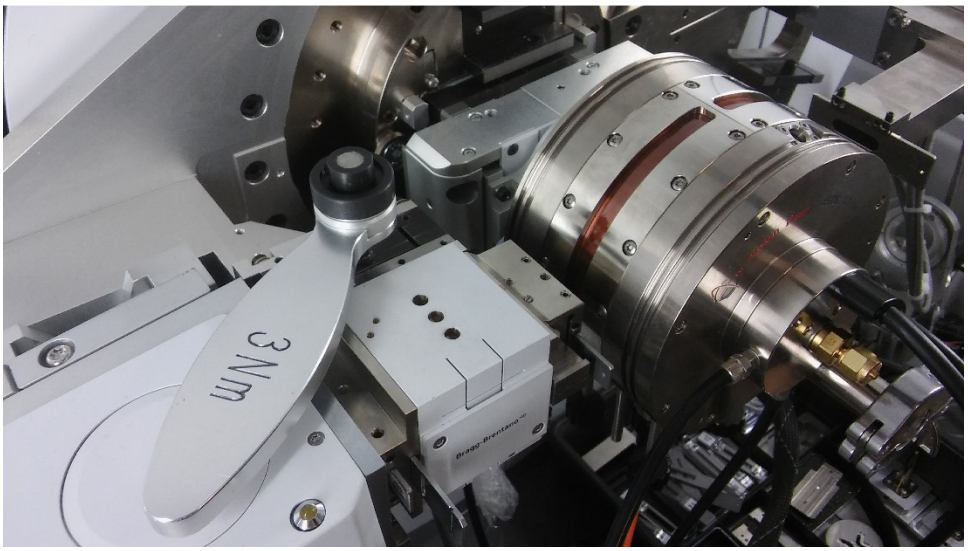


The long gray stage cable needs to be plugged in to any of the four slots to the right of the cabinet (look for red dots for cable plug orientation).

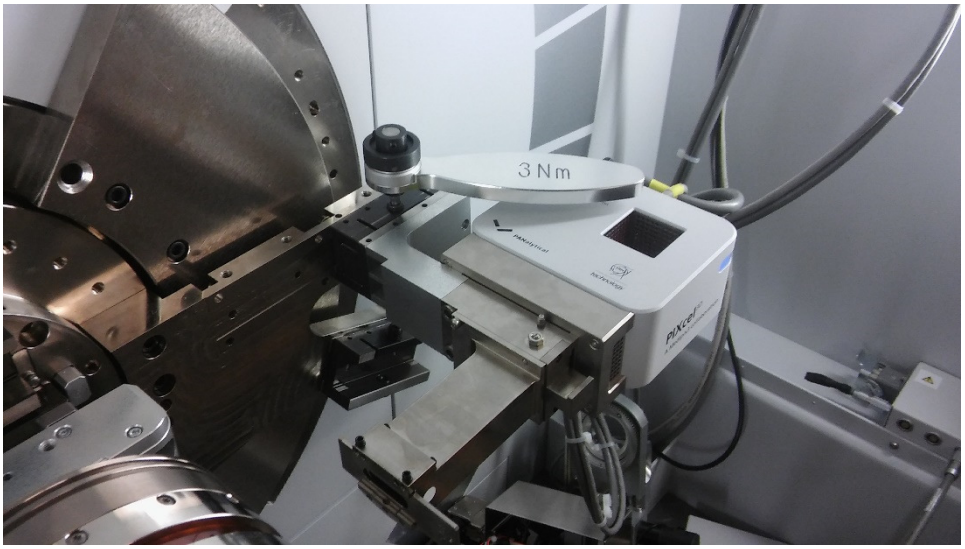
Click “Next” in the dialogue box. The instrument will drive the holders for the prefix modules (BBHD and Medipix) back into horizontal positions. A pop-up will tell you the stage is now ready to use (below). Click “Finish.”



Put the BBHD back in place. Always tightly hold the BBHD with one hand. With the other hand use the small (blue) hex screw driver to tighten the top screw until the BBHD is securely fastened. Then use the small 3 Nm torque wrench to completely tighten the top screw until you hear one click of the wrench. The BBHD is now in place.



Mount the detector to the holder of Diffracted Beam Path One (the top rail). Always tightly hold the detector with one hand (it can fall off). With the other hand use the small (blue) hex screw driver to tighten the top screw until the detector is securely fastened. Then use the small 3 Nm torque wrench to completely tighten the top screw until you hear one click of the wrench. The detector is now in place.


3 Nm

Mounting the sample

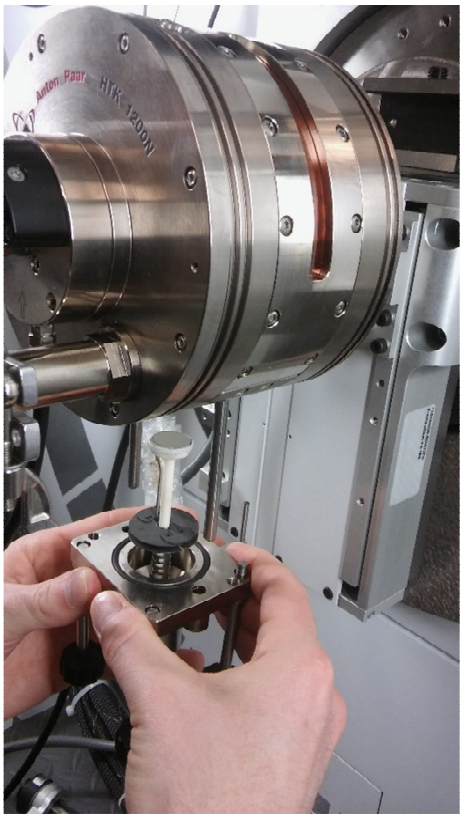
If you are using a powderous sample pack the sample into the ceramic sample carrier.

Bring over the tall ceramic sample holder from the storage cabinet.

Place your sample on top of the ceramic sample holder, carefully expand the ceramic snap ring slightly, and place the snap ring over your sample so that it holds the sample in place.

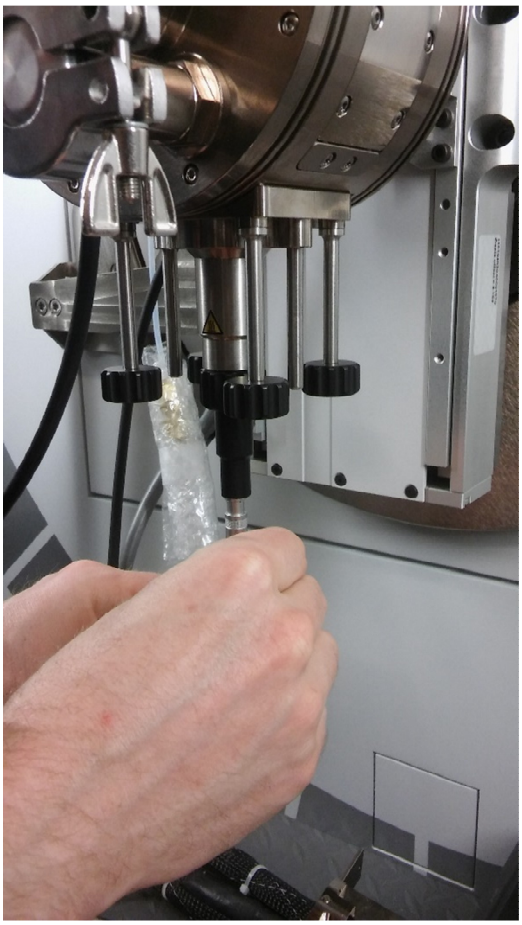
If you are using a solid sample make sure it is flat, has two exactly parallel sides, and is not much wider than the top of the sample holder. Place it loosely on top of the top of the sample holder.

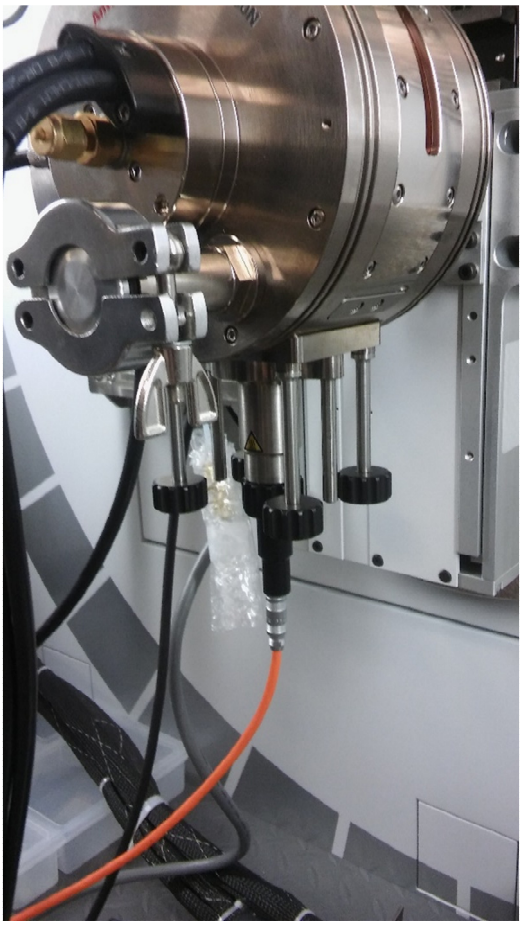
Take the sample holder out of its cradle and carefully align it into the heating chamber, making sure the small alignment pin is towards the back of the instrument. The two large pins on either side will be the first engaged alignment pins. As the sample holder is raised, take care that the small alignment pin is inserted correctly into the corresponding hole as well.



Once the sample is all the way into the chamber, begin tightening the screws on the four corners of the sample holder. These may take a little “wiggling” to align them into their threads. Tighten these until they are just beginning to get snug; do not tighten any of them completely until all four are fully engaged. At this point, tighten each consecutively in an X pattern until all four are snug and finger tight.

The orange cable must be connected to the bottom of the sample holder, again taking care to align the connectors prior to mating. Connect this cable by pushing it into the bottom of the sample holder. Disconnection requires you to pull the outer shell down, then pull the connector out.



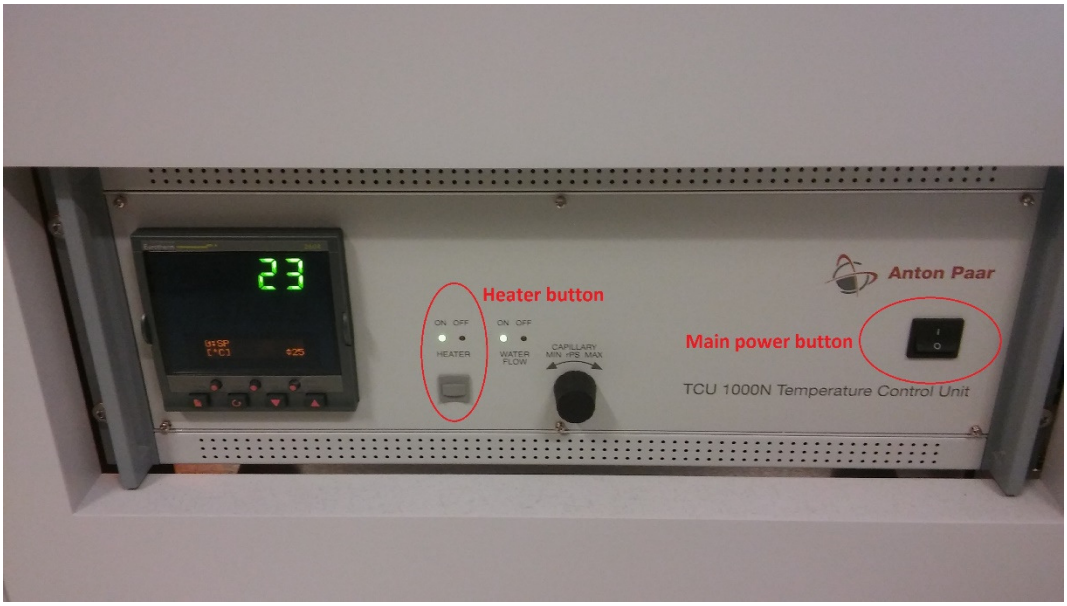


If you would like to use gases other than air, or measure under vacuum, contact Dr. Zeller (zeller4@purdue.edu).

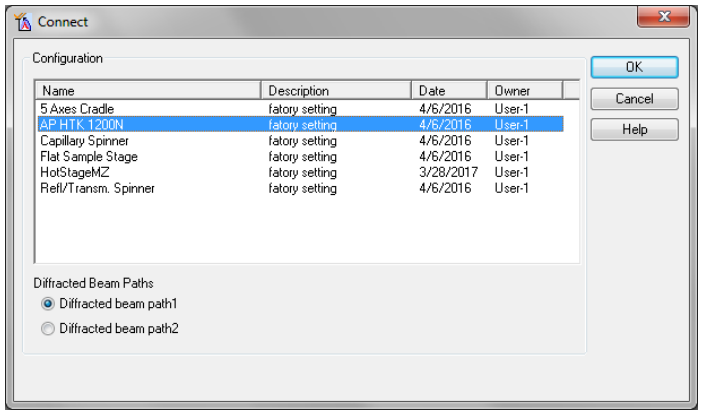
Automatic Height Alignment

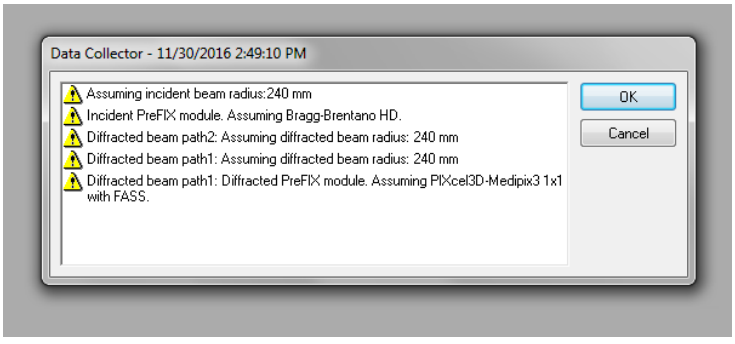
The Automatic Height Alignment procedure requires the Cu 0.1 attenuator which fits in the mask slot to be installed (pictured below). Ensure this is installed prior to proceeding, with the notch to the LEFT (the notch is on the opposite side of the attenuator from what is pictured).

Switch on the controller for the hot stage. It is located in the left lower corner of the front side of the diffractometer. Press the main black powder button. Then switch on the heater (small grey button on front of controller).



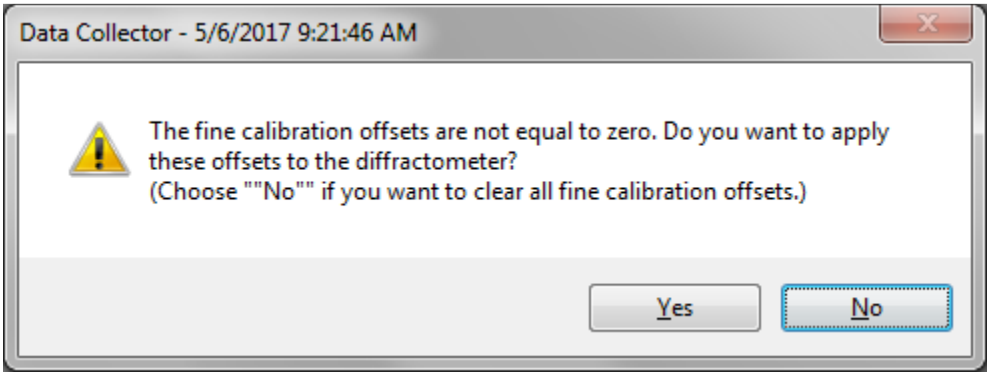
In data collector, go to the instrument tab. Connect to the instrument. Select the AP HTK-1200N heated stage when prompted to select a stage.





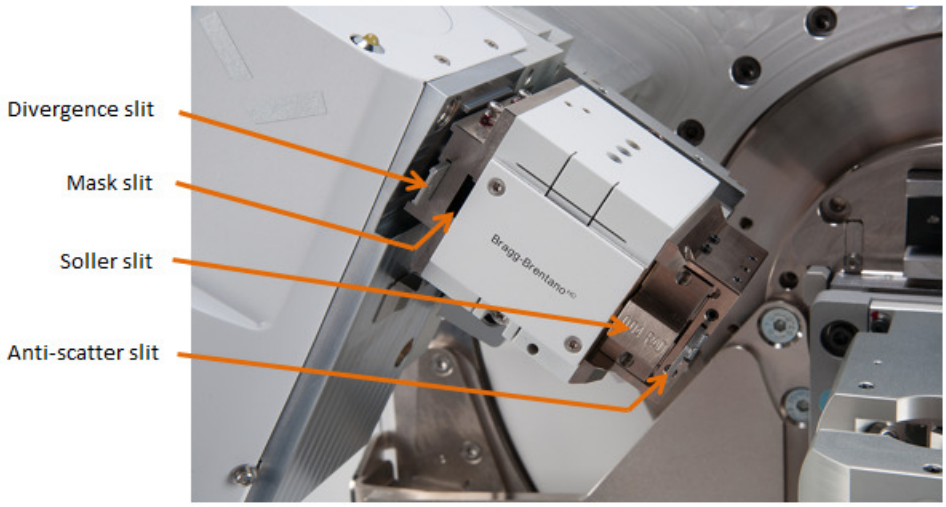
Follow the on screen instructions. Confirm the list of “assumed” modules (you will need to update these in the next steps) and wait for the “Connect” to finish.

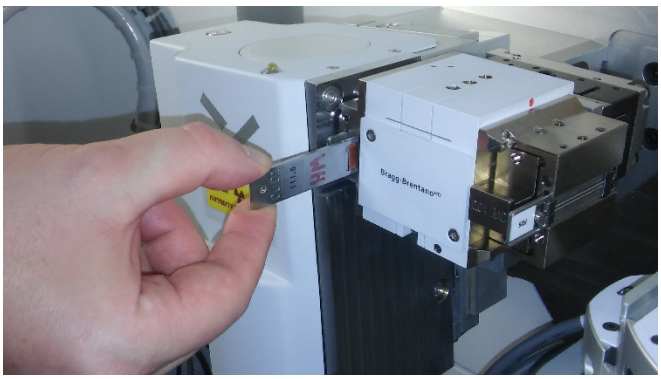
Clear the calibration offsets if the following pop-up window appears:



Run the hot stage auto height alignment program. The following configuration is needed for this procedure:

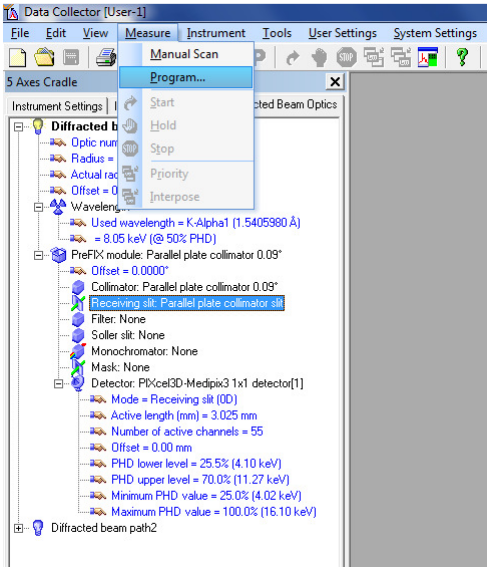
Incident beam: BBHD with 1/8° divergence slit, 1/16° anti-scatter slit, 0.04 rad soller slits, and a 0.1 mm Cu attenuator in the mask slit.



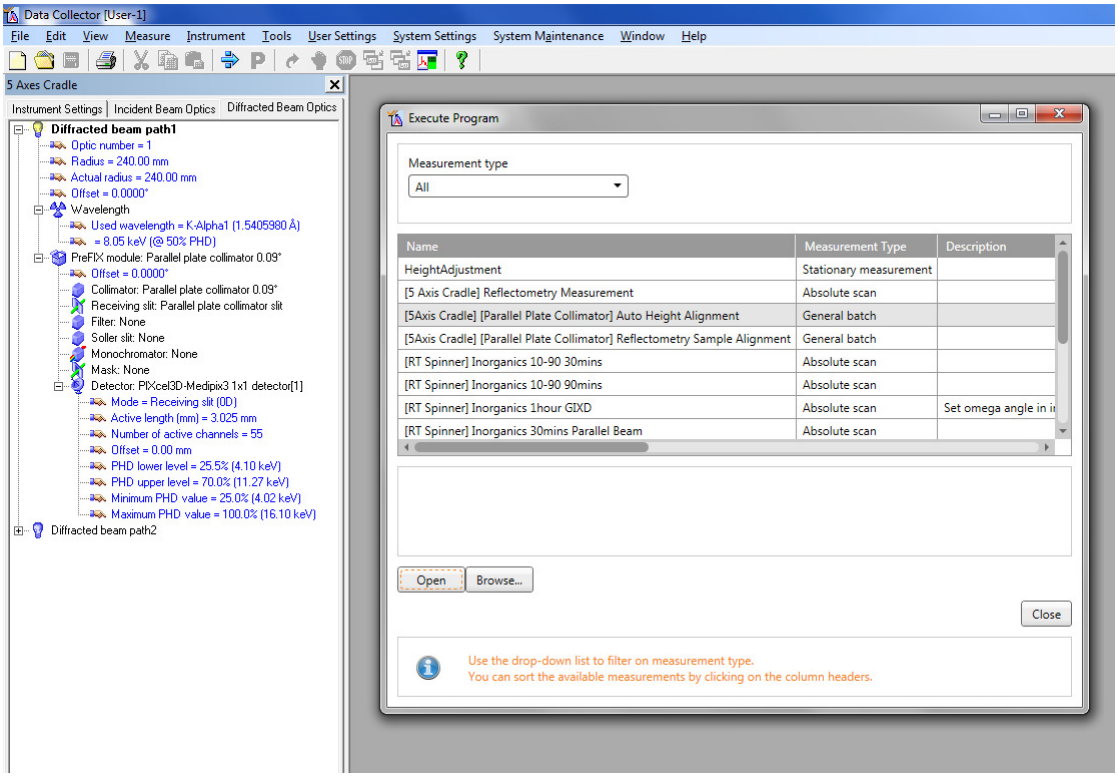


Update the configuration on the Incident Beam Optics tab.

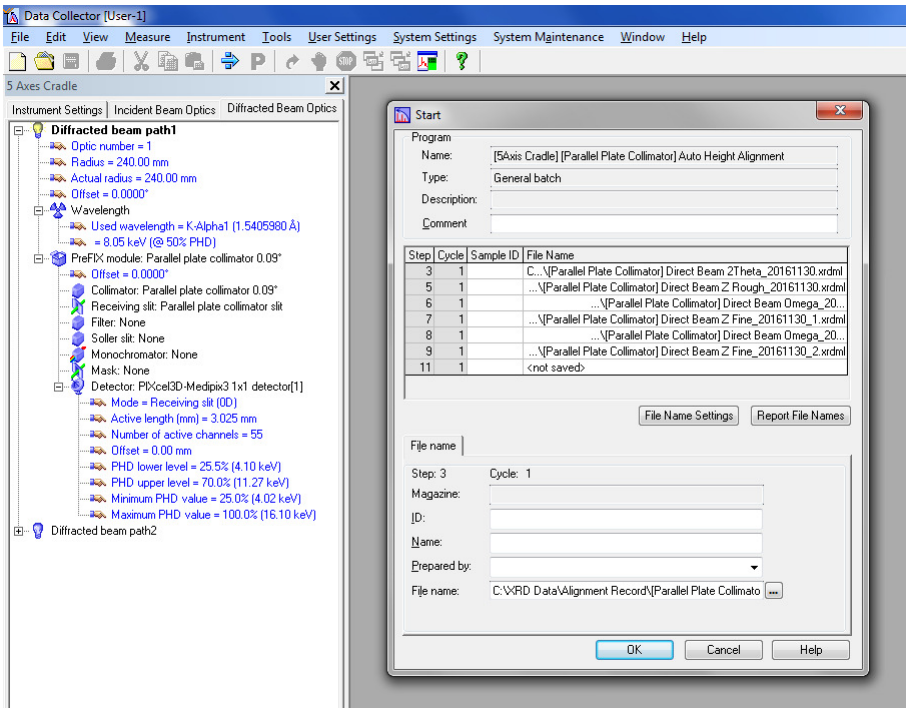
Make sure the generator is at 45kV and 40 mA. Go to Measure-> Program.



Open the program “[Hot Stage] [BBHD] [PIXcel] Auto Height Alignment.” If you haven’t inserted the manual beam attenuator, do this now (a pop-up comes up to remind you if the entries in the instrument tabs are correct).



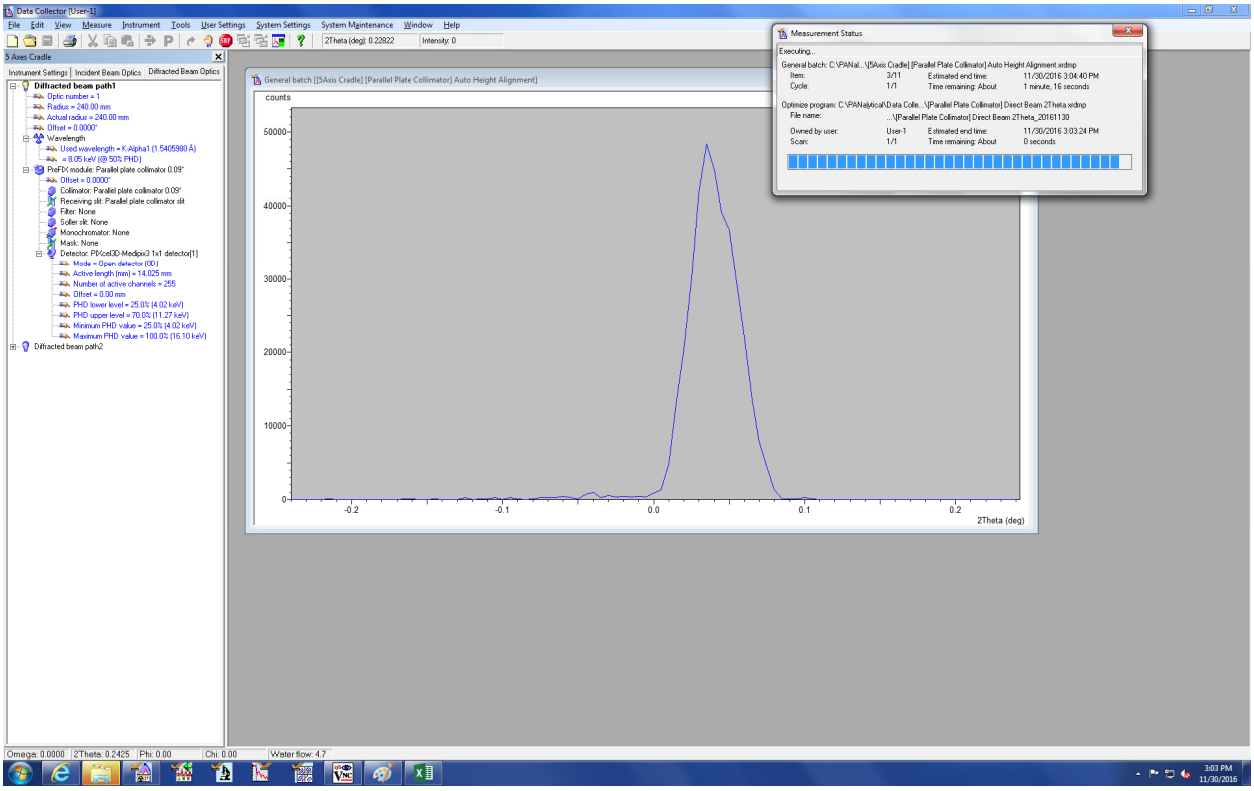
In the Start window that opens, click OK.

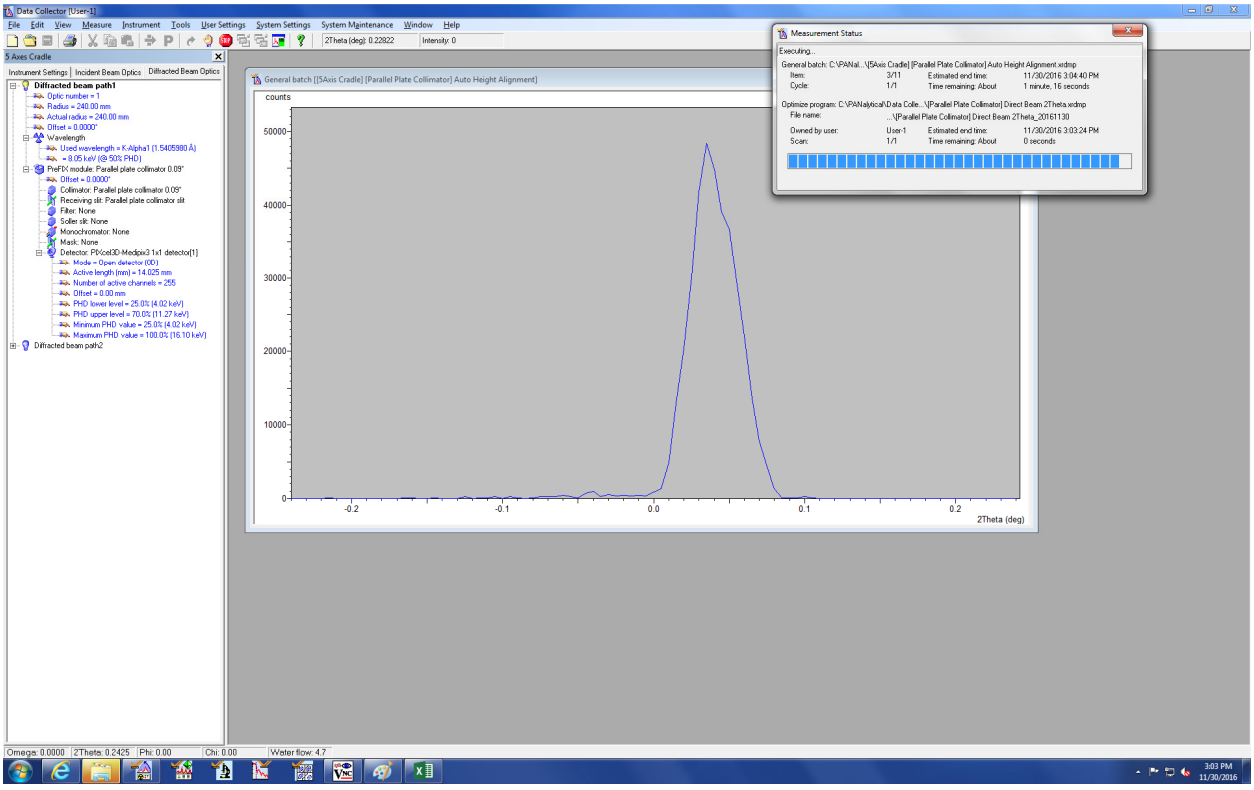


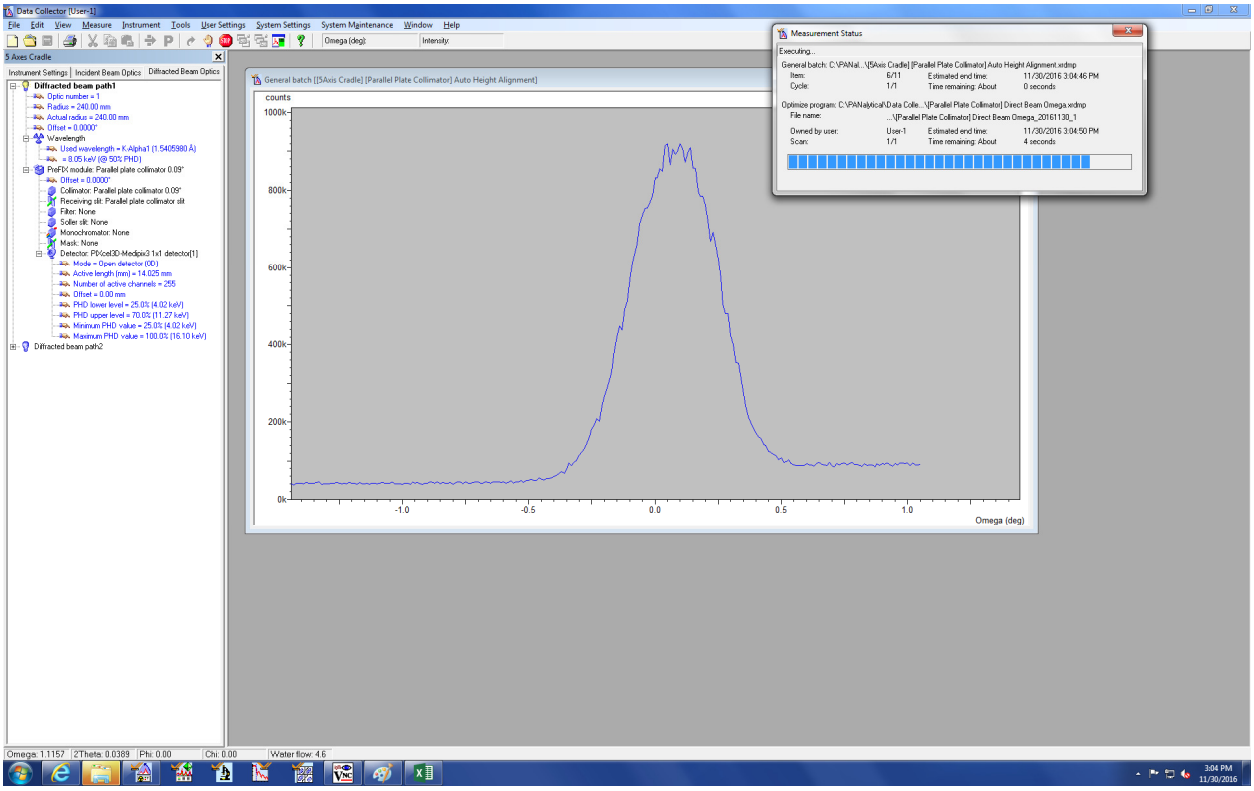
This will bring the temperature in the heater to 35°C and begin an automatic height alignment procedure. Note that it takes a while for it to stabilize at 35°C. It may also ask you to remove the fixed 4mm mask, just press OK as you have already done this to install the attenuator.

The procedure will scan 2Theta through the direct beam; scan the cradle height (Z) to identify when the beam is cut off by the sample to measure the sample height; scan omega to adjust for a slight angle of the sample wrt to the sample stage. The procedure is repeated automatically for fine adjustments.

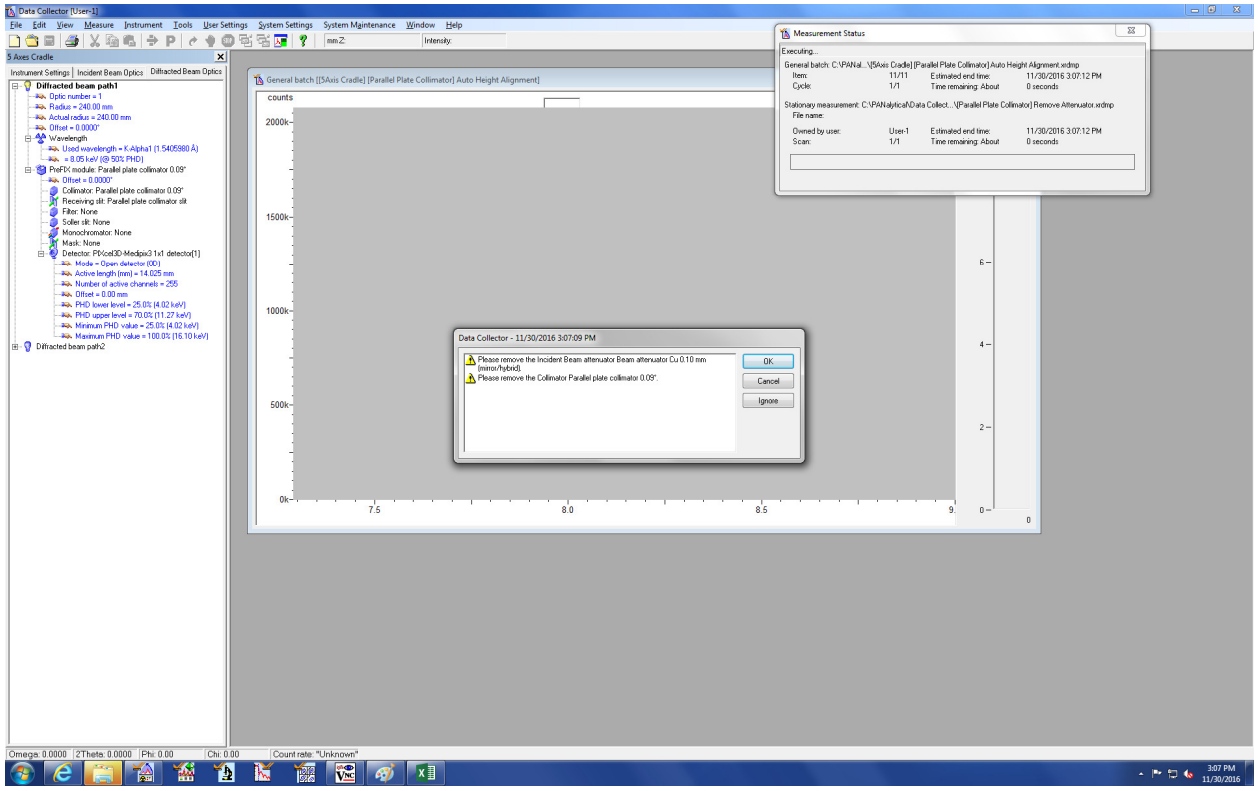
Watch the adjustment procedure. If the measurements look noisy (i.e. not like in the images below), then the procedure may need to be repeated. If you do not see well defined 2theta, Z and omega curves after three trials, ask for help.


Data Collector [User-1]
File Edit View Measure Instrument Tools User Settings System Settings System Maintenance Window Help
5 Axes Cradle
Instrument Settings | Incident Beam Optics | Diffracted Beam Optics
Diffracted beam path1
Optic number = 1
Radius = 240.00 mm
Actual radius = 240.00 mm
Offset = 0.0000°
Wavelength
Used wavelength = K-Alpha1 (1.5405980 Å)
= 8.05 keV (@ 50% PHD)
PreFIX module: Parallel plate collimator 0.09°
Offset = 0.0000°
Collimator: Parallel plate collimator 0.09°
Receiving slit: Parallel plate collimator slit
Filter: None
Soller slit: None
Monochromator: None
Mask: None
Detector: PIXcel3D-Medipix3 1x1 detector[1]
Mode = Open detector (0D)
Active length (mm) = 14.025 mm
Number of active channels = 255
Offset = 0.00 mm
PHD lower level = 25.0% (4.02 keV)
PHD upper level = 70.0% (11.27 keV)
Minimum PHD value = 25.0% (4.02 keV)
Maximum PHD value = 100.0% (16.10 keV)
Diffracted beam path2
General batch [[5Axis Cradle] [Parallel Plate Collimator] Auto Height Alignment]
counts
50000
40000
30000
20000
10000
0
-0.2
-0.1
0.0
0.1
0.2
2Theta (deg)
Measurement Status
Executing...
General batch: C:\PANal...\[5Axis Cradle] [Parallel Plate Collimator] Auto Height Alignment.xdmp
Item: 3/11
Cycle: 1/1
Estimated end time: 11/30/2016 3:04:40 PM
Time remaining: About 1 minute, 16 seconds
Optimize program: C:\PANalytical\Data Colle...\[Parallel Plate Collimator] Direct Beam 2Theta.xdmp
File name: ...\[Parallel Plate Collimator] Direct Beam 2Theta_20161130
Owned by user: User-1
Scan: 1/1
Estimated end time: 11/30/2016 3:03:24 PM
Time remaining: About 0 seconds
Omega: 0.0000 | 2Theta: 0.2425 | Phi: 0.00 | Chi: 0.00 | Water flow: 4.7
3:03 PM
11/30/2016

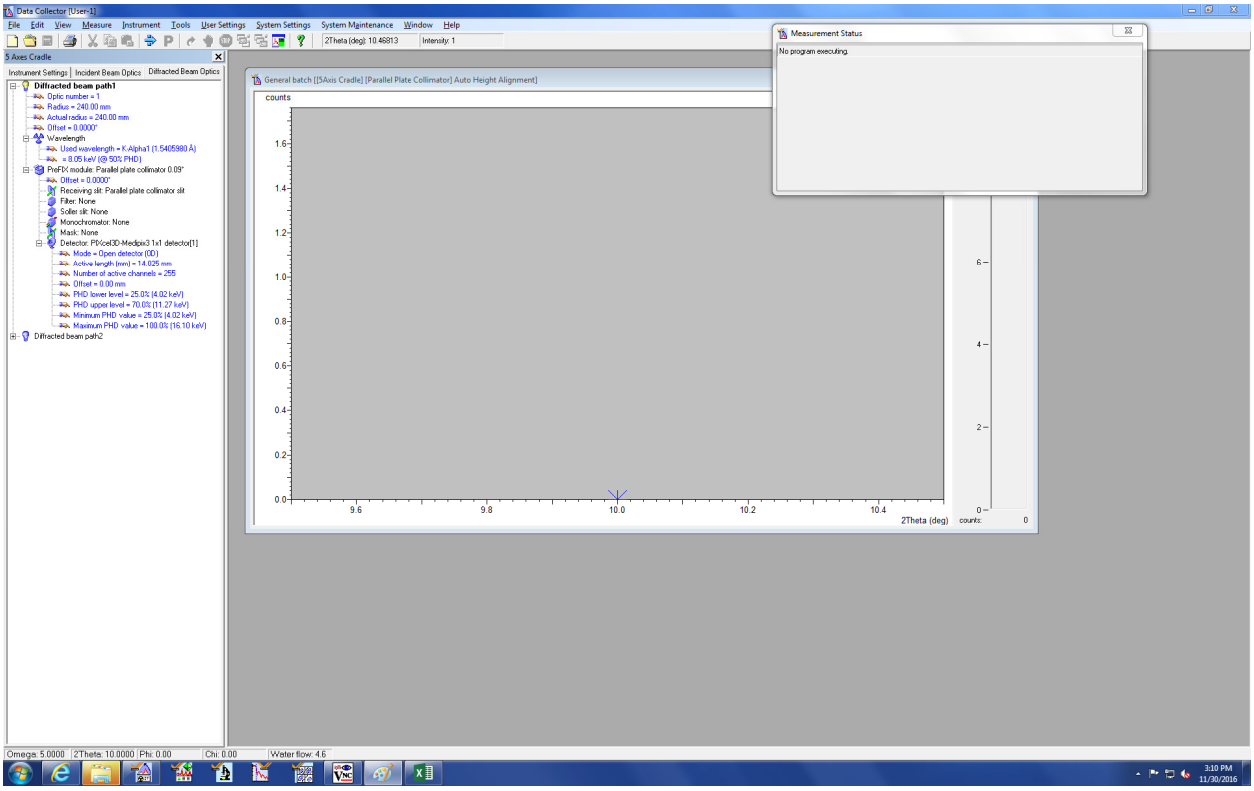

Data Collector [User-1]
File Edit View Measure Instrument Tools User Settings System Settings System Maintenance Window Help
5 Axes Cradle
Instrument Settings | Incident Beam Optics | Diffracted Beam Optics
Diffracted beam path1
Optic number = 1
Radius = 240.00 mm
Actual radius = 240.00 mm
Offset = 0.0000°
Wavelength
Used wavelength = K-Alpha1 (1.5405980 Å)
= 8.05 keV (@ 50% PHD)
PreFIX module: Parallel plate collimator 0.09°
Offset = 0.0000°
Collimator: Parallel plate collimator 0.09°
Receiving slit: Parallel plate collimator slit
Filter: None
Soller slit: None
Monochromator: None
Mask: None
Detector: PIXcel3D-Medipix3 1x1 detector[1]
Mode = Open detector (0D)
Active length (mm) = 14.025 mm
Number of active channels = 255
Offset = 0.00 mm
PHD lower level = 25.0% (4.02 keV)
PHD upper level = 70.0% (11.27 keV)
Minimum PHD value = 25.0% (4.02 keV)
Maximum PHD value = 100.0% (16.10 keV)
Diffracted beam path2
General batch [[5Axis Cradle] [Parallel Plate Collimator] Auto Height Alignment]
counts
50000
40000
30000
20000
10000
0
-0.2
-0.1
0.0
0.1
0.2
2Theta (deg)
Measurement Status
Executing...
General batch: C:\PANal...\[5Axis Cradle] [Parallel Plate Collimator] Auto Height Alignment.xdmp
Item: 3/11
Cycle: 1/1
Estimated end time: 11/30/2016 3:04:40 PM
Time remaining: About 1 minute, 16 seconds
Optimize program: C:\PANalytical\Data Colle...\[Parallel Plate Collimator] Direct Beam 2Theta.xdmp
File name: ...\[Parallel Plate Collimator] Direct Beam 2Theta_20161130
Owned by user: User-1
Scan: 1/1
Estimated end time: 11/30/2016 3:03:24 PM
Time remaining: About 0 seconds
Omega: 0.0000 | 2Theta: 0.2425 | Phi: 0.00 | Chi: 0.00 | Water flow: 4.7
3:03 PM
11/30/2016



Towards the end of the automatic procedure, the routine will ask to remove the Cu attenuator. Replace the Cu attenuator by an appropriate mask (not wider than your sample).



Wait for the program to finish, stating “No Program executing”.



The results of the Automatic Height Alignment are written to the software as offset values. They can be looked up under "User Settings", then "Fine Calibration Offsets". Typical values are 1° for 2Theta, 0.5° for omega.

The instrument is now ready for measurement using the hot stage.

Running a sample

Before running a sample under non-ambient conditions ensure that Data Collector is using the automatic height control that compensates for expansion of the stage. Under the "Instrument Settings" tab, double click on "Sample stage: AP HTK-1200N oven with z-axis." In the window that comes up, under the "Sample Stage" tab, select AP HTK-1200N Oven (select the type you are using, air, gas used, or vacuum). This will activate the automatic height control.
To set the temperature of the heater, change the set temperature in the same window by clicking the checkbox next to "Temperature (°C)", then typing in the desired temperature. Enter a ramp rate anywhere between 0-150°C. Alternatively, click on "Use Max Ramp" to use the maximum ramp rate. Click on "Apply." The stage will go to the set temperature prior to allowing you to do anything else. Once the set temperature is reached, press "OK" to exit the dialogue box.

For developing your specific program, you can use the program "[Hot Stage] Inorganics 25-90 30 mins as a starting point for your specific needs.

After measurements are complete

When finished, set the temperature to 35°C and wait for the oven to attain this temperature. Do not set it lower than this or it will take a very long time to reach the lower set point. Also, make sure to set the generator to 20 mA if the instrument will be idle for an extended period.

Disconnect the instrument and go off-line.

Switch off the heater button on the heater control box (small gray button). Switch off the heater control box.

Go to the Tools tab, select “Exchange Sample Stage”. Follow the on-screen instructions to change to the RT Spinner stage, including removing the BBHD and detector, and removing the high temperature stage (screenshots below). Follow the reverse procedure as described for mounting the hot stage.