

# Standard Operating Procedure – Thin Film Setup, Panalytical Empyrean Diffractometer

X-ray Crystallography Laboratory  
Purdue Department of Chemistry

*Measurements of thin films and reflectivity measurements make use of the five axis cradle for sample mounting. This SOP describes how to change the stage to the five axis cradle if it is not already installed.*

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

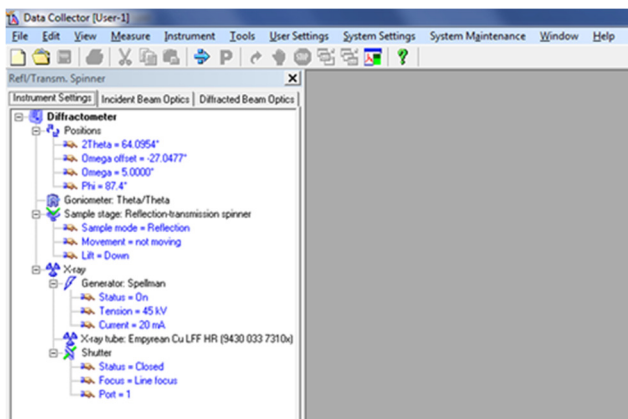
Before getting started, inspect the instrument and check which stage and optics are mounted, and what slits and masks are used.

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, and the PIXcel Medipix 3D detector with the “Fixed Anti Scatter Slit” (FASS) on the diffracted beam side. 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.

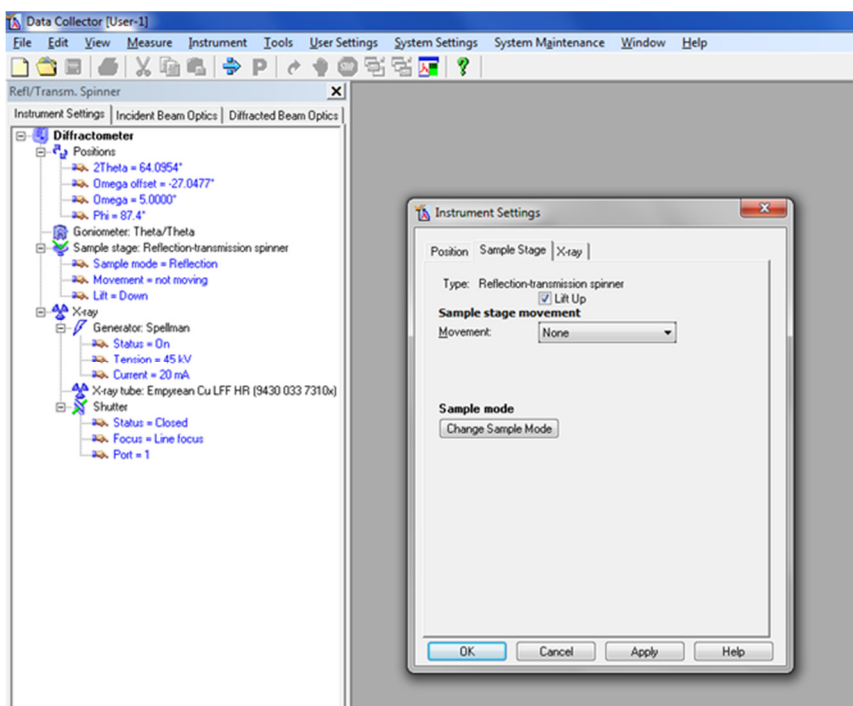
The image below shows the typical view of Data Collector after connection is established. Toggle between the tabs for “Instrument Settings”, “Incident Beam Optics” and “Diffracted Beam Optics” to check that the physically present stage, optics, slits and masks are identical to what is listed in Data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



The 5 axis cradle is usually used in combination with the BBHD and with the PIXel Medipix 3D detector mounted on the “Parallel Plate Collimator” (PCC). The following procedure describes how to change the setup from the former configuration to the latter. If you encounter a different setup, or would like to install a different type of optics, contact Dr. Zeller (zeller4@purdue.edu) before making any changes.

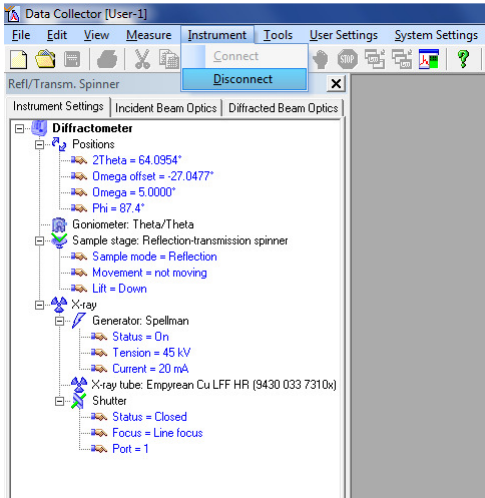
Changing the stage:

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.

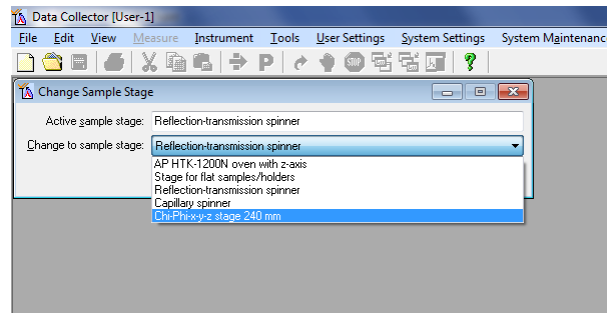
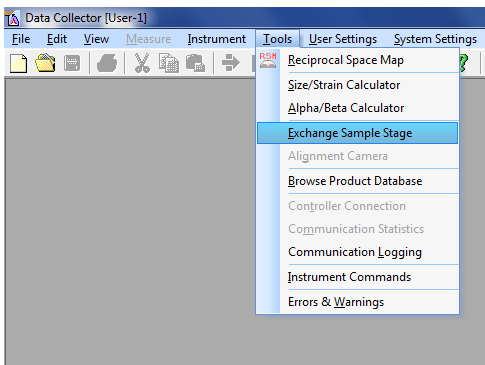


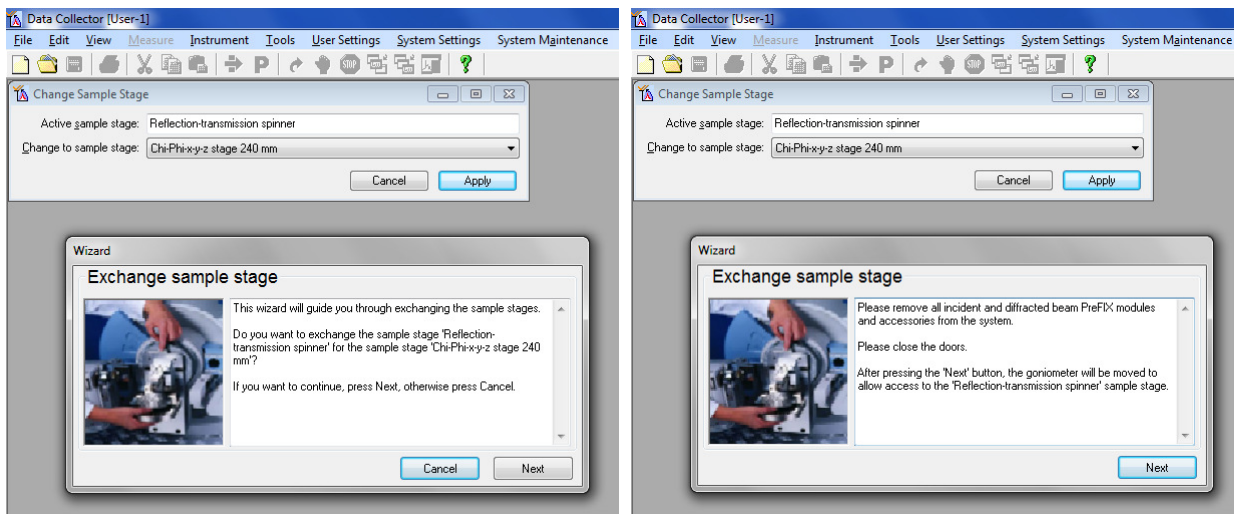
Check “Lift up”, Click “OK” to move the lift up again prior to changing the stage and close the pop-up window.

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.

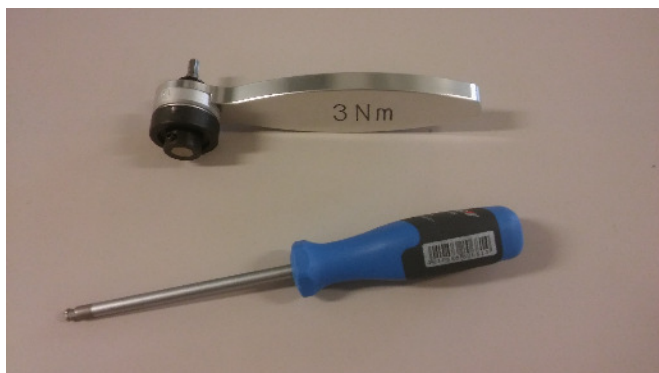




When prompted to remove the incident and diffracted beam Prefix modules unmount the BBHD and the detector as described below. Do not press the next button until the modules are removed.

#### 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 BBHD, it can fall off! Remove the BBHD and carefully place it in a save corner of the instrument housing.



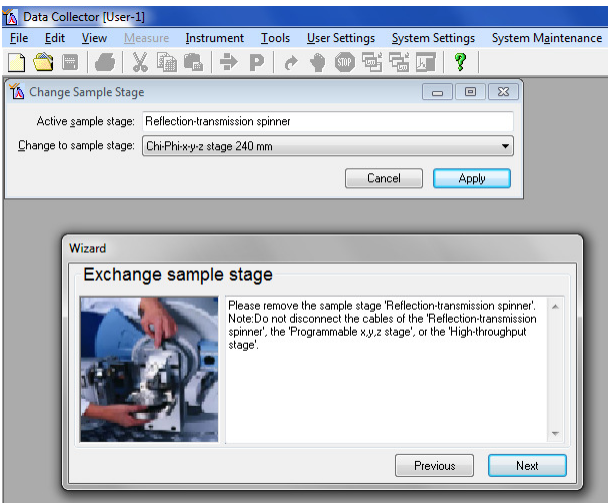
#### Unmounting the Detector:

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.

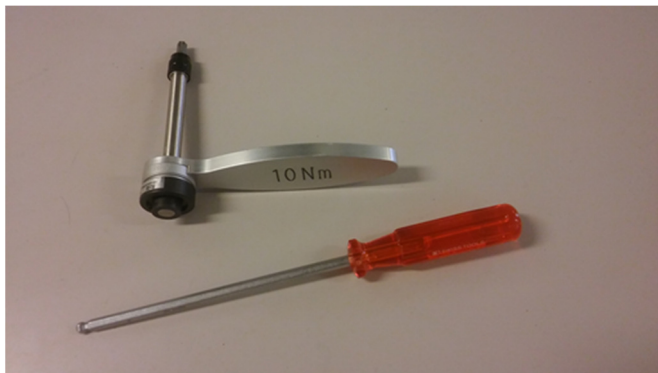
Once the modules are removed press the next button. The goniometer will drive the omega and theta arms up and out of the way to give you access to the stage.



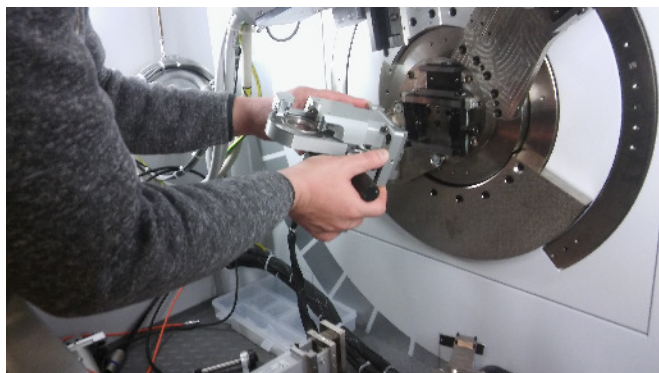
When prompted by the on screen messages remove the reflection transmission spinner stage.



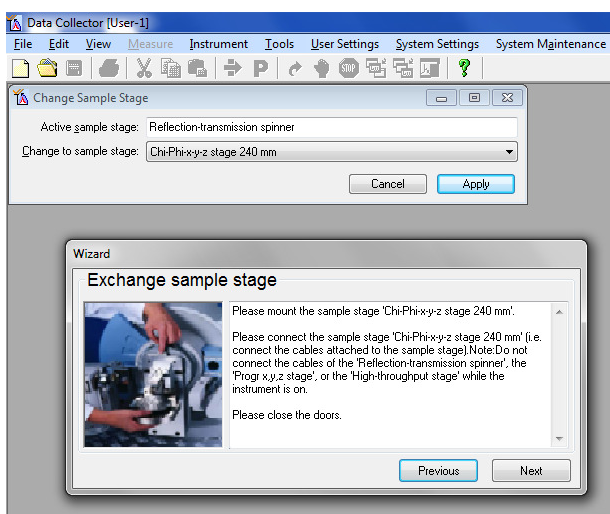
Use first the larger 10 Nm torque wrench to loosen the side screw (to the right of the stage). Then use the large (red) hex screw driver to completely loosen the side screw. The stage is securely locked even with the screw all the way out, it cannot fall off.



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.



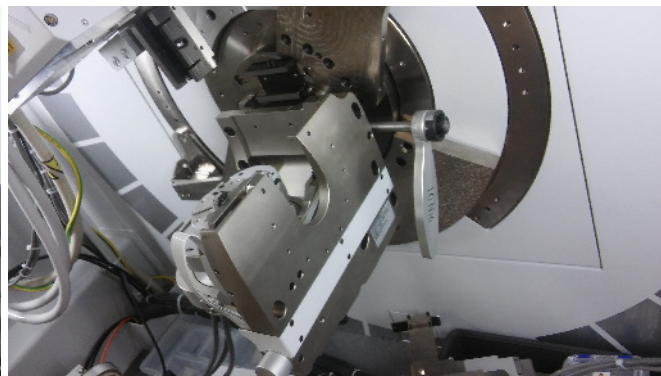
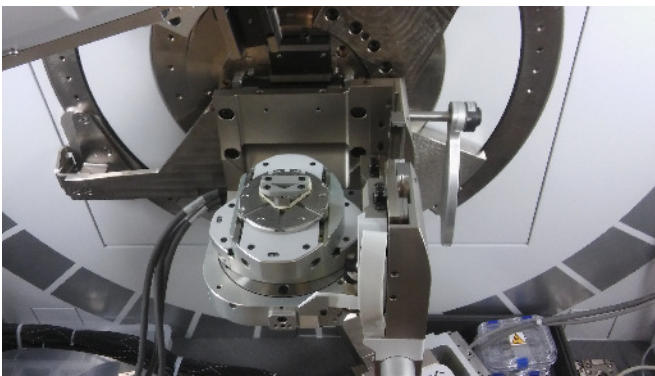
Press next in Data Collector.



Now the 5-axis cradle can be mounted. It is by default located inside the metal cabinet opposite the diffractometer.



The cradle is very heavy and bulky. Ask a second person to assist you with the next steps. The cradle sits on a set of rubber feet, with its back side (the “mounting side”) towards the back of the cabinet. The cradle has grips for your fingers on its underside. Locate these before proceeding. To lift out the cradle, first securely wrap the cables of the cradle around one of your arms or shoulder so that they are not in the way, or ask the second person to carry the cables. Then lift the cradle out of the cabinet using the finger grips on its underside. 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. Don’t 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 use the large 10 Nm torque wrench to tighten the screw until you hear one click of the wrench.



Connect the stage cable to any of the five slots to the right of the cabinet (look for red dots for cable plug orientation).



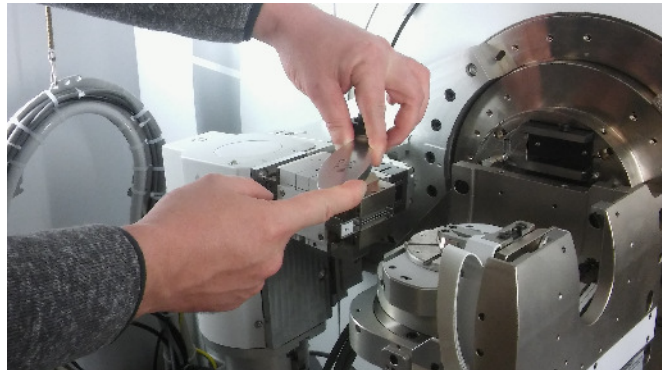
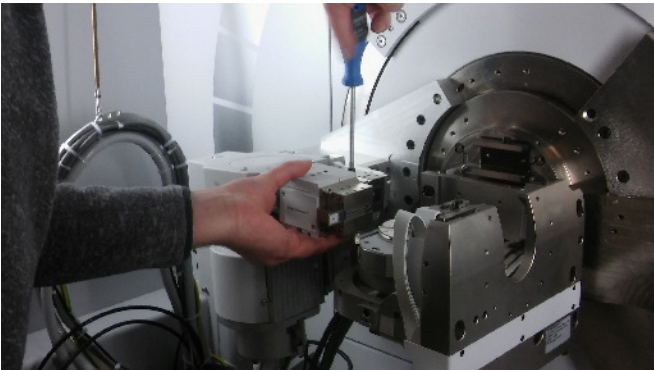


The stage is now in place.



Follow the on screen prompts.

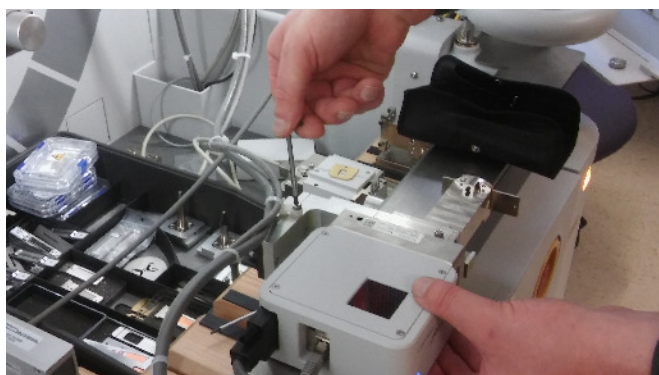
Put the BBHD back in place. Always tightly hold the BBHD with one hand. With the other hand use the small (blue) hex screw driver to tighten the top screw until the BBHD is securely fastened. Then use the small 3 Nm torque wrench to completely tighten the top screw until you hear one click of the wrench.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



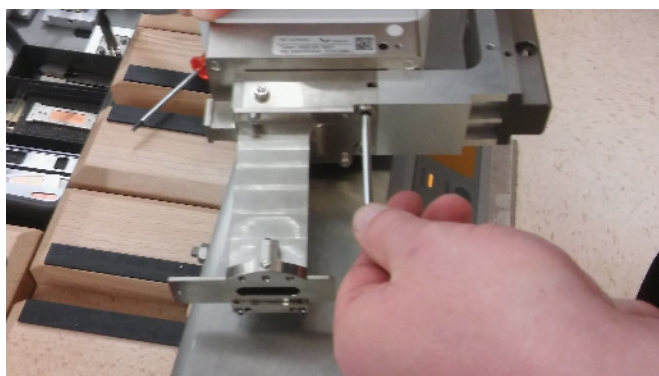
The detector cannot be remounted right away. It first has to be taken off the FASS mount and affixed to the PCC mount instead.

The detector cable is still connected and the detector cannot be taken out of the diffractometer housing for disassembly. Place the detector with the FASS mount carefully on the edge of the diffractometer housing. Always carefully hold the detector. If possible, have a second person assist you with these steps.

The cable is connected with a screw and plastic tie to the FASS mount. Unscrew it.

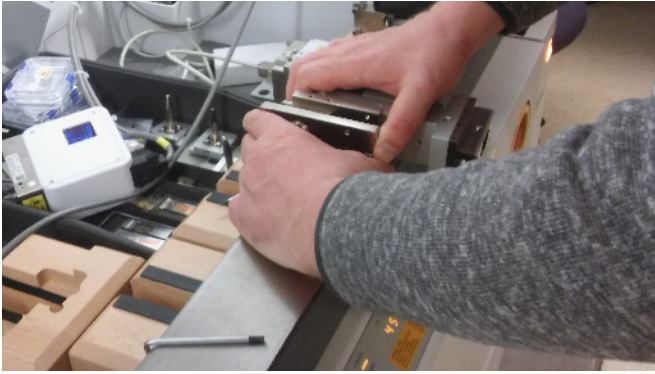


The detector is mounted to the FASS holder with two hex mounting screws and one pin. Loosen the screws completely. Use the T20 hex Allen wrench from the black pouch inside the cabinet for this. The screws will remain with the FASS holder.

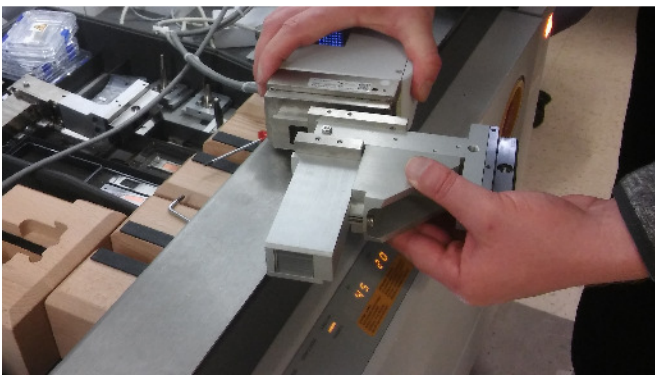
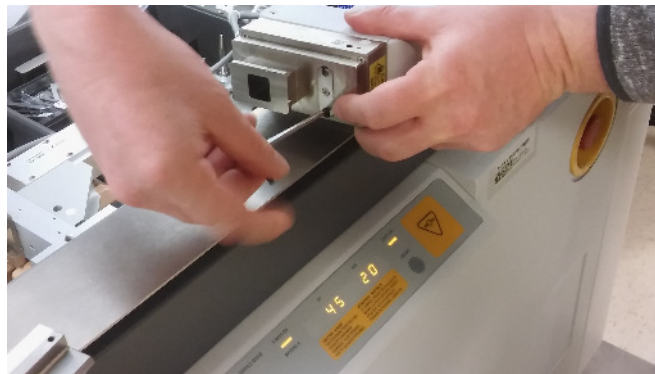


Take off the FASS holder and carefully place it in a save place inside the cabinet.

Take the PCC mount and slide off the holder that mounts onto the detector to access the hex mounting screws.

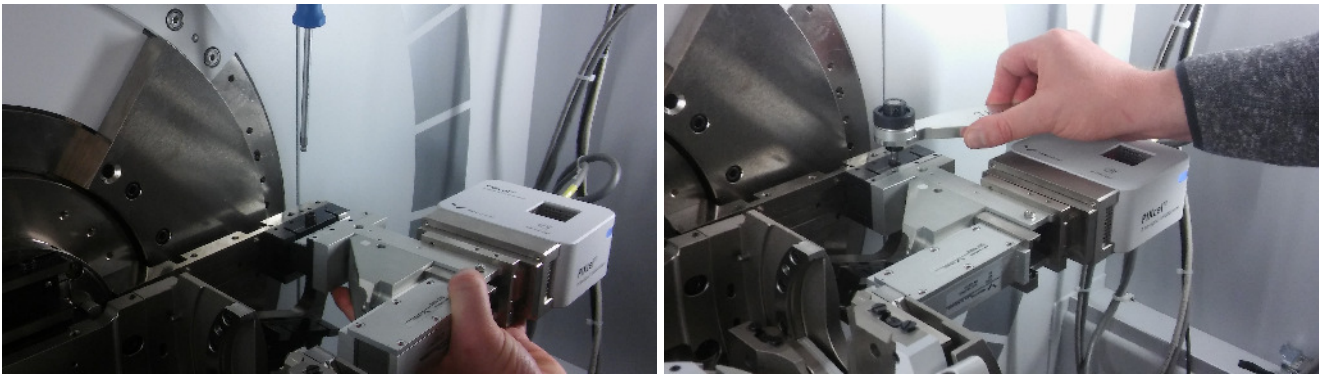


Mount the slid off holder onto the detector using its two hex mounting screws and the pin. Slide the mount and detector back onto the PCC holder.

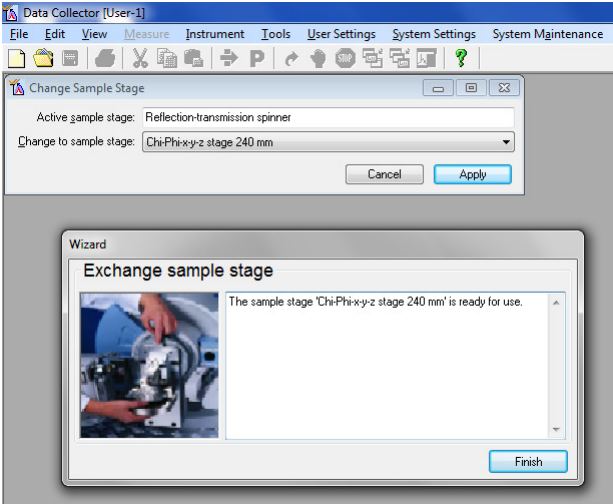


The detector is now ready to be remounted.

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.

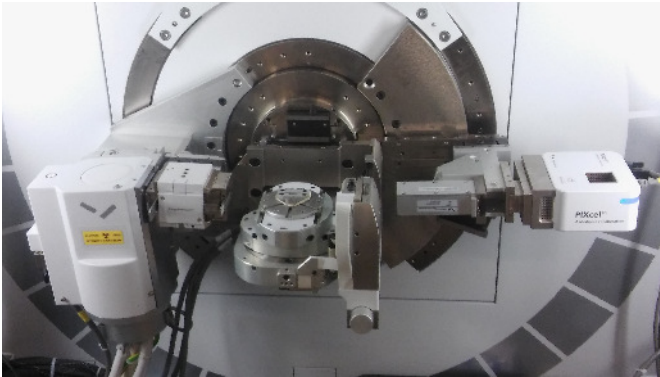


Press the “next” button in Data Collector and wait for the positioning of the instrument to finish.

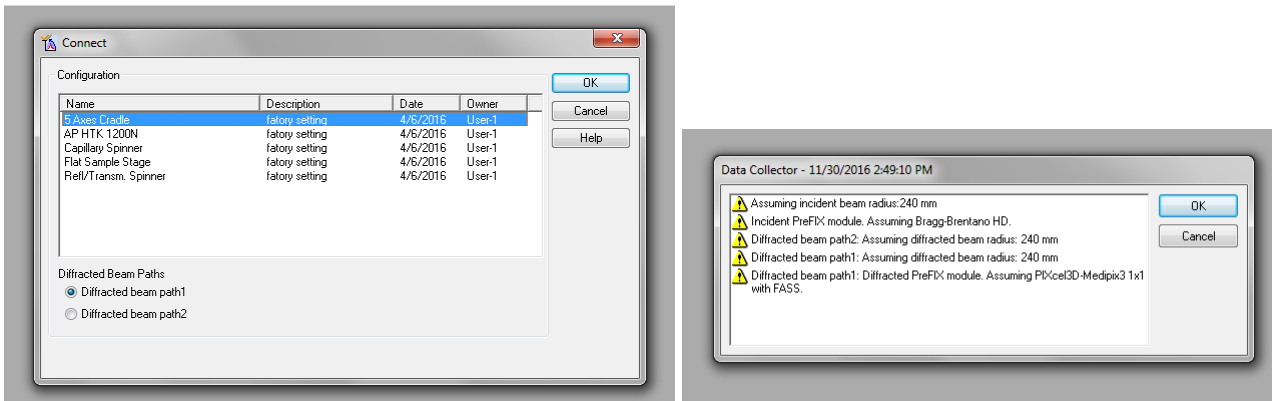


Click the “Finish” button.

The stage, the BBHD and the detector with PCC mount are now in place.

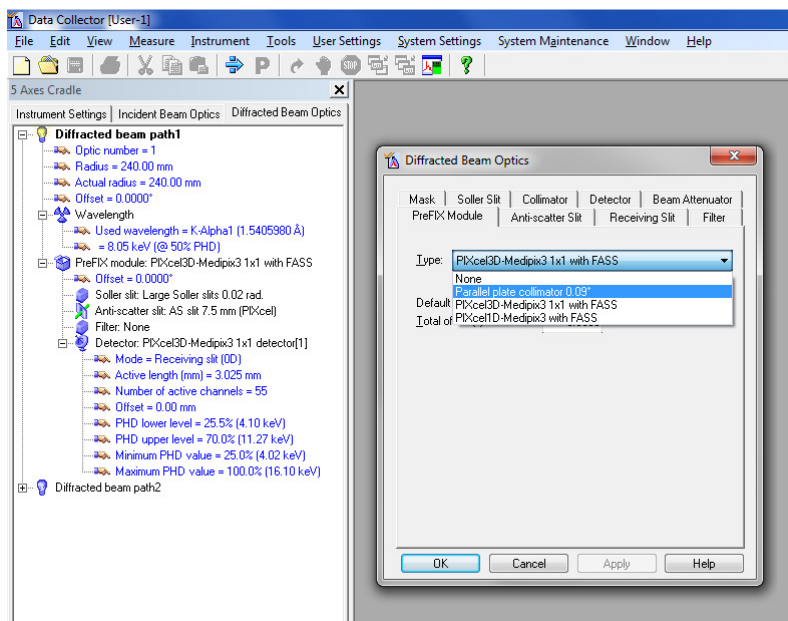


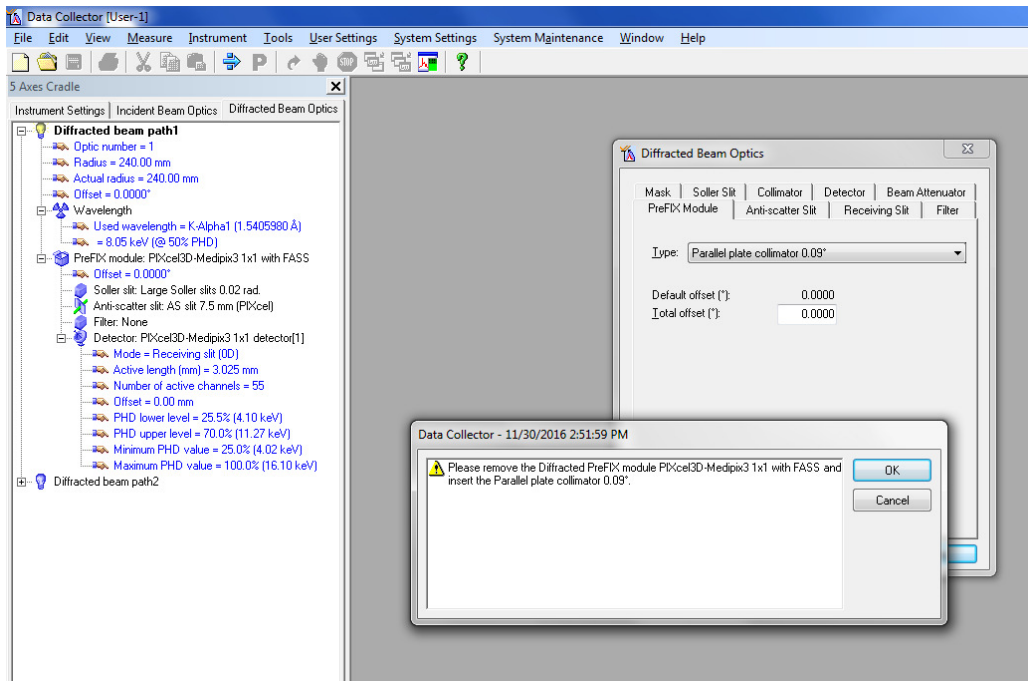
In data collector, go to the instrument tab. Connect the instrument. Select the 5 axis cradle when prompted to select a stage.



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

Update the configuration in Data Collector. On the incident beam path side, for the BBHD, no changes are needed at this time. On the diffracted beam path side, double click the Prefix module line. In the pop-up window change the PreFix Module from “PiXcel3F-Medipix3 1x1 with FASS” to “Parallel plate collimator 0.09°”. Follow the prompts.

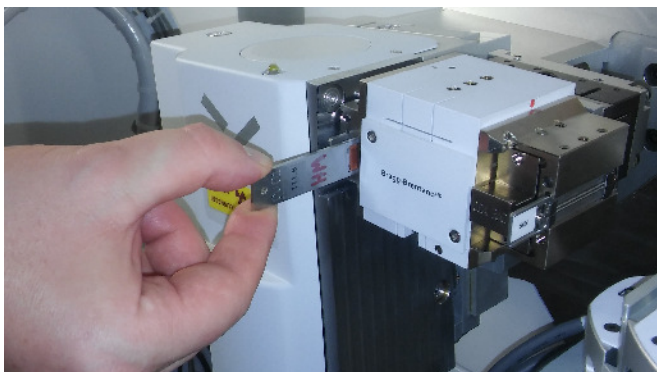
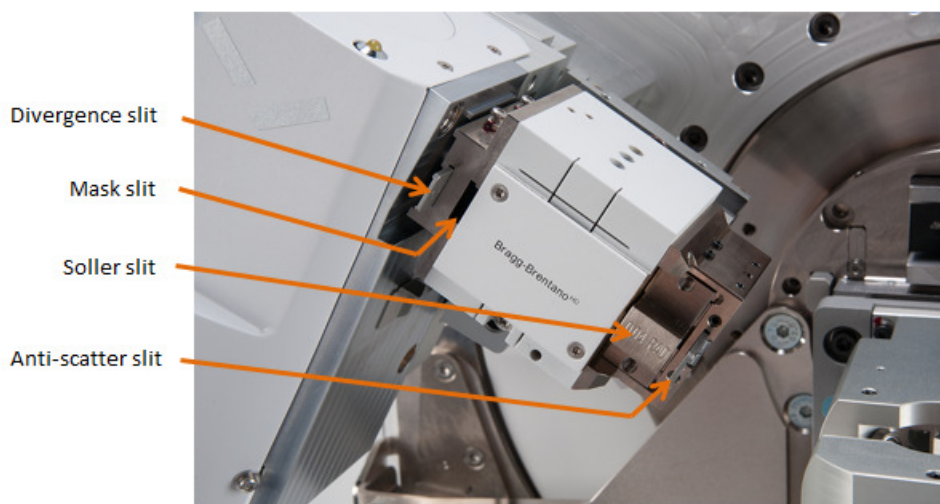




## Automatic Height Alignment for Thin Film measurements:

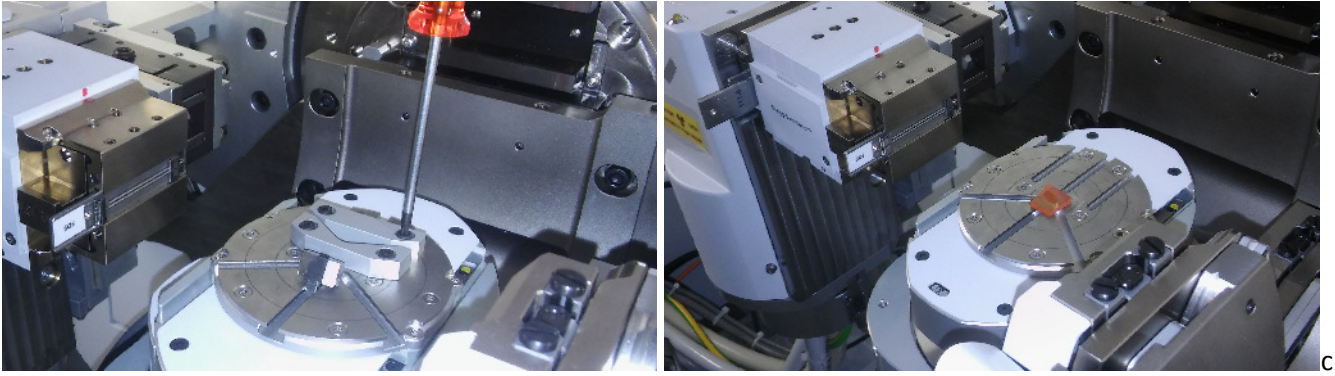
The following configuration is needed for this procedure:

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



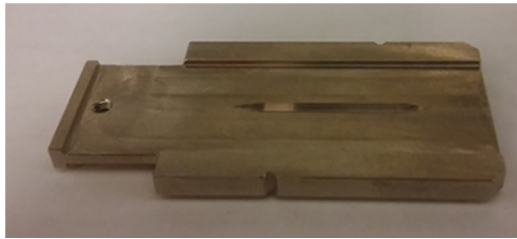
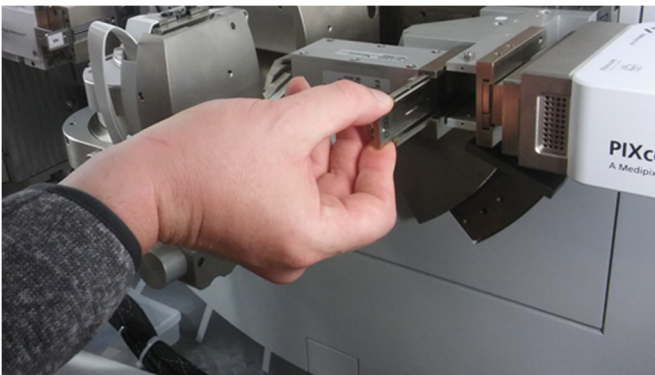
Stage: 5 Axis cradle with your sample mounted.

Remove any sample holders from the surface of the fix axis cradle (if present) using the small red hex screw driver. Fix your sample in place with double sided tape or other measures. The sample has to be centered and be absolutely flat, and as parallel to the stage as possible.



Diffracted beam: PIXel Medipix 3D detector with PCC, 0.09 mm PCC slit, no soller slits, masks or attenuators.

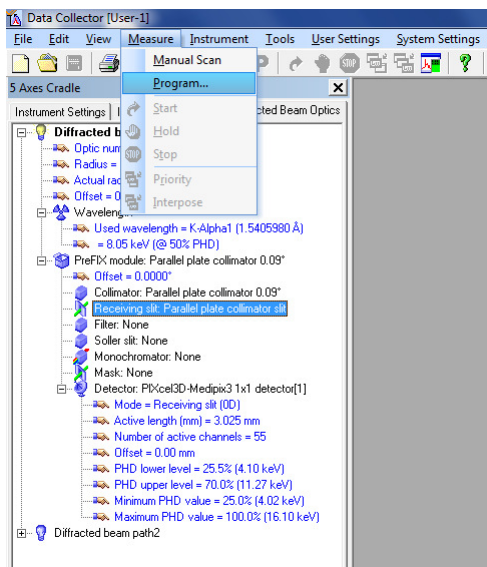
Place the 0.09 mm PCC slit into the slot behind the PCC. Announce that change in Data Collector if it is not already listed.



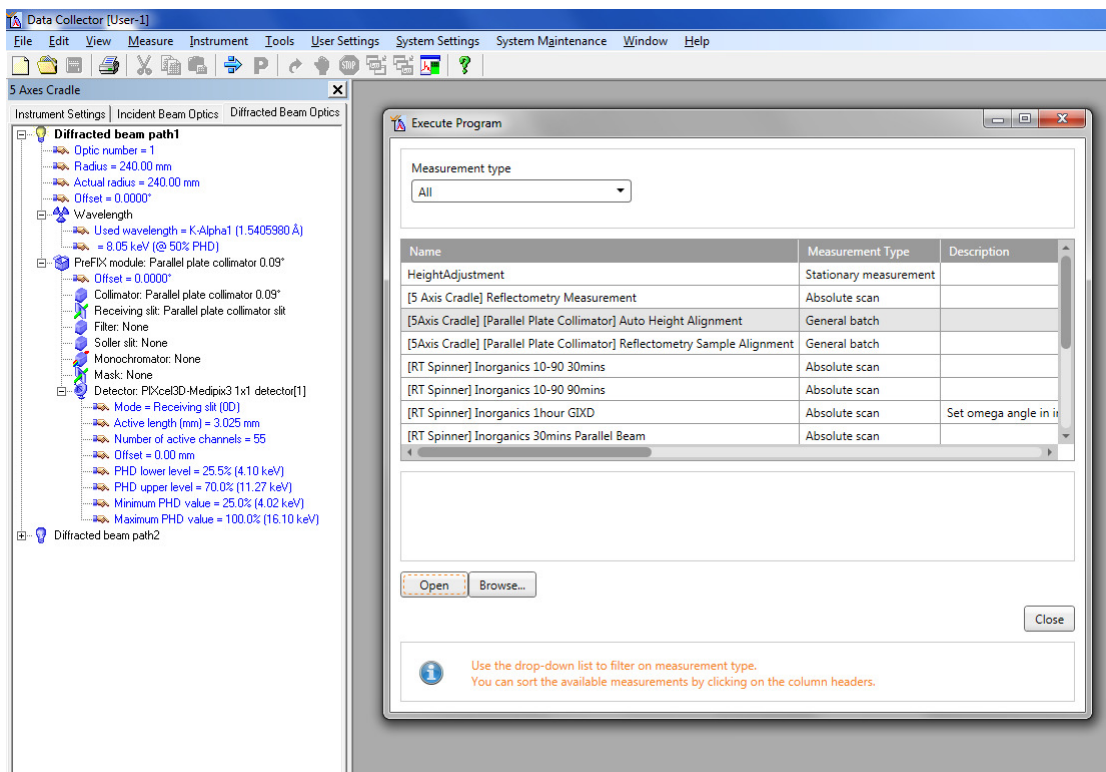
Make these changes if needed and announce them in Data Collector.

In Data Collector, go to the measure tab, program.

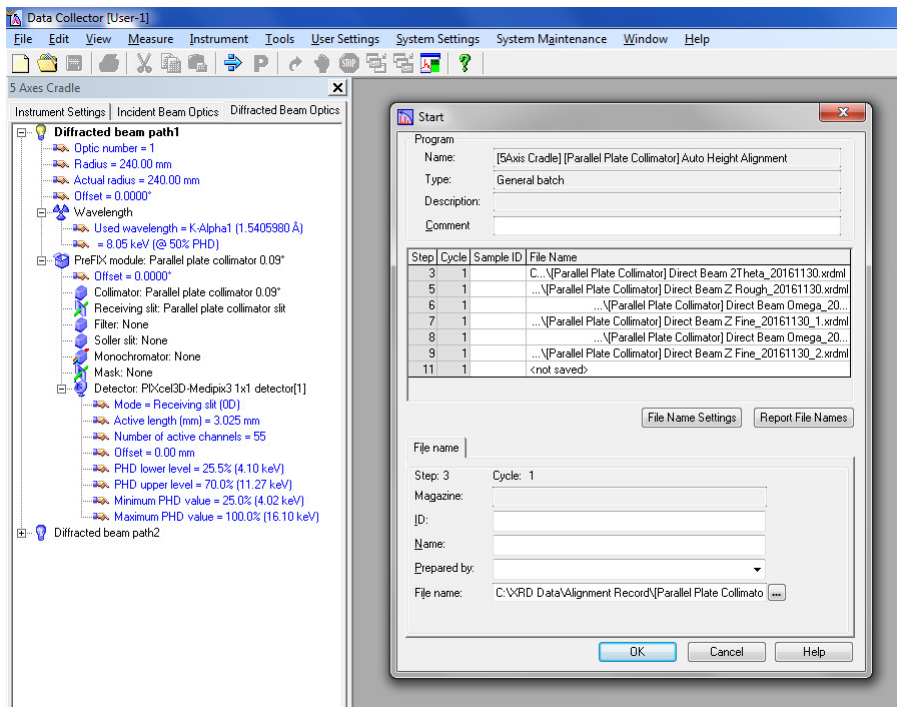




From the menu of programs, select “[5 Axis Cradle][Parallel Plate Collimator] Auto Height Alignment”.

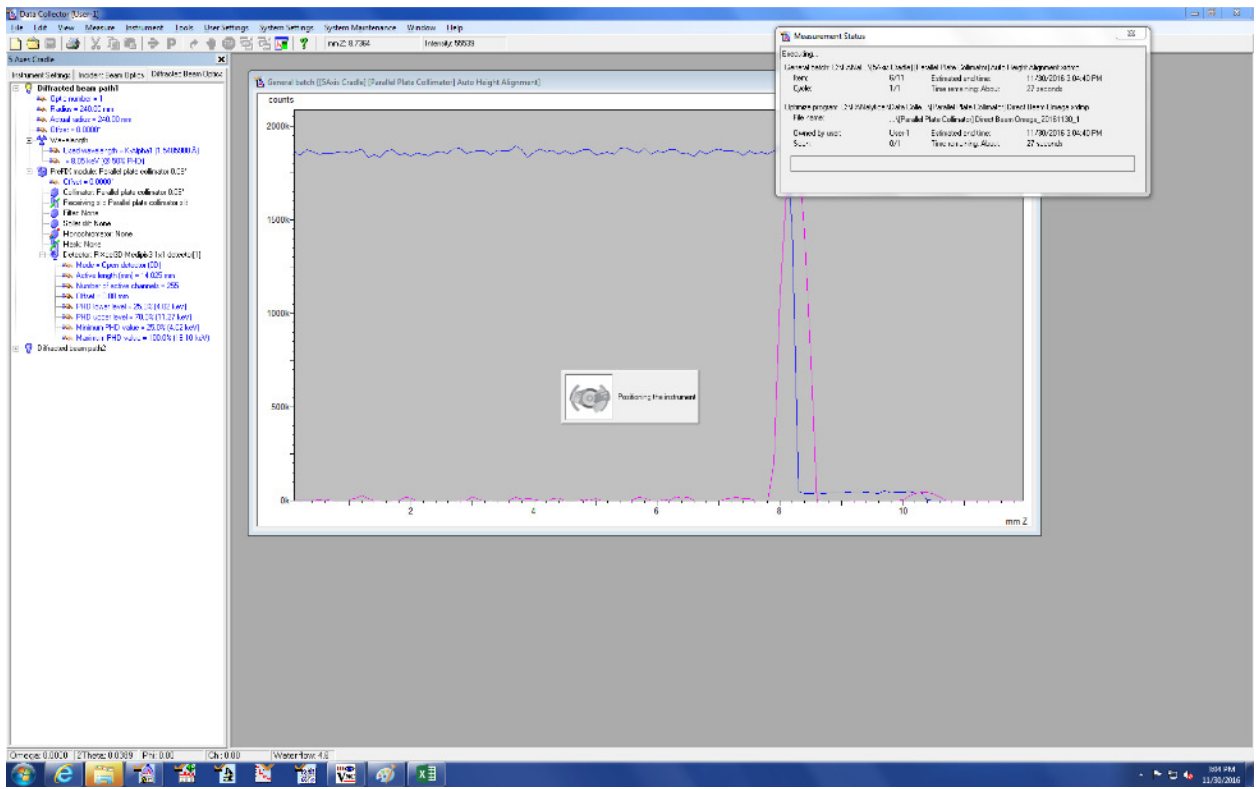
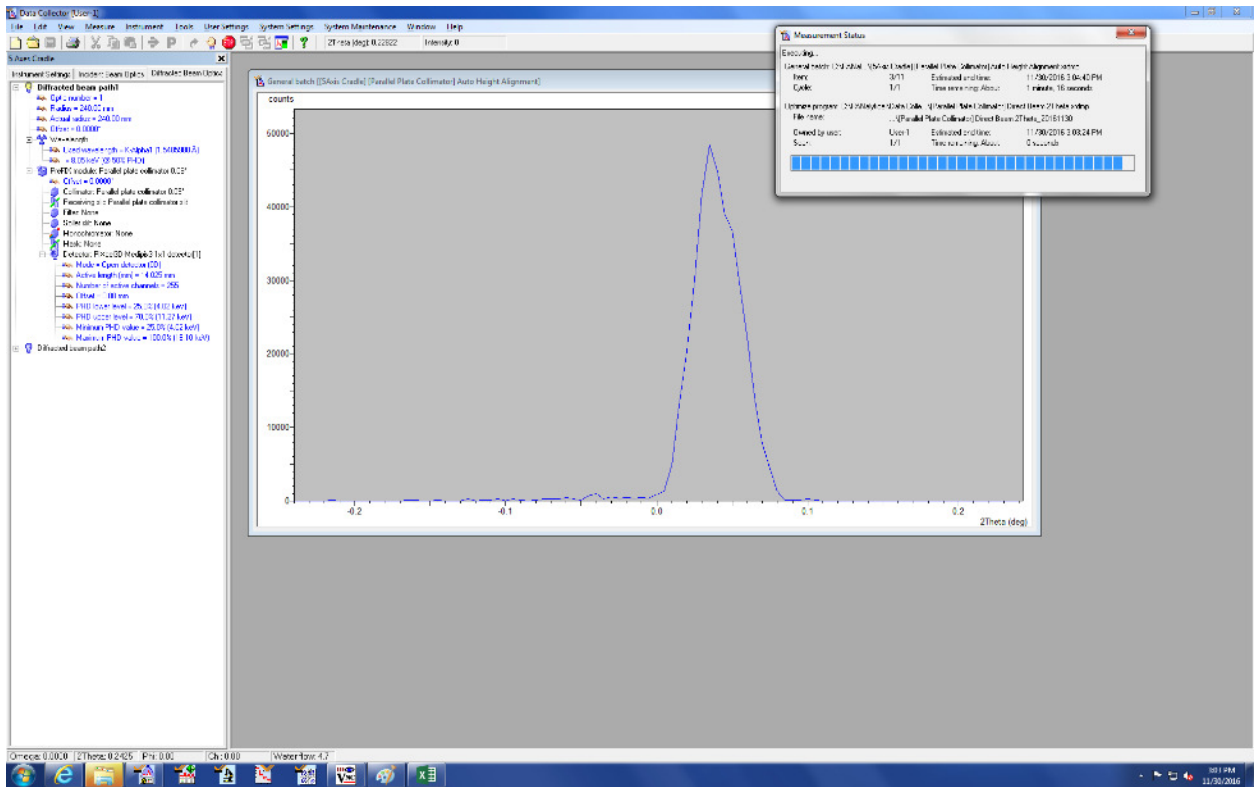


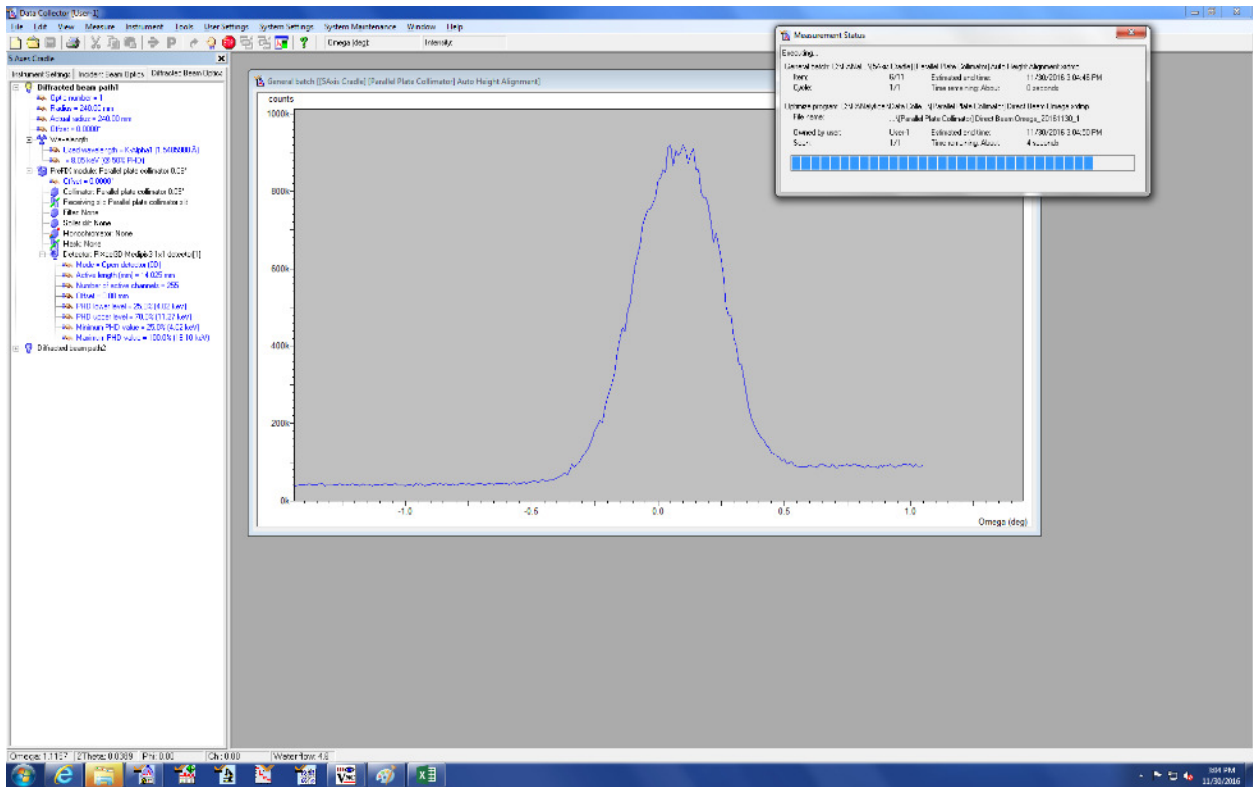
In the Start window that opens, click OK.



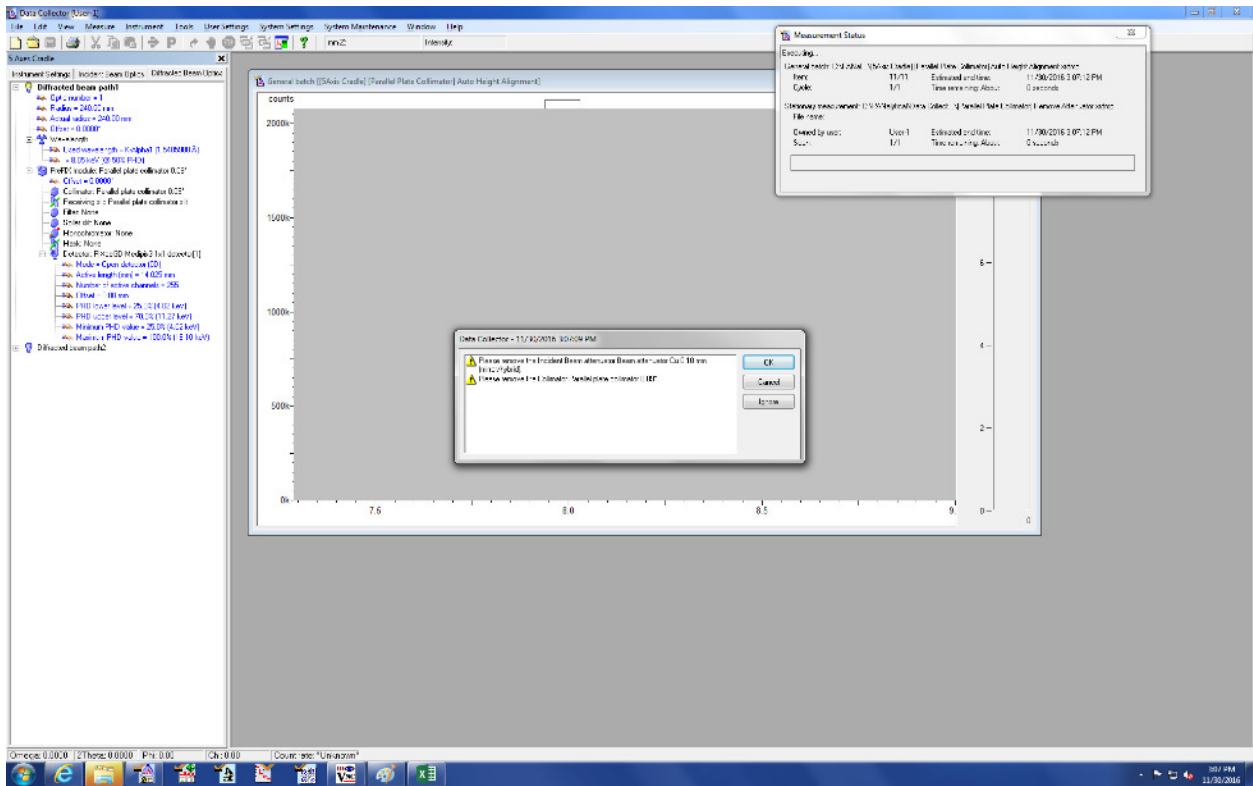
The procedure will scan 2Theta through the direct beam; scan the cradle height (Z) to identify when the beam is cut off by the sample to measure the sample height; scan omega to adjust for a slight angle of the sample wrt to the sample stage. The procedure is repeated automatically for fine adjustments.

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

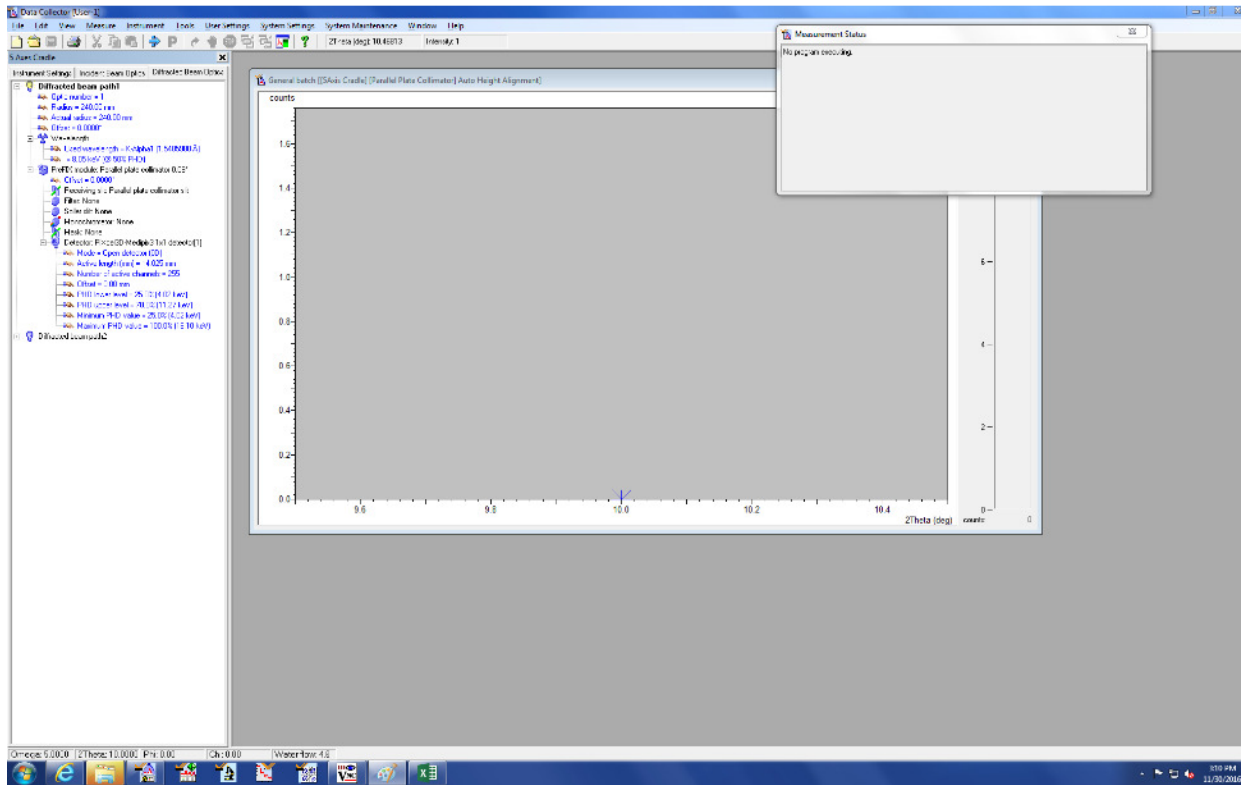




Towards the end of the automatic procedure, the routine will ask to remove the Cu attenuator and the Parallel Plate Collimator Slit. Replace the Cu attenuator by an appropriate mask (not wider than your sample) and remove the PCC slit.



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 for thin films.

Changing the sample stage back to the Reflection Transmission Spinner:

Follow the procedure given above in reverse order. Replace the 5-axis cradle by the Reflection Transmission Spinner, and change the detector from PIXel mounted on the PCC to PIXel mounted on FASS. Place the 5-axis cradle back into the metal cabinet opposite the diffractometer with the mounting side facing back. No alignment procedure is needed for the Reflection Transmission Spinner. When asked whether to use the stored “Fine Calibration Offsets”, choose to not use but to delete them (they apply only to the stage they were measured on).

Thin Film Measurements:

For thin film measurements the 0.09 mm PCC slit can usually be taken out (to gain intensity).

The choice of omega angle depends on the sample. For a thin film on a crystalline support you want to set omega shallow enough such that the beam travels far enough through the thin film that nearly no X-ray intensity reaches the support (or diffraction spots from the support will be visible). The angle thus depends on the thickness of the film, and the absorption coefficient of the film material. Choose an omega angle as high as possible so that you can use wider angles for the divergence slit and the anti-scatter slit (to gain intensity).

Once you have chosen an omega angle calculate the slit sizes for divergence slit and anti-scatter slit using the “BBHD Setup Guide with PIXel” based on your sample length.

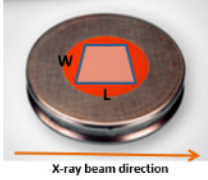
**This Worksheet Shows you how to configure the Bragg Brentano HD for powder measurements with the PIXcel Detector**

**Step 1. Enter the Scan Parameters that you will use**

Start Angle (deg 2Theta)	15	Holder Used (mm)	16
Stop Angle (deg 2Theta)	90	Max. possible L (mm)	11.81
Omega Offset (deg)	0		

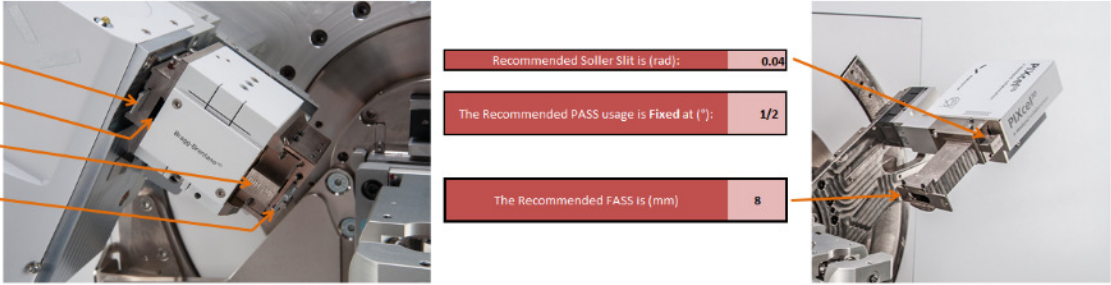
**Check irradiated length, width and area at the sample:**

Maximum irradiated length (at start of scan), L (mm):	12.85	Reduce divergence slit size!
Irradiated width, W (mm):	10.80	
Maximum irradiated area (mm <sup>2</sup> ):	138.7	



**Step 2. Select the divergence slit and mask to irradiate the maximum possible area of the sample without over-irradiating (red text above means over-irradiated).**

Select a divergence slit (°)	1/4	Recommended Soller Slit is (rad):	0.04
Select a mask (mm)	4	The Recommended PASS usage is Fixed at (°):	1/2
Select a soller slits (rad)	0.04	The Recommended FASS is (mm)	8
The Recommended Incident-Beam Anti-Scatter Slit Size is:	1		



**Do not forget to announce these changes in the configuration in Data Collector. Also, remember to set the PIXcel to Scanning Line (1D) Mode with a full (3.34°) active length in your measurement program.**

Choose a mask that is as or less wide than your sample (e.g. 4 or 10 mm).

Drive the instrument to the chosen omega angle.

In Data Collector, go to the measure tab, program. Select one of the “GIXD 5-axis” programs. (if none of the preset programs has the angle or exposure time settings you need, open one of the “GIXD 5-axis” programs, edit it as needed, and save it under a new name. Ask for help if you do not know how to edit programs). The “GIXD 5-axis” programs will use the set omega angle and scan through 2theta as specified.

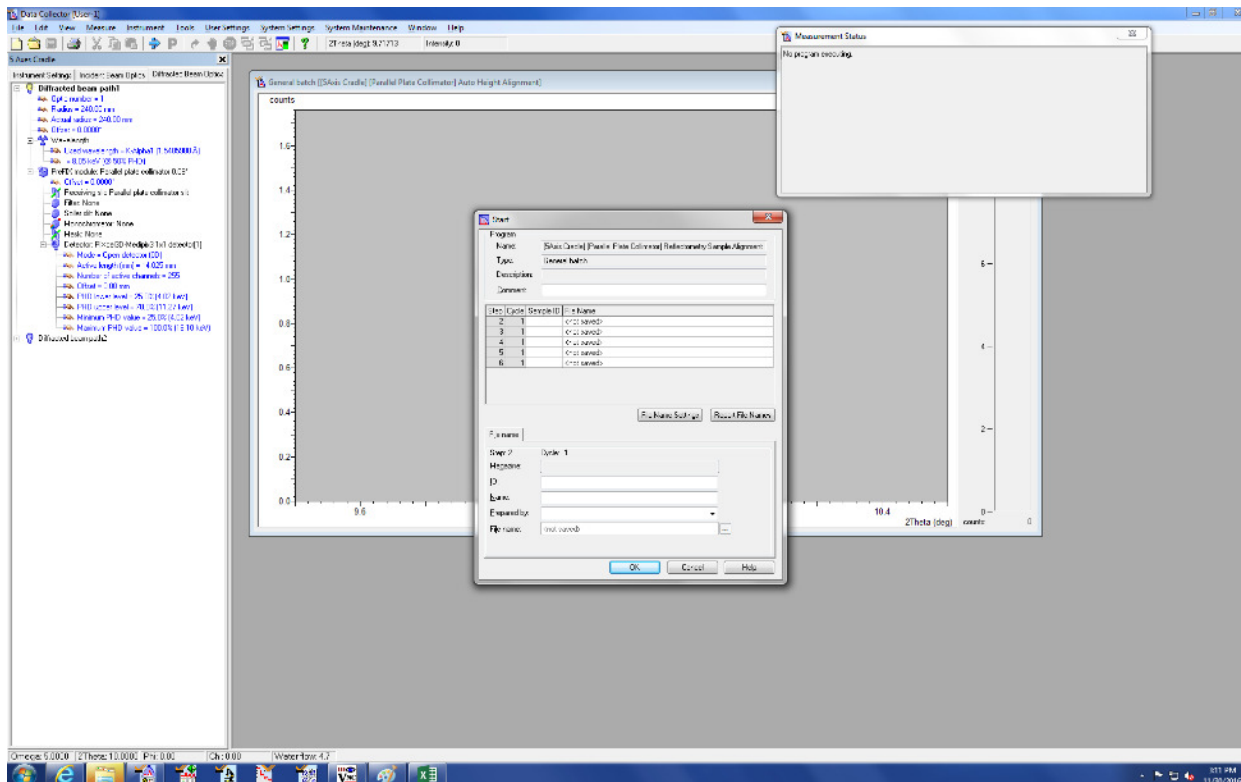
For Reflectivity measurements:

### Reflectometry Sample Alignment:

Skip this step for normal thin film measurements. It will not work for samples not absolutely mirror flat and polished.

The configuration is the same as for the Automatic Height Alignment, but the 0.1 mm Cu attenuator is replaced by a mask. Use a mask that is as or less wide than your sample (e.g. 4 or 10 mm).

In Data Collector, go to the measure tab, program. Select “[5 Axis Cradle][Parallel Plate Collimator] Reflectometry Sample Alignment”.



The procedure will scan omega, chi and omega again to adjust for sample angle and tilt. The procedure is repeated automatically for fine adjustments. The results are written to the software as offset values. Typical values are 0.5° for omega and 5° for Chi.

The instrument is now ready for reflectivity measurements (small angle diffraction).