Institut des Sciences et Ingénierie Chimique (ISIC)

XRD and Surface Analytics Platform

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Standard operating procedures for:
D8 Discover Plus – TXS, BCH 2118
Application: GID – alignment and measurement (update Jan 2025)

GENERAL POINTS TO WATCH:

Remember that the most likely cause of serious problems is collisions between different moving parts of the goniometer and goniometer arms itself (e.g. primary optics and sample stages) and bad handling of the detector. Keep this in mind, protect the detector when you handle it and check with your eyes for potential collisions **before** moving motors.

- **DETECTOR HANDLING**
- **COLLISIONS ON GONIOMETER**

Logins:

Local PC: password
Diffrac: no pwd (user: Lab Manager)

Generator:

The instrument is ready to go when you see a yellow light (top, with a radiation symbol) and green (bottom) light on the left hand side of the enclosure.
Operating power 45 kV / 120 mA (if you cannot stabilize try lower settings, e.g. 40 kV / 80 mA)
Standby power 20 kV / 6mA

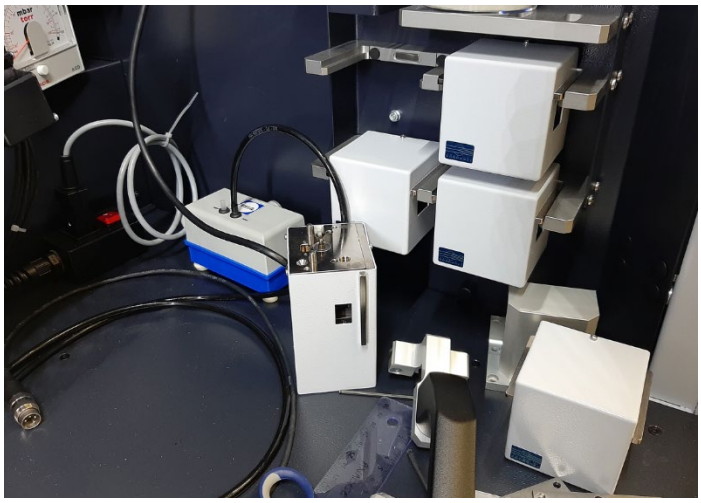
Data and resources:

See https://www.epfl.ch/schools/sb/research/isic/platforms/x-ray-diffraction-and-surface-analytics/

Where to find components:

Primary side optics are stored in the racks on the left hand side of the enclosure, or on the floor of the enclosure. This includes the Goebel mirror, Soller slits, collimators (single and double pinhole, nozzle), slits and masks.

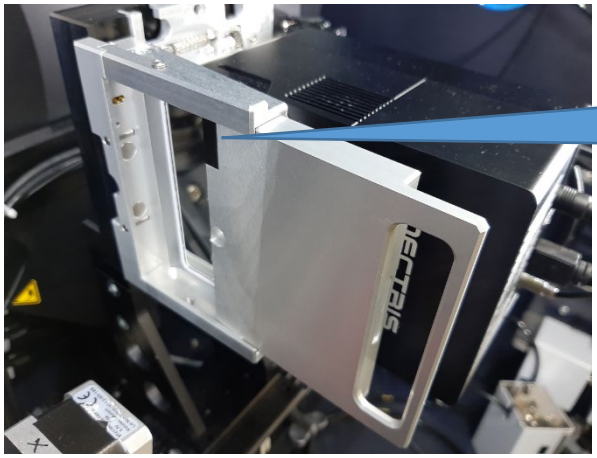
Secondary side optics and covers/optics for detector are stored on the right-hand side.



Sample holders, tools:
Sample holders and tools/material for sample preparation are found either in the enclosure or in the cupboard to the left of the fume hood. Clean after you leave, and place back where you took from.

General handling of the Detector

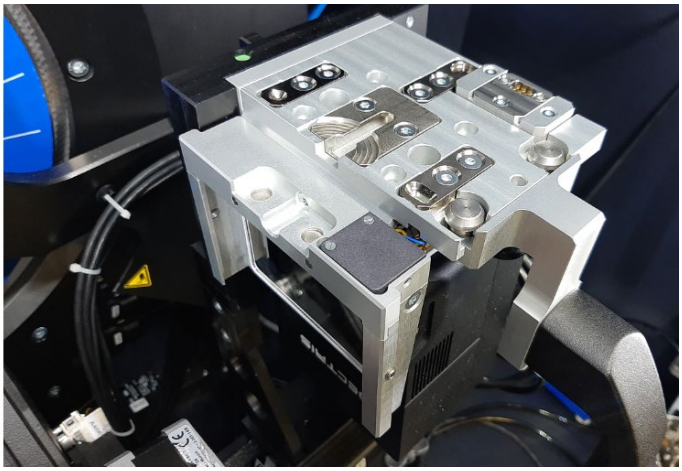
- **Avoid damage to the Detector:**
- NEVER touch the detector window.
- When moving along the track or unmounting the detector first move the detector (2θ) arm to a position where this is practical, e.g. 0 °2θ.
- If you need to mount/unmount the detector to change its orientation see procedure below. ALWAYS PLACE THE PROTECTIVE COVER when unmounting the detector.



- In the control software, do not move the detector with anything other than drive "detector".
- Pay attention and be ready to stop any stage movement that gets too close to the detector or collimator, by clicking on "stop" in the Diffrac-Commander or by opening the doors of the enclosure.

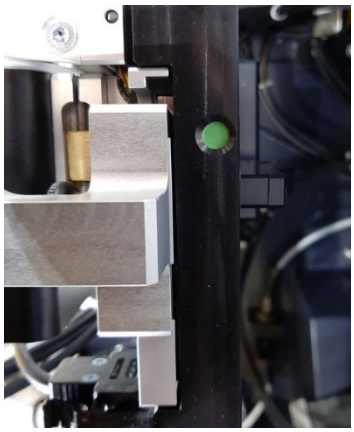
Changing orientation of the Eiger2: You will only need to do this if the detector is mounted in gamma-optimized orientation when you arrive, you need to change it to 2Theta optimized orientation for GID. The detector is mounted on the arm by means of a latch mechanism (backside of arm) and a magnet (front side of arm).

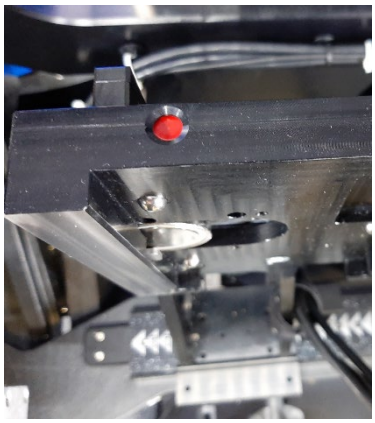
A. ALWAYS PLACE THE PROTECTIVE COVER FIRST.
B. Insert handle (stored at right hand side inside enclosure) to hold on to the detector.
C. Hold on to the handle (be prepared to hold the weight of the detector upon unfastening the latch behind the track).



D. Unfasten the latch on the back of the track by pulling it the left with your fingers. The little circle shows green when it is locked and red when it is free. CAREFUL. When you have unfastened you will feel weight on the handle, the magnet supports the detector but does not hold it in place.

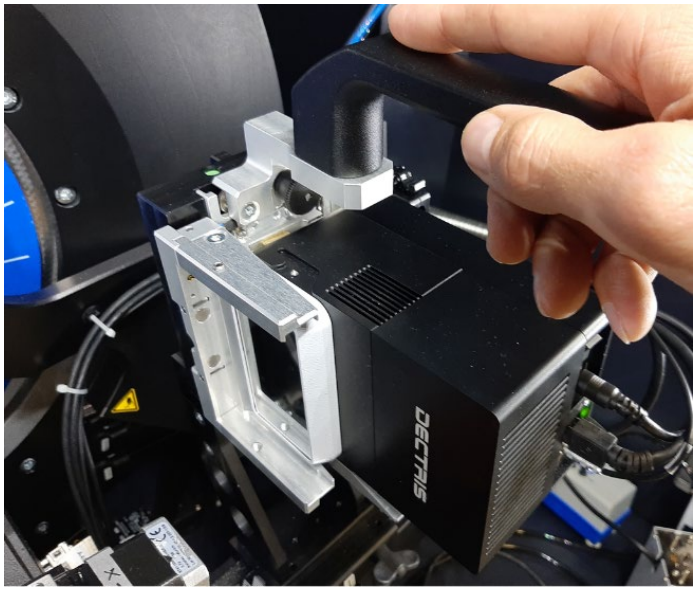
   To unfasten you need to use your left hand and pull the lever to the left until the circle becomes red. It can take some time getting used to it, it can help pushing the handle upwards a little.





E. Mount detector again in rotated position and fasten latch. The detector orientation is now 2Theta-optimized (see below, long dimension parallel to scanning direction)

F. **Remove handle from detector before measurement** and place at the right in enclosure.

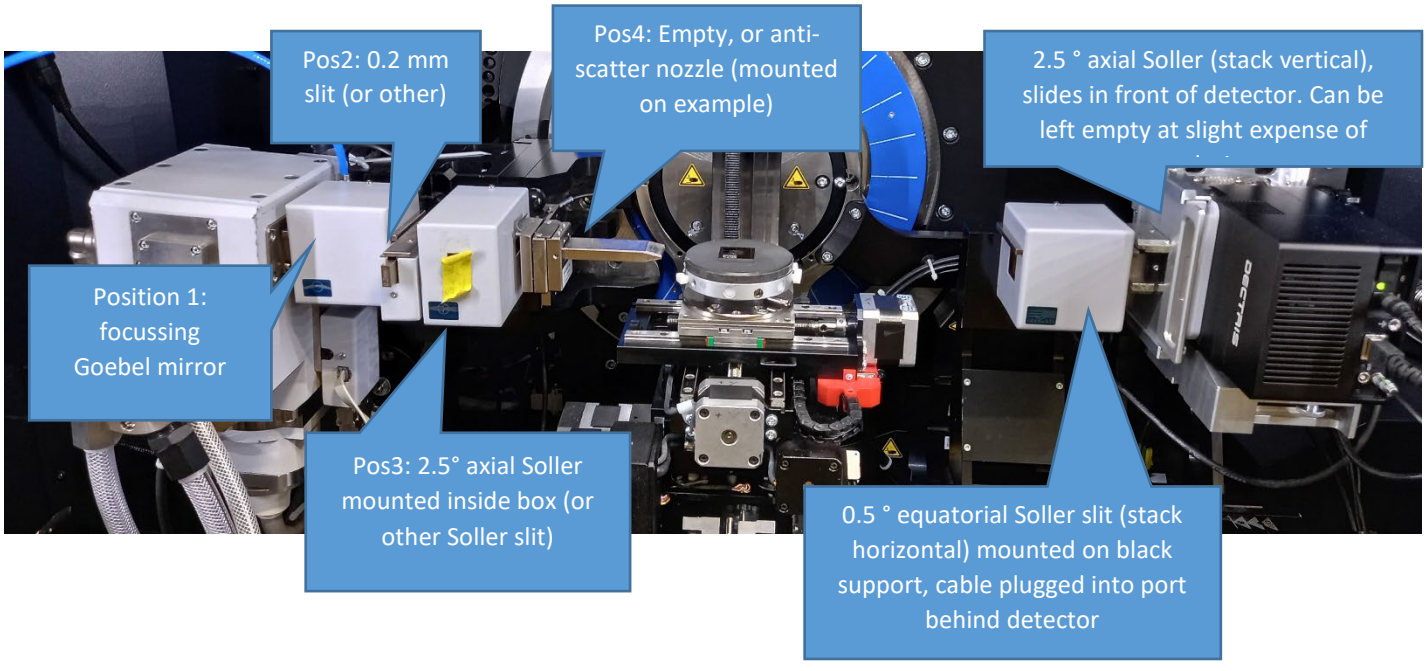


GID MEASUREMENT: Alignment (alignment identical for In Plane-GID)

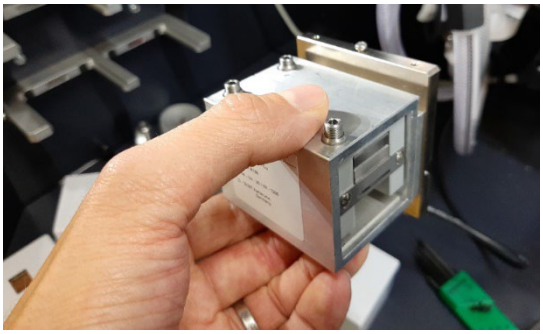
Note: sample alignment can be automated by using a predefined script when launching the measurements. However, manual alignment is usually faster and possible problems can be spotted quicker. It is suggested to align manually, and when screening multiple samples saving aligned motor positions for each sample into a job file. If you want use automated alignment and do not know how to please get in touch with the facility.

Before mounting components and preparing the instrument configuration for GIWAXS move both the θ- and 2θ-arms to positions where this is easily done, i.e. not too high, if they are placed in impractical positions when you arrive. If the previous user has measured a Theta-2Theta scan e.g. to 120 °2θ the position of the 2 arms is not practical to exchange components.

1. **Mount optics required for GID.** Besides the required focusing Goebel mirror, it is up to the user to decide upon optics. This will depend on incidence angle, film thickness, desired probing depth and scattering behavior. If you want to calculate different parameters for yourself, use e.g. https://gixa.ati.tuwien.ac.at/tools/penetrationdepth.xhtml
2. A good general all-purpose combination can be (mounted from mirror to sample):
    - 0.2 mm selection slit (smaller for really low incidence angle, larger for higher). When mounting it you should be able to read the labelling by tilting your head to the left. The slit is not symmetric!
    - 2.5° axiall Soller slit mounted and covered by white box (the one with the cutaway and a yellow sticker on it). Other Soller slits are available if the sample is very crystalline and you require higher resolution (usually not the case amongst our user pool).
    - Antiscatter nozzle, or empty. If nozzle mounted watch collision when using the 4” wafer chuck (not mounted in figure), move the stage away from the primary track in that case.



2.1 **Mount the Goebel mirror**, if not mounted already. Handle the mirror CAREFULLY and do NOT LET FALL (shown below without white protective box). If you do let it fall please inform the facility immediately, as realignment is maybe necessary. If you fail to inform then it will be difficult to associate intensity decrease with a certain event.



Touch the mirror at top and bottom only, and carefully fasten it with the snap-lock mechanism on the track, as all other optics that you mount directly on the track (those in white boxes).

2.2 **Slide the 0.2 mm (or other) into pos2, after the mirror**, gently making sure it is all the way in.

2.3 **Mount the 2.5° ax Soller slit and cover it with white box with cutaway (usually yellow sticker on front).** You may need to remove the monochromator if it is mounted, be careful and place it in the rack on left if this is the case.

2.4 **Mount the anti-scatter Nozzle (optional)**. The nozzle has a line-shaped opening, do not take the collimators (shown in images below, which can be mounted in the same position). **Handle it only by the base**, not touching the rectangular tubing. It is mounted magnetically and will fit well only when it is in the correct position.

2.5 **Mount the optics holder for the secondary side.** GID measurements will require an equatorial Soller slit (usually 0.5°) which needs to be placed in front of the detector. You will need to mount the black support on the dove-tail track which the detector mount is fixed to as well. The black support is in the cupboard next to the fumehood. Mount it in front of the detector on the track, push it up against the detector, fasten its butterfly screws and connect the cable it to the empty port behind the detector. Leave it empty during alignment, and mount the Soller before measurement.

3. Prepare sample mount.

The sample can be mounted on different stages. (i) The small thin film stage (plastic cone mounted on base), (ii) the small wafer chuck and (iii) the large wafer chuck. If you use the large wafer chuck and want to use the anti-scatter nozzle (or collimators) at the same time you will need to move the sample stage toward the right, to avoid collision.

The measurement height is 40 mm!! Your sample needs to be mounted within approx. 40 mm +/- 1 mm before starting alignment. This is due to the motor range of z: -1 … 1.8 mm.

If using the wafer chucks use spacer plates 1,2 or 3 mm to be able to mount sample within the suggested height range (there is a caliper on the table or inside the enclosure). If you are measuring a single sample without the nozzle then motors x,y should/can be at zero.

4. Start alignment:

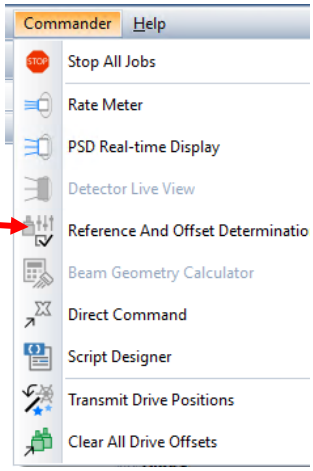
Note: The entire machine and sample alignment (usually 4 steps) is done with a 0D detector choosing a small line aperture, e.g. 200 x 1 pixels. No optics should be mounted in front of the detector, the Cu attenuator can be slid into the mount on the detector itself if you are unsure about saturation. But, the detector is radiation hard and does not need an absorber, even if it can be useful to mount it.

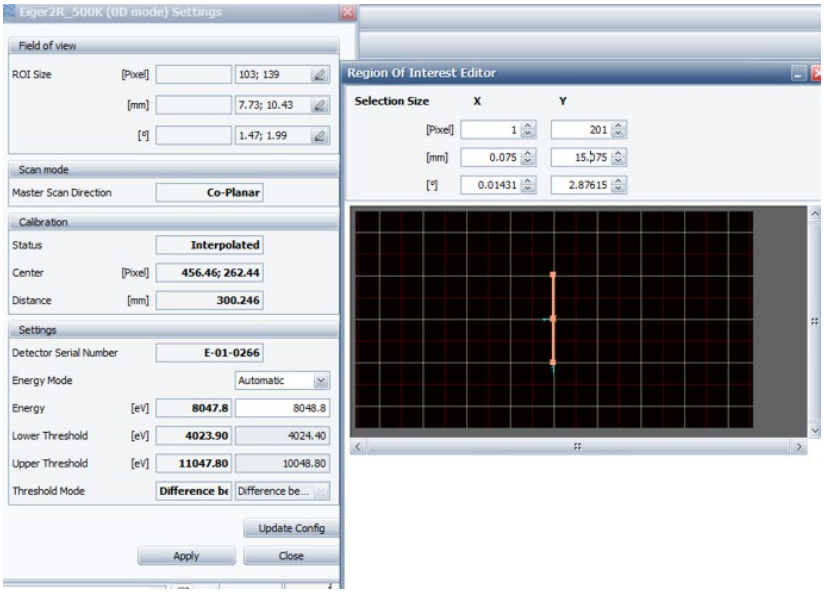
4.1 Step 1. Reference 2Theta scale to 0°: 2T-scan

In Diffrac chose the application mode "Powder Diffraction", unless you want to use an alignment script.

In Commander, click on “clear all drive offsets”. This removes potential offsets from the previous alignment (other user). Move the motor z to -1.0.

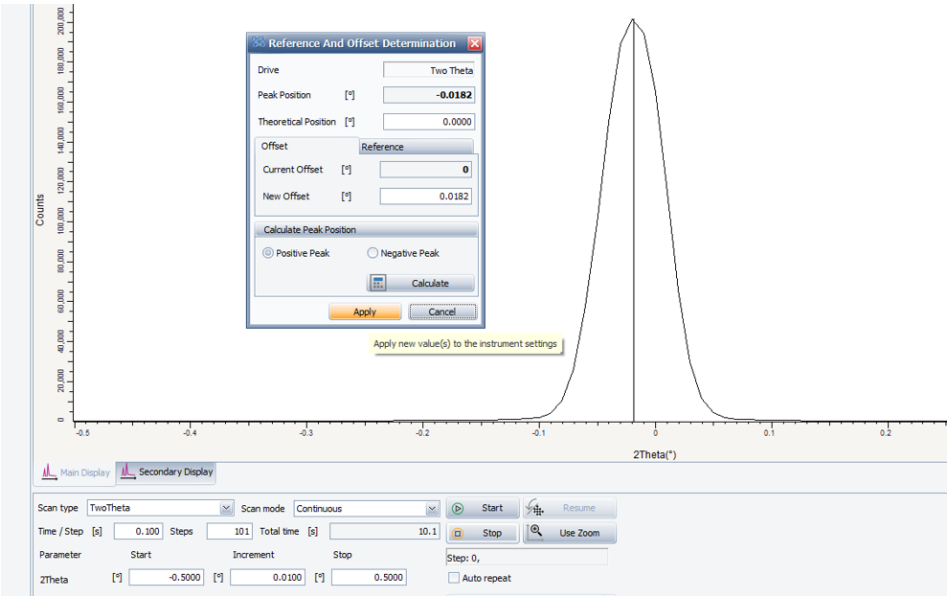
Select the 0D detector from the dropdown menu and chose a region of interest (ROI = active area) of about 200 x 1 pixels (the line-shaped ROI should be parallel to the short dimension of the detector, i.e. perpendicular to scanning direction)





Make sure there is nothing in the beam path (you can remove the sample or move it aside to be sure).

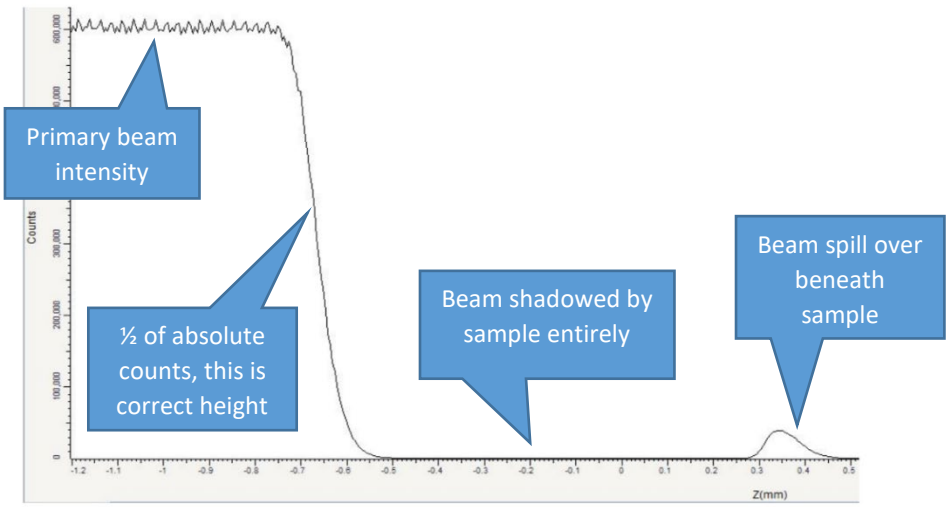
Move motor theta to 0°. Select 2Theta scan from -0.5…0.5, step size 0.01°, time per step 0.1 s. Perform the 2Theta scan. You should see a clear sharp peak. If this is not the case check your ROI is as described above, the opening in scan direction defines the resolution and should be small. In the Commander tab (where you deleted offsets), click on “Reference and Offset Determination”. This will mark a line on the peak maximum. Apply this offset once, then cancel the window. If you want to check that your offset has been registered you can click on the icon “additional settings” next to the setpoint window of the respective motor.



4.2 **Step 2. First height alignment: z-scan 0. Find height were sample shadows the beam by half.**

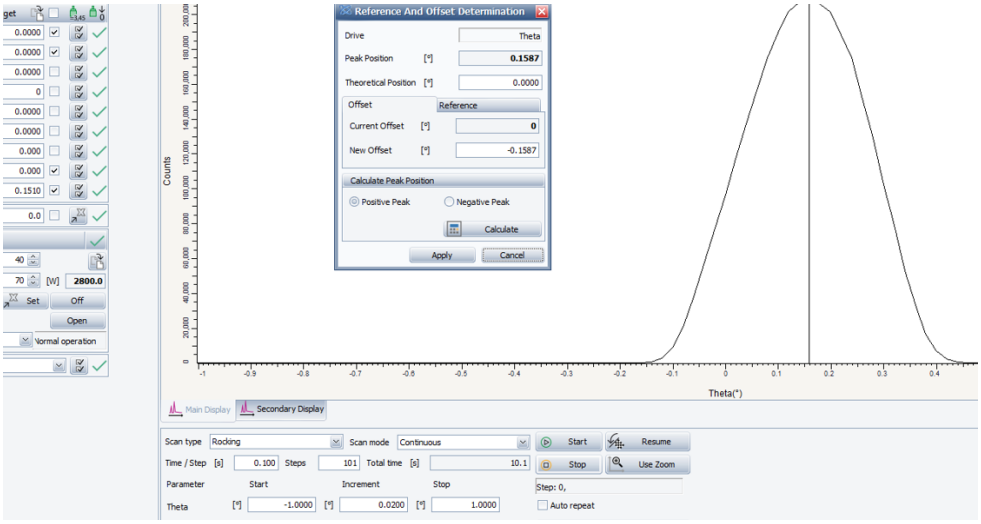
Note: the range of z is -1.0 … 1.8 mm. **Do not move outside this range**, even if the software may allow it. If you cannot align with this max range, manually adjust the height of your sample. Stepsize 0.01 °, time/step 0.1s.

Ensure that 2Theta and Theta are both at 0° or move them to 0 after the preceding 2T scan. Mount sample or move into center of stage if not yet the case. Perform a z-scan from -1 to 1.8 mm. You will see the z-scan, which represent the absorption of the sample. It should start with a plateau (if not your sample is too high at z = -1.0 mm or your 2Theta drive is not at 0°), then a step-like drop as the sample passes through the beam, then zero intensity, you may observe another weak peak if the beam passes below the sample during the scan. Calculate the half of the primary beam intensity (first 3-4 digits is enough) and search for the z-value corresponding on the slope with your mouse cursor, zooming into the scan. Double click on the corresponding point in the curve, this takes over the new z-value as set-point.



4.3 Step 3: reference theta (omega) scale to 0°: Rocking Curve RC. This aligns your sample surface with the X-ray beam to be collinear.

At the new height determined in step 2 perform a rocking curve.
Chose RC from scan type, scan from -1 to 1 in theta (or omega depending which application type you are in). Step-size 0.03°, time/step 0.1s. You should see a scan that reminds you of a triangle, it is sometimes asymmetric depending on current sample mis-tilt or also sample shape. Sometimes you may see just a broad peak if the sample mounting is very far from the reference of omega (i.e. there is a considerable sample tilt). The image below show such an example.
When you see the RC, as in step 1 (2Theta scan) go to the commander tab and click on “Reference and Offset Determination”. A line will appear on the RC which you can accept, or if far away from the maximum, double click on the maximum. Then apply the new offset in the appearing window and cancel the window.



4.4 Step 4. Second height alignment: z-scan 0. Find height were sample shadows the beam by half.

Repeat step 2.

After step 4 alignment should be finished. If you are unsure you can always iterate over the RC (step 3) and a height-scan. The RC should look sharp when the sample is parallel to the beam, and the z-scan should no longer move between iteration z-scan – RC. In the very most of times the 4 steps described are enough though.

5. Start measurement

Note: when the global alignment (previous 4 steps) has been done it should be enough to change samples with your hand, without further alignment before measurement, provided you do not change the setup and that the substrates are the same thickness.
If you want to screen multiple samples then you should perform, after the previous 4 steps, one z-scan per sample. The determined height should then be entered, along with the sample's xy positions, in the respective tab of the job file of the experiment.

Remove the Cu absorber from in front of the detector if you were using it for alignment.

Place an equatorial Soller slit (usually 0.5 °) into the black support in front of the detector. If purposeful you can add a 2.5° axial Soller slit, which is mounted in an Al frame (found on the right-hand side of the enclosure) slides into the front of the detector (where the absorber goes).

Adjust ROI. You need to open the active area, otherwise you will be measuring with the 200 x 1 pixel alignment window. Chose an area of 100x200 …. 200x200.
If you expect low angle signals it can be useful to mount the anti-scatter screen (found in the cupboard next to the fume hood) into the goniometer and manually bring it down until very close above the sample. Watch for collisions in that case, ensure your sample(s) is at the correct previously determined height before bringing down the anti-scatter screen manually.

Set incidence angle(s). Chose angle depending on layer thickness, penetration depth required, etc. If you notice during the measurements that your incidence angle may require a change in divergence slit (position 2 om primary optics side), then this is ok. The slits are all factory-aligned and can be exchanged without further alignment scans. You can also set a series of incidence angles into a job file, this allows you to choose the best data once collected, and also provides you with a depth profile.

Set scan parameters. Chose Two-Theta scan and take a look at a region where you expect a peak. GID measurement are low resolution, you can assume that a step size of 0.05 ° 2T is small enough. However, you should measure several seconds (up to 10s per step for thin organic layers) per step. The incidence angle can be further adjusted based on peak intensity before measurement, if wanted.

Start scan(s).