Institut des Sciences et Ingénierie Chimique (ISIC)

EPFL

XRD and Surface Analytics Platform

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Standard operating procedures for:
D8 Discover Plus – TXS, BCH 2118
Application: 2D-GIWAXS

GENERAL POINTS TO WATCH:

Remember that the most likely cause of serious problems are collisions between different moving parts of the goniometer and 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: Bruker
Diffrac: no pwd

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 50 kV / 105 mA
Standby power 20 kV / 6mA

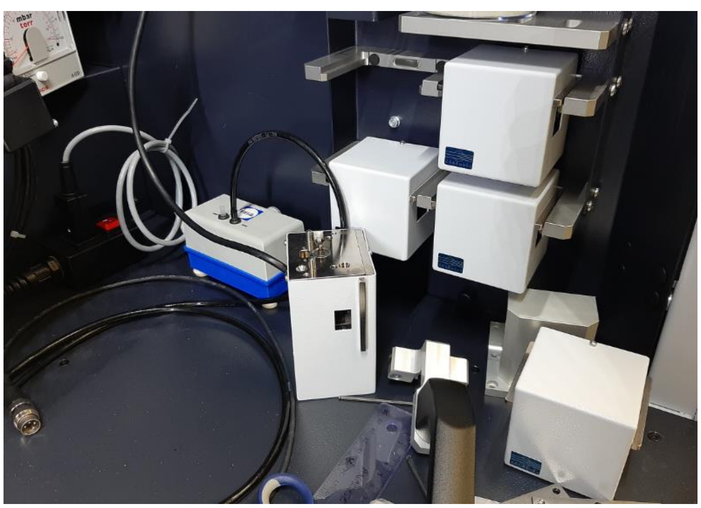
Data and resources:

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

<u>Where to find components:</u>

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), slits and masks.

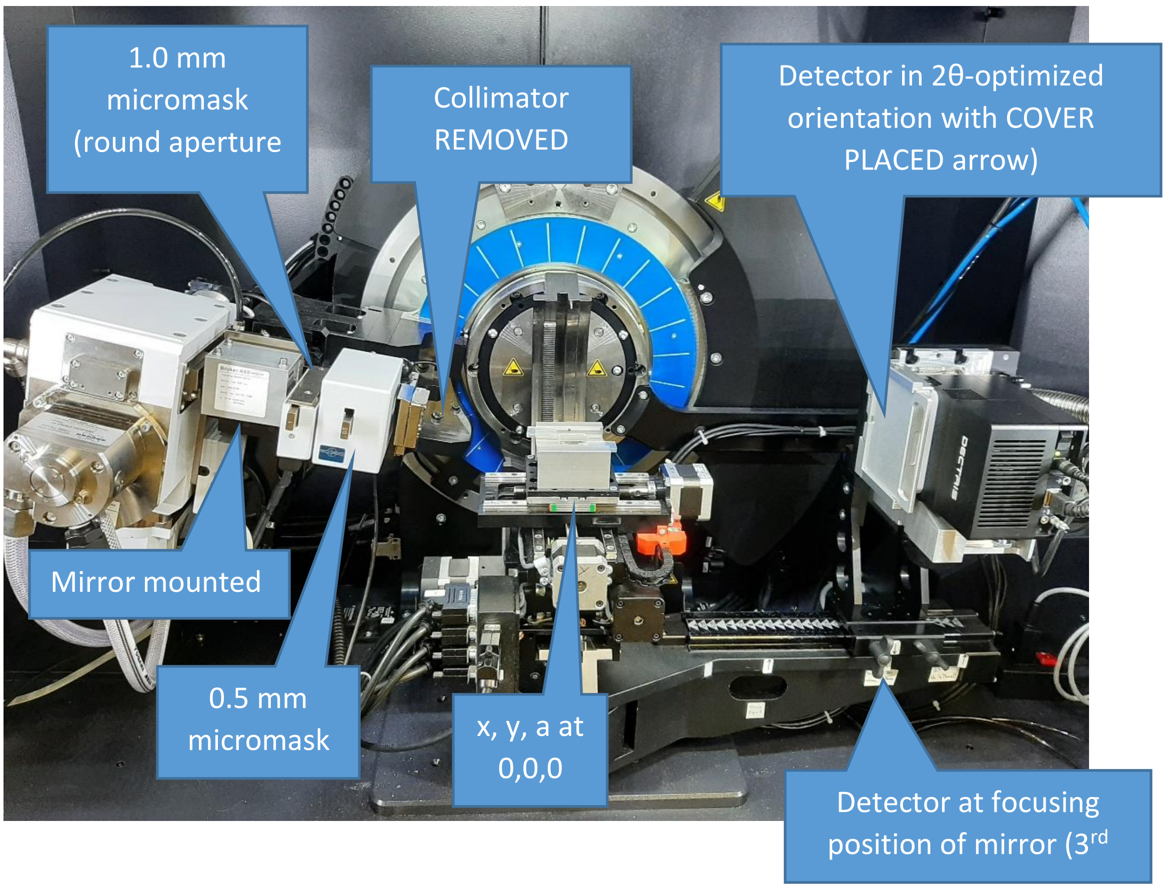
Secondary side optics and covers/optics for detector are stored on the right hand side. On the floor you will find sollers (in and out-of-plane) and the detector handle. On the wall you will find inserts to place in front of the detector, such as the protective cover, axial Soller slits, the evacuated beam path... (the beam stop is stored in the cupboard).



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.

This is the configuration you should find and leave the instrument in:

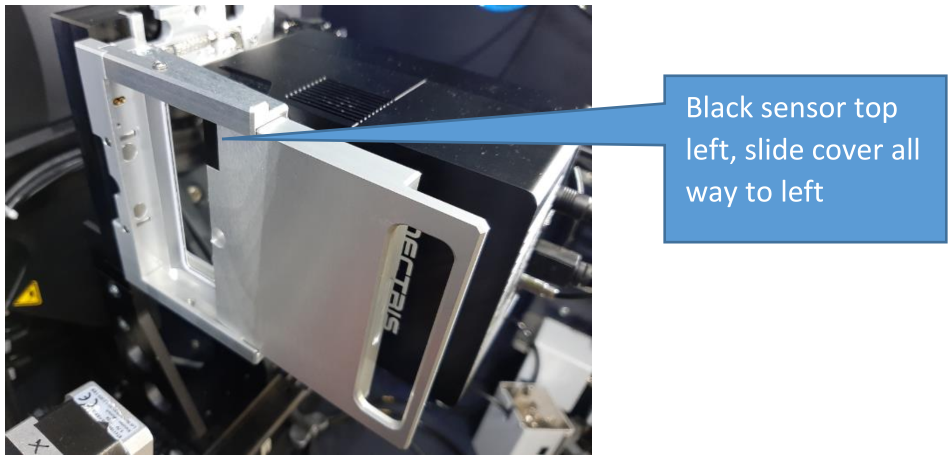
- **Please take care you mount/unmount all optics and move motors to the indicated positions:**
- Optics:
  - Goebel Mirror mounted (take care about not dropping, nor touching with fingers on mirror, See point 1.1 of the manual)
  - 1.0 mm micromask on position after mirror
  - 0.5 mm micromask on next position
  - Collimator position EMPTY!!! (place collimator handling on base only, see 1.3, on shelf on left)
  - Detector cover placed properly (see below)



- Motor positions (IN THIS SEQUENCE!):
- Move motors ONE AT A TIME!
- Before moving into positions after measurement slot remove the collimator if there is any (see above) and place the detector cover. Then move:
  - Theta at 10 deg (move in Diffrac, watch collisions!!!)
  - Detector at 0 deg (move in Diffrac, watch collisions)
  - Detector distance at dd_min label on dovetail track (manually)
  - Phi to 0 deg (in Diffrac)
  - x,y,z at 0,0,0

General handling of the Detector

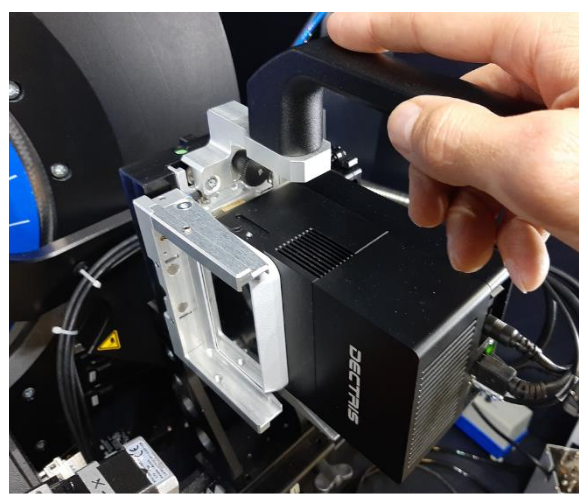
- **Do not damage the Detector:**
- NEVER touch the detector window.
- When moving, unmounting the detector first move the detector (2θ) arm to a position where this is practical, e.g. 10-20 °2θ.
- If you need to mount/unmount the detector to change its orientation see procedure below. ALWAYS PLACE THE 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.

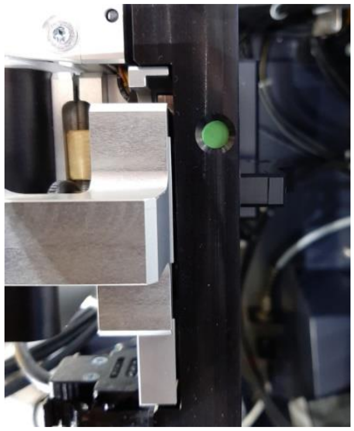
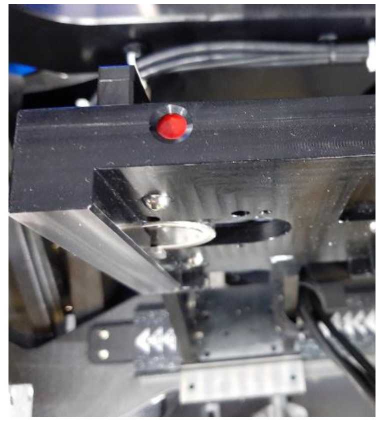
Changing orientation of the Eiger2: You will only need to do this for gamma-optimized images/scans and when you want to measure a full quadrant.
The detector is mounted on the arm by means of a latch mechanism (backside of arm) and a magnet (front side of arm).

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).

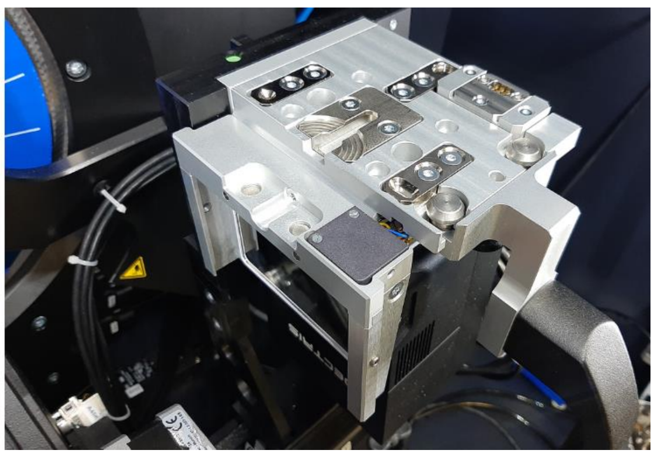


D. Unfasten the latch on the back of the track by pulling it the left with your fingers. The little circle shows green when it is locked and red when it is free. CAREFUL. When you have unfastened you will feel weight on the handle, the magnet supports the detector but does not hold it in place.

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 gamma-optimized (larger accessible range in gamma).



F. Remove handle from detector and place at the right in enclosure.
G. After your measurement slot, make sure you leave the instrument with the detector mounted in the 2θ-optimized position and without adapter plate.

Comment: full optimization of gamma (in-plane direction) is achieved by mounting the adapter plate (stored in the cupboard left next to the fume hood) on the detector mount and then fastening it back on the 2θ arm.

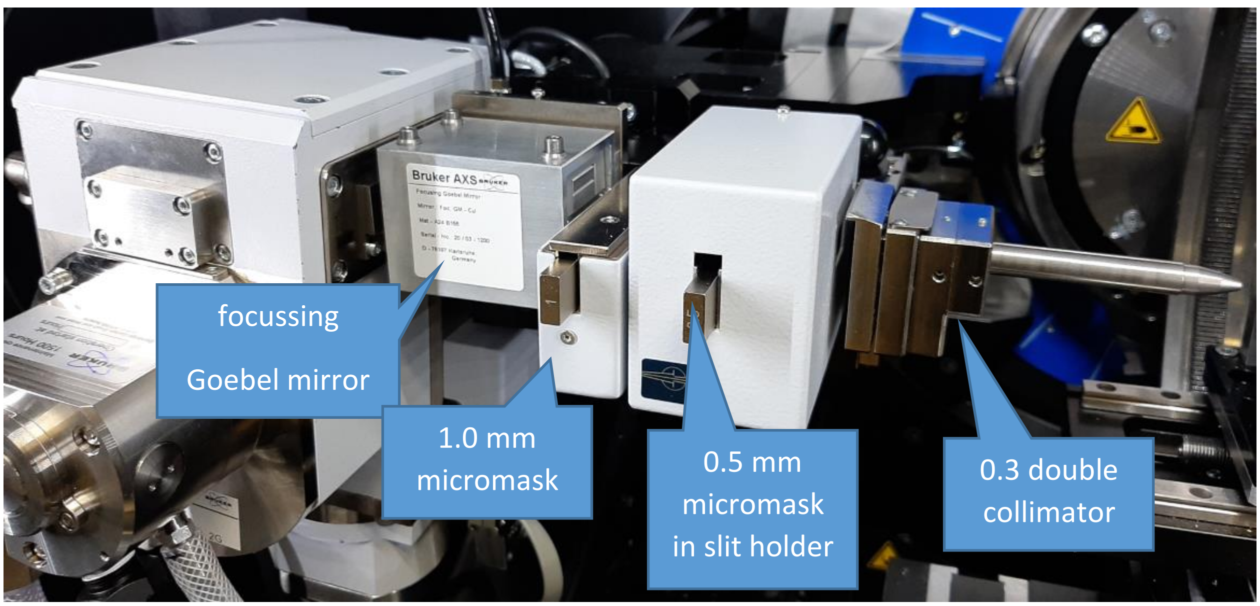
GIWAXS MEASUREMENT

Before mounting components and preparing the instrument configuration for GIWAXS move both the θ- and 2θ-arms to position where this is easily done, e.g. at 25 or 30°, if they are placed in impractical positions when you arrive. If the previous users has measured a Bragg Brentano scan e.g. to 120 °2θ the position of the 2 arms is not practical to exchange components.

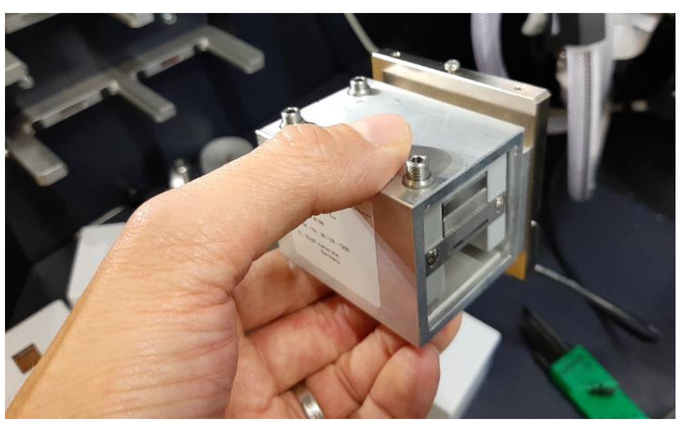
1. Mount optics required for GIWAXS, if not in place.

Besides the required focusing Goebel mirror, it is up to the user to decide upon optics. This will depend on incidence angle, film thickness and scattering behavior. A good general combination can be (mounted from mirror to sample):

- 1.0 mm micro-mask
- 0.5 mm micro-mask
- 0.3 mm double-pinhole collimator



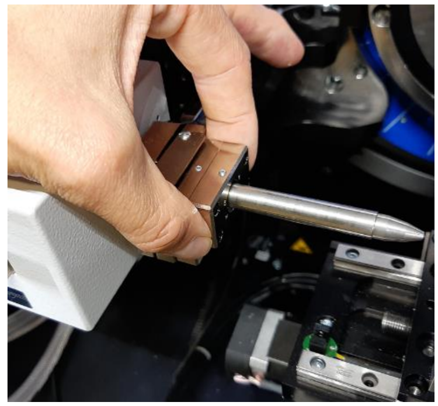
1.1 **Mount the Goebel mirror**, if not mounted already. Handle the mirror CAREFULLY and do NOT LET FALL. If you do let it fall please inform the facility immediately, as realignment is maybe necessary. If you fail to inform then it will be difficult to associate intensity decrease with a certain event.



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).

1.2 **Slide the 2 micro-masks into their holders**, gently making sure they are in all the way.
  1.2.1 If the slit holder is not mounted you need to place this.

1.3 **Mount the collimator**. Two types of collimators are available, those with one pinhole (image below at right) and those with 2 pinholes (image below at left). Those with one hole have a single-piece base and those with two have a "step" in their base. 2 pinholes will give a more circular beam, more adapted to 2D diffraction, but one pinhole will give a little more intensity. Up to you to choose. A suggested combination is given above in point **1.**

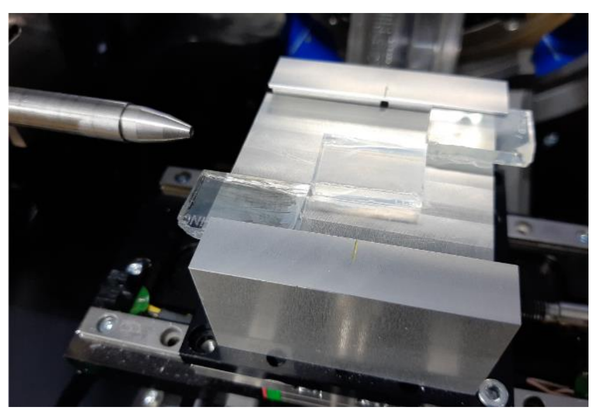



1.4 When mounting any collimator make sure to handle only the base, not the collimator itself (see image top, right)!!!!

2. Mount your sample

The sample can be mounted on different stages. The vacuum stage can be used but then the collimators cannot be mounted for sample alignment, and it is not necessary unless you scan the sample in psi (Eulerian cradle). It is suggested to mount the sample on the standard stage. Since aligning it requires at least one z-scan, you may need to use spacers on the standard stage (something that is similar in thickness to your sample), to compensate for a lack of travel on the z-motor, which can travel from -1.2 mm to 1.9 mm.

2.1 Mount sample on standard stage, using spacers if necessary. To the right you see standard glass substrates, sample in center between two spacers clamped on the sides.

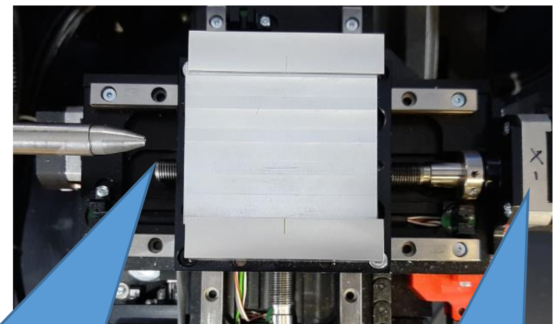


3. **Align your sample: height alignment (z) and optional tilt (rocking curve)**
   Aligning the sample requires finding the correct height (z-scan) and if wanted verifying that the tilt of the sample is ok (rocking curve). For very accurate results these two scans need to be iterated until the difference is within precision of the instrument between consecutive scans. For the very most purposes, it is fine to do just one z-scan and a quick rocking curve.

   3.1 Ramp up generator to 50 kV / 105 mA

   3.2 **Verify that phi (sample spinner) is at 0, as well as x and y** (z does not matter) and that the X-ray tube and detector arms can move freely to 0 degrees, i.e. there is nothing placed beneath them in the enclosure that could physically block them. – CHECK FOR POSSIBLE COLLISIONS (optics, x, y – motors of Eulerian Cradle, detector)

DetectorDistance	[mm]	179.7	179.7	☐
Phi	[°]	0	0	☐
Theta	[°]	1.0000	0.0000	☐
Two Theta	[°]	12.0000	0.0000	☐
X	[mm]	0.000	0.000	☐
Y	[mm]	0.000	0.000	☐
Z	[mm]	-0.8235	0.0000	☐



x,y = 0. Check that there is enough space between collimator and stage and between detector and stage before moving detector and tube to 0°.

x – motor of cradle, y – motor looks identical and is facing the enclosure doors when

   3.3 **Bring the motors "theta" and "detector" to 0.** VERIFY that space is free below the 2 arms.

   Type the set values in the white fields (column Edited) of the corresponding motors, check the white box to the right and click on the cogwheel symbol "3,45" at the top, which will position checked drives.

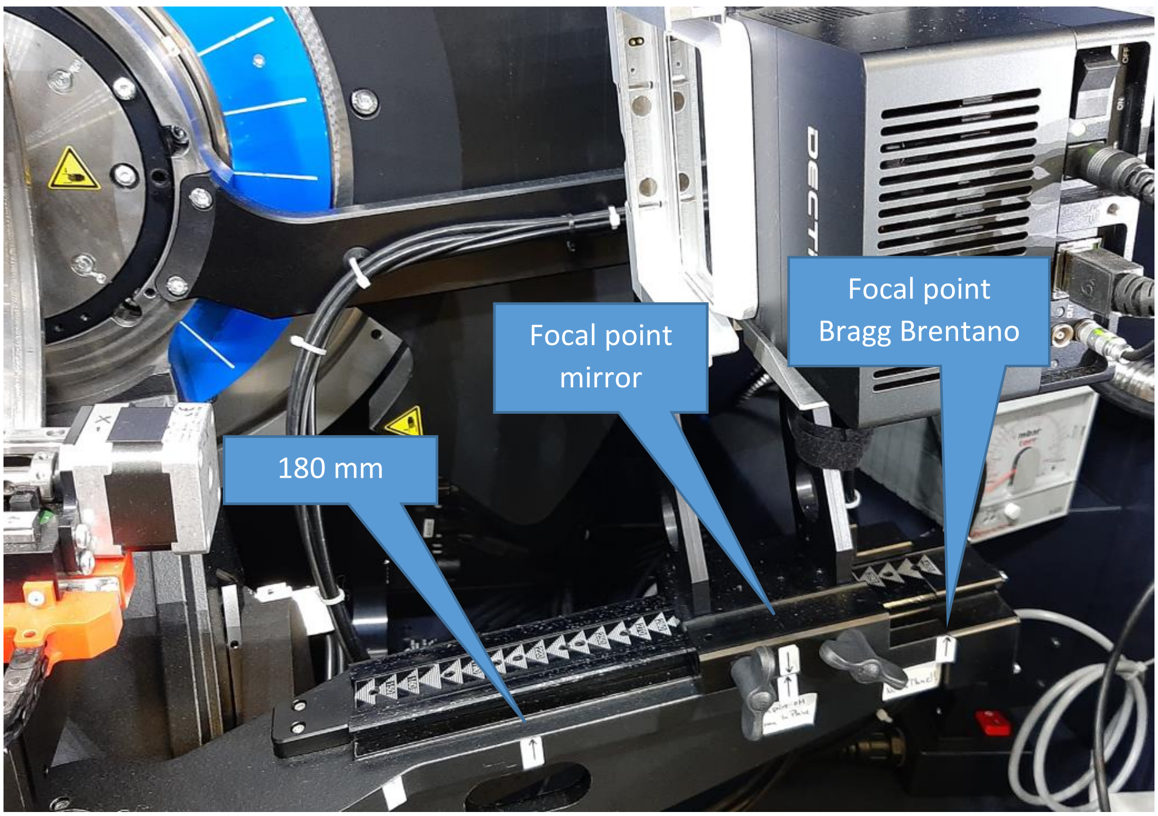
Drive	Unit	Actual	Edited	
Beam Transl.	[mm]	0.0000	0.0000	☐
Psi	[°]	0.0000	0.0000	☐
Detector	[°]	25.8406	0.0000	☑
DetectorDistance	[mm]	380	179.7	☐
Phi	[°]	0	0	☐
Theta	[°]	26.4276	0.0000	☑
Two Theta	[°]	52.2682	52.2682	☐
X	[mm]	0.000	0.000	☐
Y	[mm]	0.000	0.000	☐
Z	[mm]	0.0000	0.0000	☐
Variable rotation	[/min]	0.0	0.0	☐

3.4 Move detector to the desired sample-detector distance

3.4.1 Place protective cover if you do not feel confident.

3.4.2 Unscrew the two butterfly screws fastening the detector to the track and carefully slide it where you need it (bear in mind min and max marks). It may rattle a little when moved.

3.4.3 The best resolution of peaks will be obtained by placing the detector at the focusing point of the mirror, but this is rather far away and since GIWAXS produce inherently broad peaks it is better to place it closer, e.g. at 180 mm (see below).



3.5 **Prepare the z-scan:** you must chose the 0D mode of the detector and define a small ROI (region of interest).

In the Diffrac Commander on bottom left select the **0D option** from the detector dropdown menu.

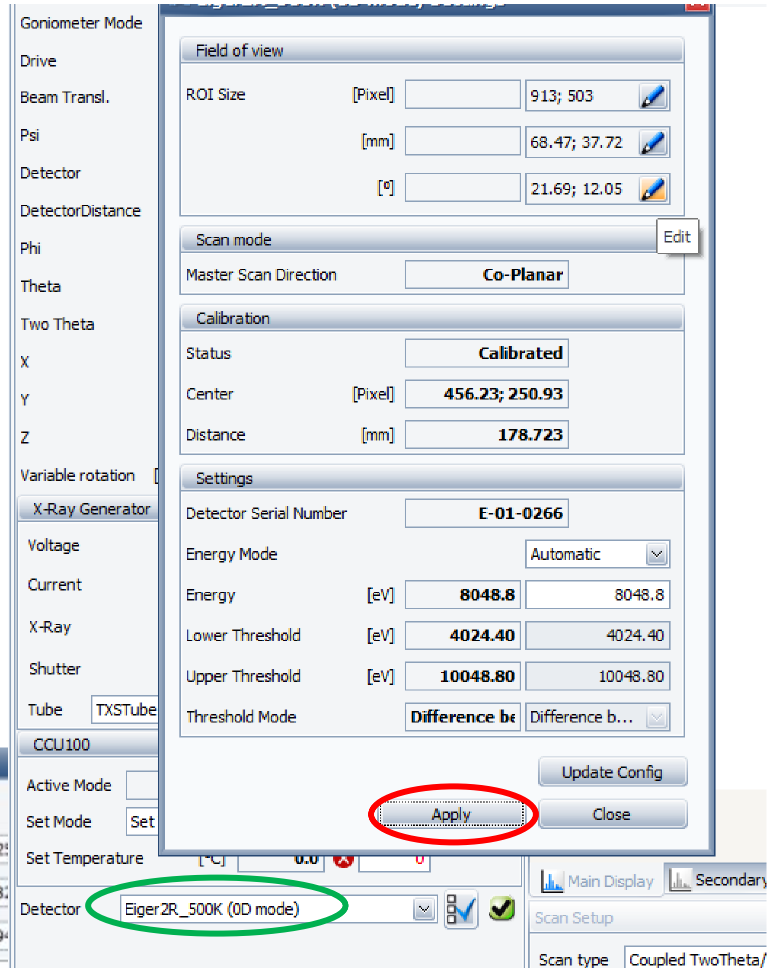
Then click on the blue symbol to the right of the selected mode. The window shown on the right will appear.

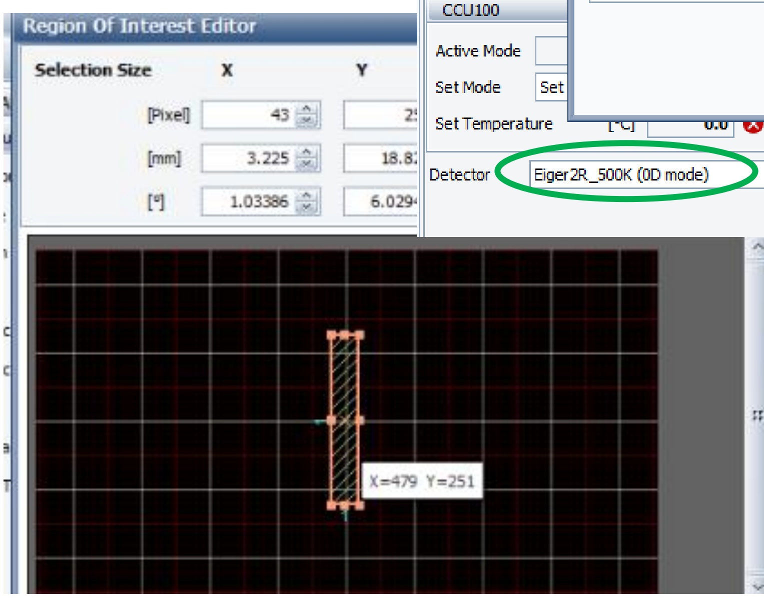
Click on the pen (marked in image) and the ROI editor will appear (image below).

Drag the orange box to the size you need, in this orientation the x-axis is 2Theta and the y-axis is Gamma.

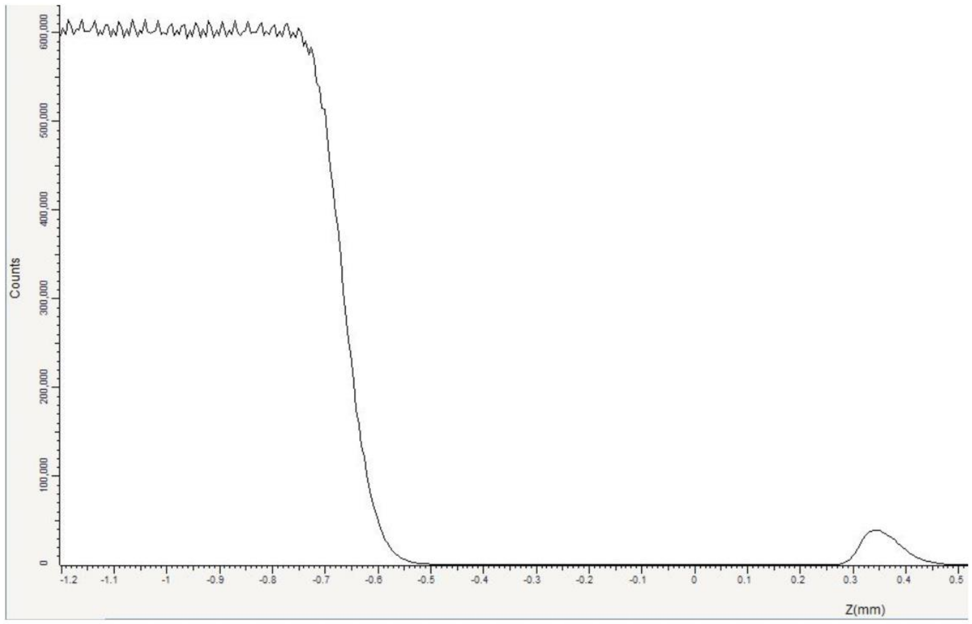
For the z-scan you can choose a ROI as below, of about 1 deg 2Theta.

Apply the Changes.





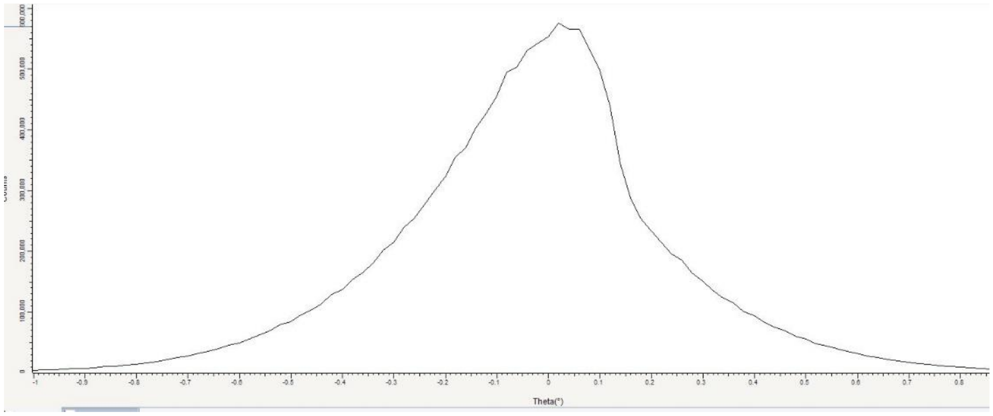
3.6 **Do the z-scan**. Do a z-scan from -1.2 to 1.8 mm (-1.2 is the lower limit!!). This way you are exposing the detector to the primary beam while moving the sample upwards. In scan type on the Commander chose z-scan from the drop down menu. Enter the correct starting and ending values mentioned above, time per step = 0.05s, step size = 0.005 deg. Start the scan.



The z-scan is shown above. Search with the cursor for the z-position on the obtained scan where the counts of the initial plateau are halved, and double click on this position. The value will automatically be taken over as set point for the z-motor, e.g. at -0.8235 mm (see below), but this value depend on the thickness of your substrate, it represents the new sample height.

3.7 **You can at this new height perform a rocking curve** which should be approximately symmetric and peak around 0° theta. This is however not essential, you can also skip this point. As said in 3.6 double click on the z-position to update the z-setpoint and chose rocking curve from the scan type drop down menu. Measure from -1 to 1 °Theta

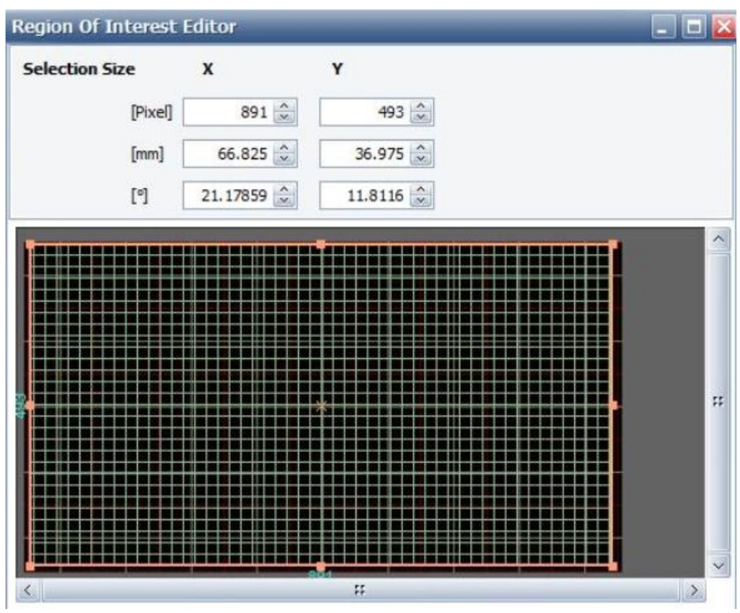
If the rocking curve is approximately symmetric around zero (+/- 0.2) then all is ok to continue with measurement. If it is very far off you may need to iterate z-scans and rocking curves, but this is usually not necessary.



4. Measure 2D Still Image

This manual only explains how to take still images and 2D scans. For GID (0D) scans or symmetric reflection and other refer to the respective manuals or get in touch.

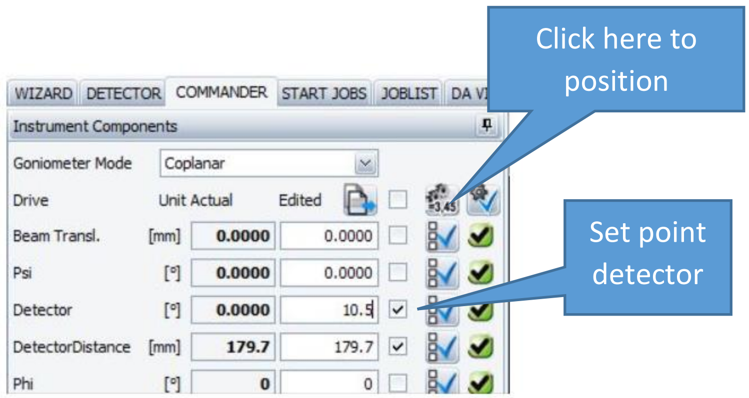
4.1 **Chose region of interest.** As previously, for the z-scan (3.5), you need to choose the ROI, now for the 2D mode of the detector. From the detector dropdown menu at bottom left in the commander chose the option mentioning 2D. Click on the blue symbol (see 3.5) and on the pen to define the ROI size in the field of view. Open it fully dragging the orange box nearly to the boarder.
Do not forget to APPLY THE CHANGES!



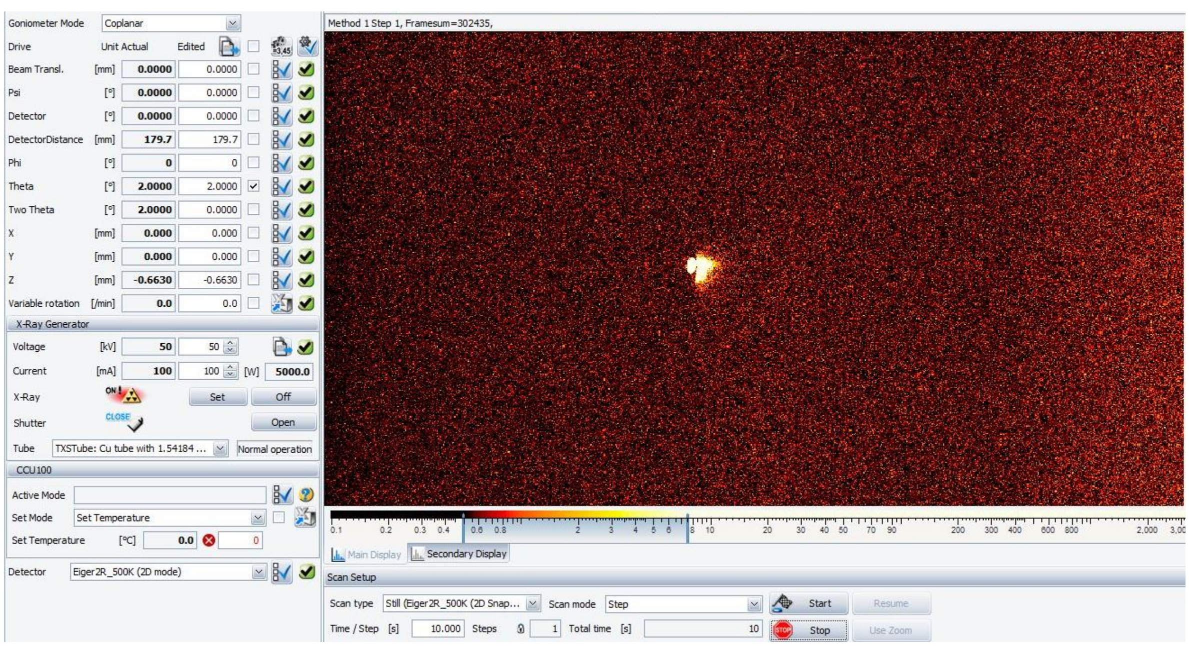
4.2 **Choose the 2Theta position of the detector.**
It is useful to place the bottom edge of the detector at the 2θ value you want to start the image at. This will depend on your detector opening (ROI) and incidence angle (θ). If you want to measure at low angles, the aim is to position the detector such that the primary beam is barely or not at all visible on the lower edge of the detector.

Example. You want to have an image which starts at 2θ = 0°, and the detector is open to 20 °2θ. You should place the detector at 2θ = 10°, which will bring the lower edge to 0° (detector center is at approximately half the opening, i.e. 10 °2θ in this example). Further adjustment are then useful depending on the chosen incidence angle.
You may want to place the detector approximately in 2θ, and then test with a few quick images and further optimizing 2θ to see as little primary beam as possible, while maintaining all the information you need. Once you have optimized you can use this for all samples in a series.

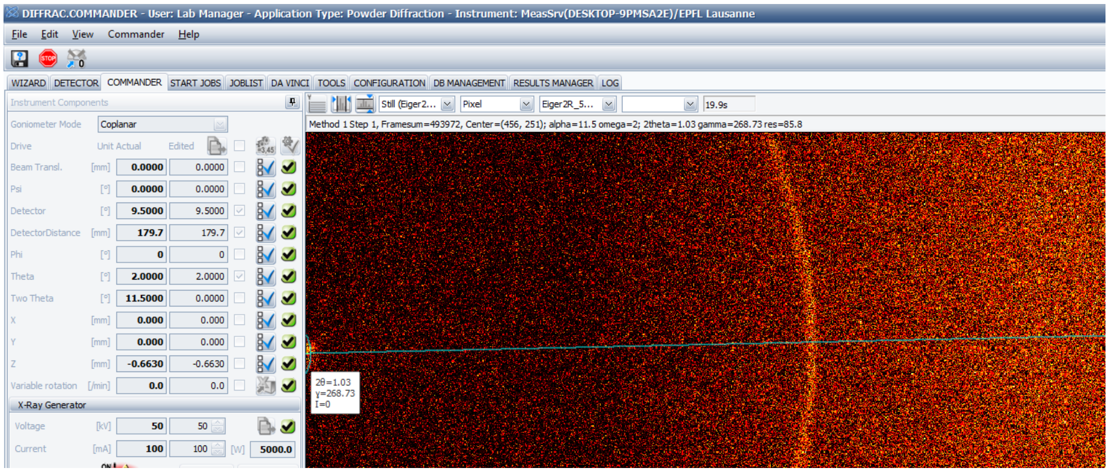
The detector is positioned by typing a set point in the corresponding field (see image below), clicking the square box to the right and then moving the motor by clicking the 3.45 symbol with cogwheels at the top.



Below e.g: a 10s image with the sample height aligned to -0.663 mm in z and the detector at zero during data acquisition. The beam is visible in the center, the substrate shadows the entire detector on the left of the beam. You can increase the 2Theta range by moving the detector to higher angles.



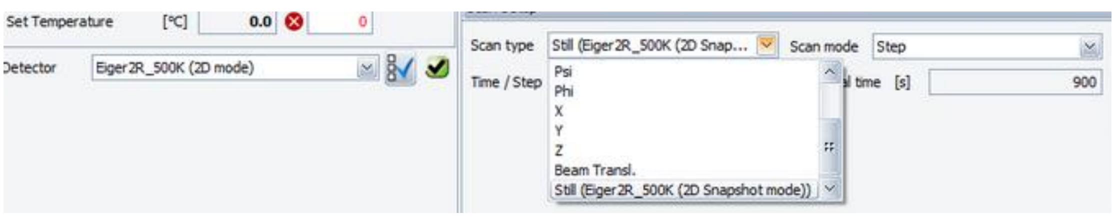
Below, detector moved to 9.5 degrees. The beam is now at the lower edge (on left), and the Debye Scherrer ring previously not captured is now within the accessible 2Theta region.



4.3 **Set incidence angle θ**. You can start at 2 °θ (not 2θ!!!) as a reasonable value, then go to lower incidence angles to become more surface sensitive. Bear in mind that the smaller θ the more surface sensitive you will be measuring, but the broader peaks will become (sample footprint effect) and it is also possible that intensities will be weaker.

4.4 **Checklist:** With detector distance set, sample aligned, 2D mode chosen with ROI and θ positioned you are now ready to measure a still image.

4.5 **Start measurement:** From the scan type drop down window chose "still"
Define an exposure time and start measurement.



Images are saved in a format gfrm that you can open with EVA or Leptos, treat and integrate (2Theta or Gamma integrations). Leptos also allows you to transform pixel positions into qxyz.

5. After measurement make sure you get the machine into the configuration that you found it in and ramp the generator down to 20kV/6mA.

<u>**Measuring 2θ Scans with the 2D detector mode:**</u>

The 2D mode of the detector (both orientations, with or without adapter plate) can be used to acquire still images or 2θ-scans.

- Non-expert users should refrain from performing 2D scans below 170 mm detector distance due to **danger of collisions between the detector and the phi-stage**.
-

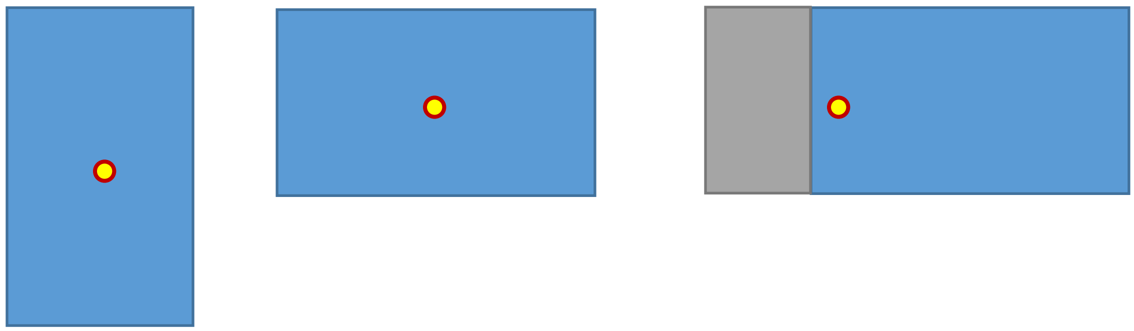
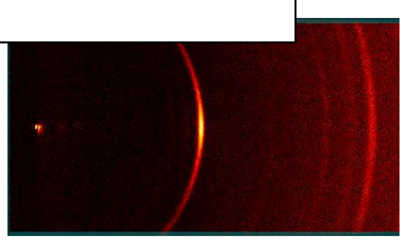


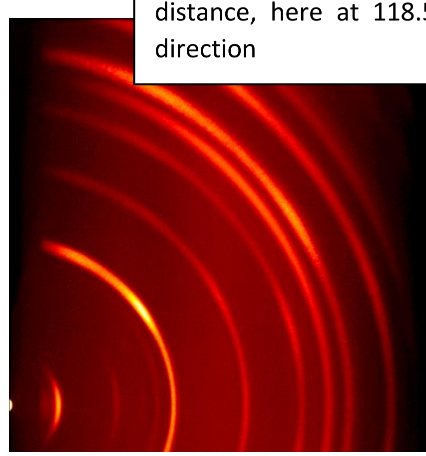
Figure: different detector mountings with beam center at detector position = 0 deg 2θ. Left: 2θ-otpimized, center: γ -optimized, right: γ -optimized with adapter plate (grey).

The largest fraction of reciprocal space (i.e. Debye Scherrer rings) is captured by mounting the detector in γ -optimized orientation with the adapter place and performing a 2Theta scan (bottom right). This way the primary beam is positioned in the detector corner (if the detector is not at zero) and nearly a full quadrant of the ring can be collected. Bear in mind that the image is symmetric across the beam center in all directions, and that **one quadrant contains all the information** you need!
Which detector-orientation you chose depends on your samples and which information you need.

Still image, detector in 2θ-optimized orientation, capturing approx. 22 deg 2θ in out-of-plane direction and 10 deg 2θ in in-plane direction



2θ-scan, detector in γ -optimized orientation with adapter plate (beam in corner of detector), capturing approx. 35 deg 2θ (scan) in out-of-plane direction and 30 deg 2θ (fixed by detector side and distance, here at 118.5 mm) in in-plane direction

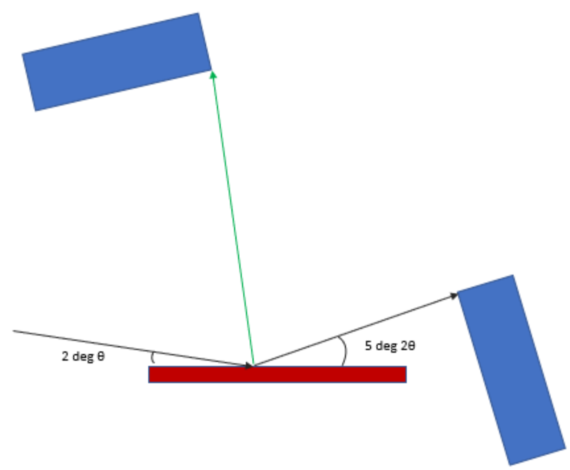


The procedure for 2D scans is very similar in all orientations, regardless wether you measure in 2Theta, gamma-optimized mode, or with the gamma-adapter plate.
In the dropdown menu scan type, you need to choose a 2θ scan instead of a still image. Different options are available; continuous, NDO and VDO. These options relate to if/how the detector opens gradually during the scan, in order to minimize primary beam noise when close to zero. The smartest option is VDO.

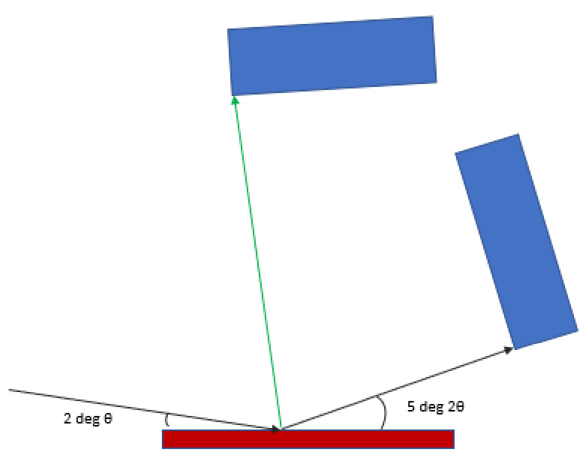
- **Continuous**: Full under- and over-travel of the scan range, all data share same statistics, since every pixel registers the same amount of intensity over the same time of a given Bragg reflection.
- **Continuous-NOUT** (no over-/under-travel): Scan range is not over- or under travelled. This leads to different exposure times of detector sections during the scan. The beginning and the end of the scan will be underexposed.
- **Continuous-VDO** (virtual detector opening): Like NOUT, but opening and closing behavior can be specified independently.

The choice of scan type depends largely on the scan range, the detector opening and the lowest angles you are trying to reach. 3 examples should illustrate the 3 different scans, on a sample where you want to measure a scan range of 5 – 95 deg 2θ, and a detector distance where the full opening is 26 degrees.

Continuous (full under and overtravel:



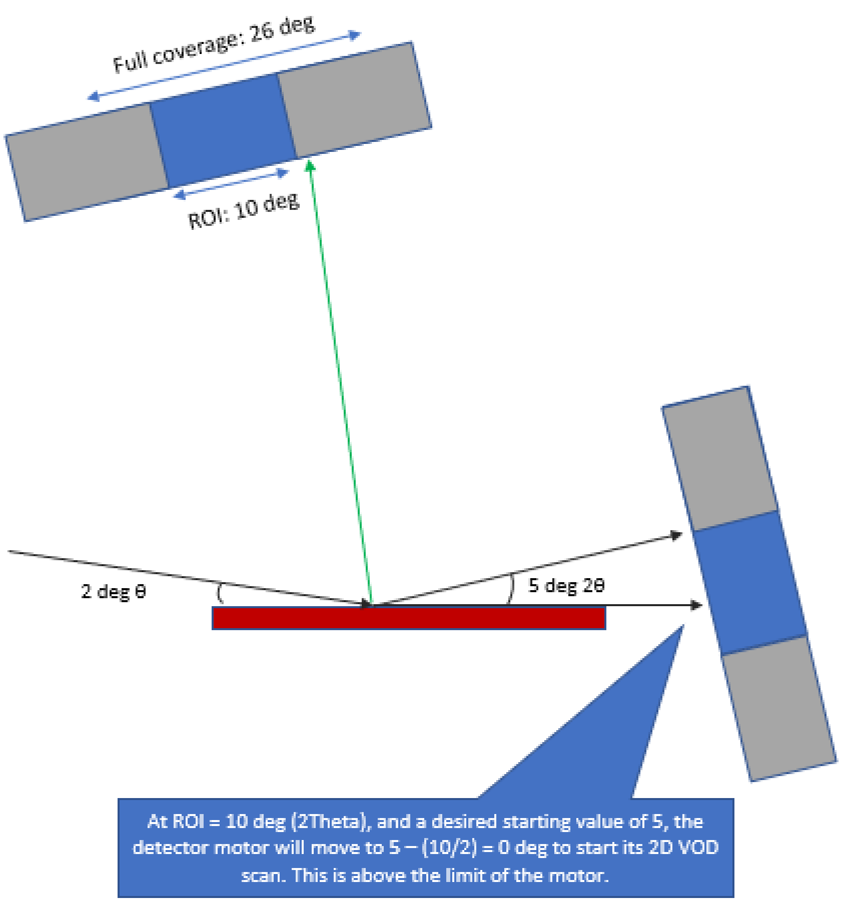
Continuous NOUT (no over- /under travel):

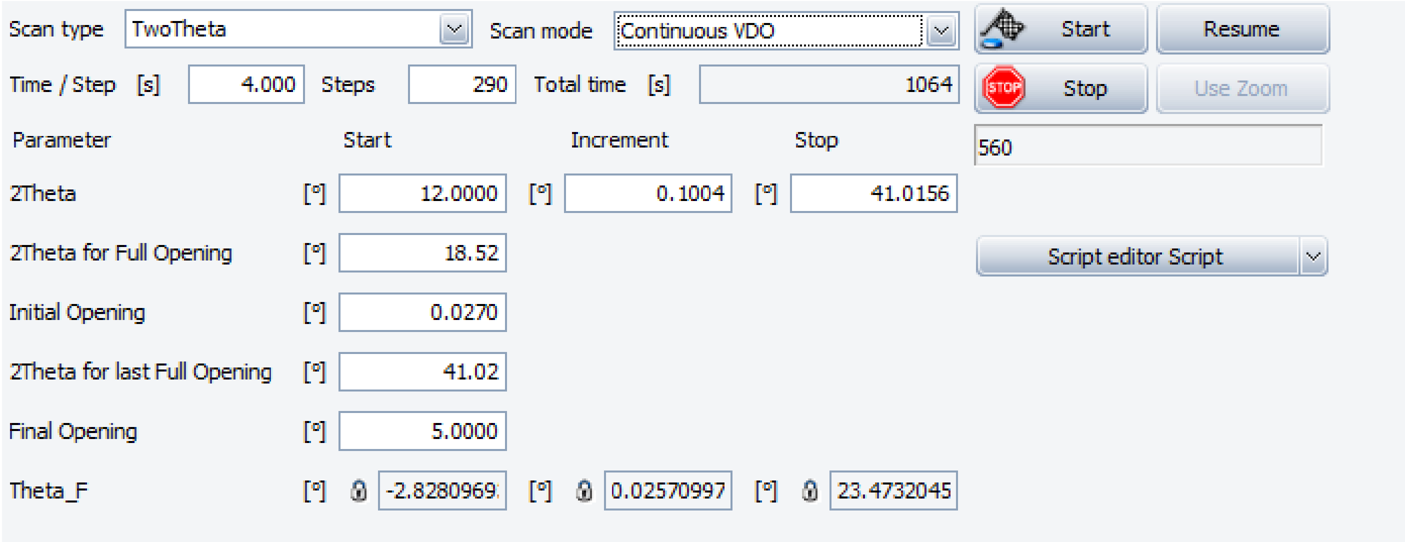


Continuous VDO (virtual detector opening):

In the VDO scan you control the way the detector opens at the start and end of the scan range.
This allows you to use a larger ROI of the 2D sensor at the lowest angles while adjusting it to still prevent either primary beam or the lower limit of the motor of the detector drive. Example, below:

- In a continuous scan with a 26 deg ROI the detector would move with its motor to -8 deg 2Theta, which is below the limit of this motor and will thus return an error. The detector opening is too large for the low angles.
- In a continuous VDO scan you can set a smaller ROI at low angles (high angle side less important), e.g. you could choose the detector to measure the lowest angles (5 deg) with an opening of 10 deg 2Theta (box 'initial opening' in Diffrac Commander), which will drive the detector to approx (some overtravel is always involved to allow the encoder to get the exact position) 0 deg 2Theta to start the scan. The detector can start opening up and be fully open at 21 deg 2Theta (5 + 16).





Continuous scans with low angle starting values:

You can always run a continuous scan starting at low angles if you chose the ROI (fixed ROI in continuous scan) small enough to allow the detector to under travel. This way all pixels have same statistics but you can still run continuous. The disadvantage, since the ROI is now smaller, e.g. 5 deg instead of 20 deg, the intensities will overall be lower because less pixels registers the same angle. You will have to increase exposure time to compensate for this.