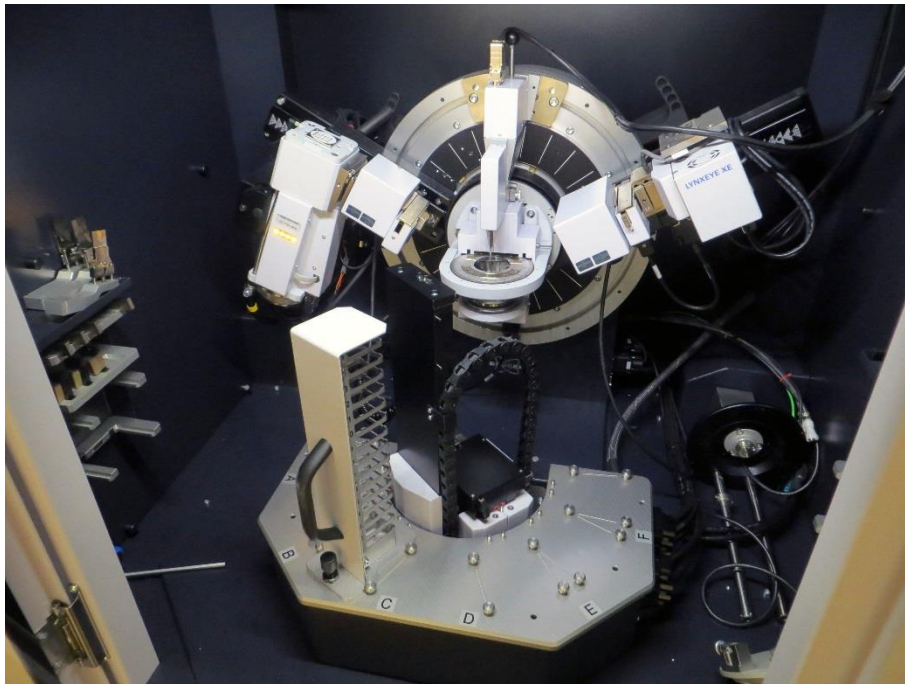


Standard Instructions for the Bruker D8 Advance Diffractometer, EPFL Valais Bragg Brentano and GID (Reflection)

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This instrument allows performing 3 different basic kinds of measurements:

- Bragg Brentano measurements in reflection mode
- Basic GID (grazing incidence) measurements in reflection mode
- Debye Scherrer capillary measurements in transmission mode

The basic configuration for each kind of reflection measurement is discussed in the following. A sample changer taking up 15 samples is available for reflection measurements. A heating chamber ($T > 1000^{\circ}\text{C}$) is available for transmission measurements. Transmission measurements are discussed in a separate document.

General notes when using this instrument:

- Prepare samples in your lab, remove as soon as possible after measurement and dispose of them in your lab. Bring back CLEAN sample holders, users do usually not wear gloves.
- Note your name in the logbook, along with lab and number of samples (comments if needed).
- Always watch for collisions before moving any drives! Especially when measuring in GID or Transmission.
- Emergency shut-off (cuts power) by pressing one of the 3 red buttons (see image below).
- No external storage devices on any instrument.

Logins:

Local PC: Bruker

Difffrac: User (user) / User (pwd)

Generator :

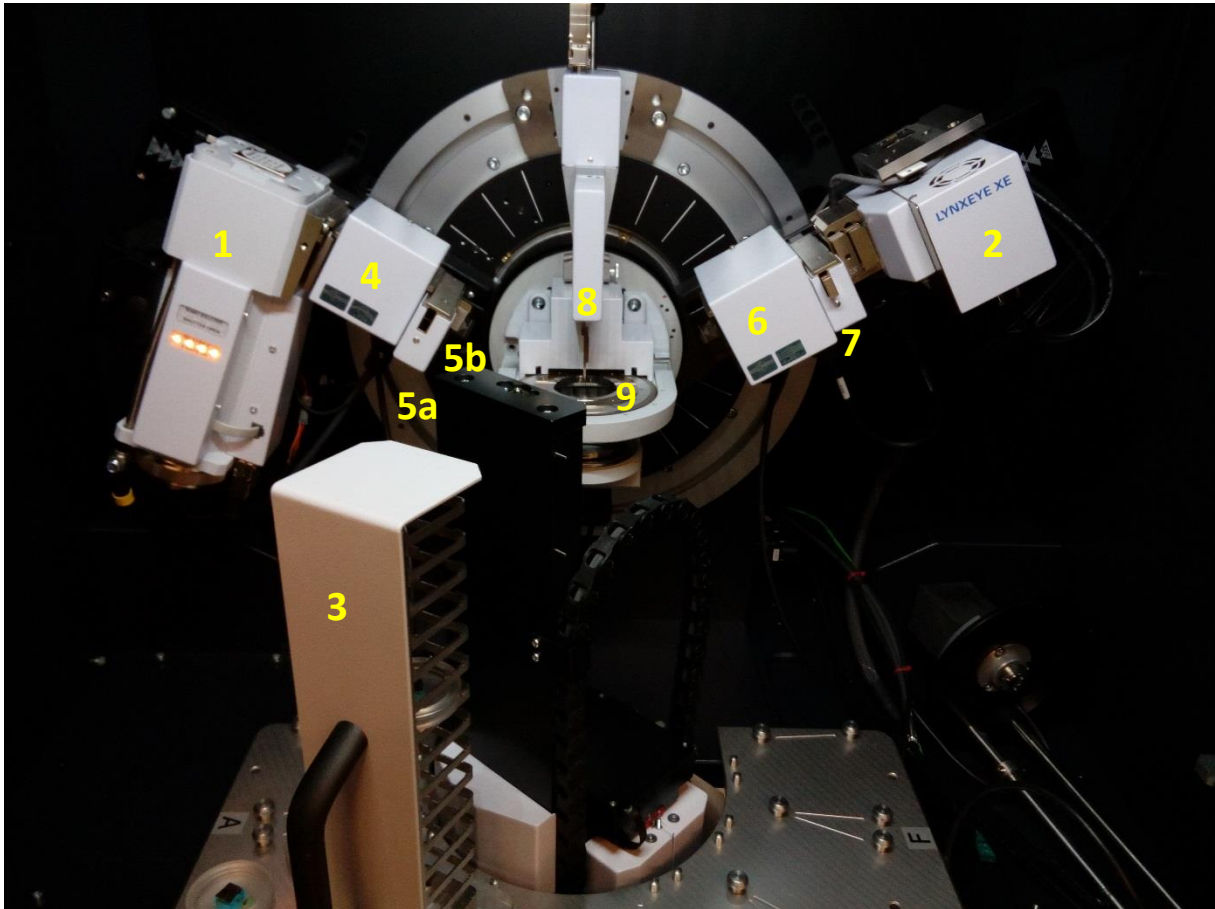
The instrument is ready to go when you see a yellow light (top, with a radiation symbol) and green (bottom) light on the left hand side of the enclosure.

Operating power 40 kV / 40 mA

Standby powder 20 kV / 5mA



Configuration Bruker D8 Advance:



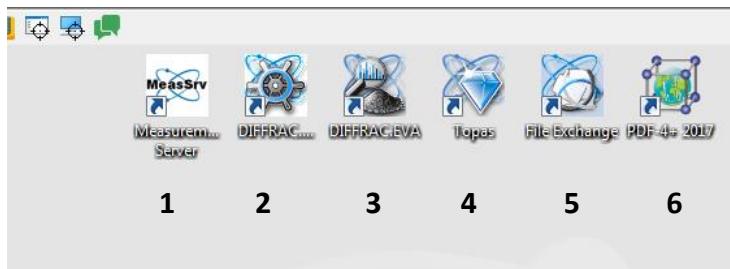
1. X-ray source (line or point focus)
2. X-ray detector (1D or 0D)
3. Sample Changer (15 per rack)
4. Primary Optics (Göbel mirror, motorized slits)
5. a) Incoming slit (empty in image)
b) Incoming axial Soller slit
6. Secondary Optics (equatorial Soller, motorized slits)
7. Ni filter (K_{beta})
8. Knife edge (anti air scattering)
9. Standard sample stage (options: capillary stage or furnace)

Changes of optics between configurations on this instrument are done by mouse-click in the Commander or Da Vinci plugins of Diffrac.

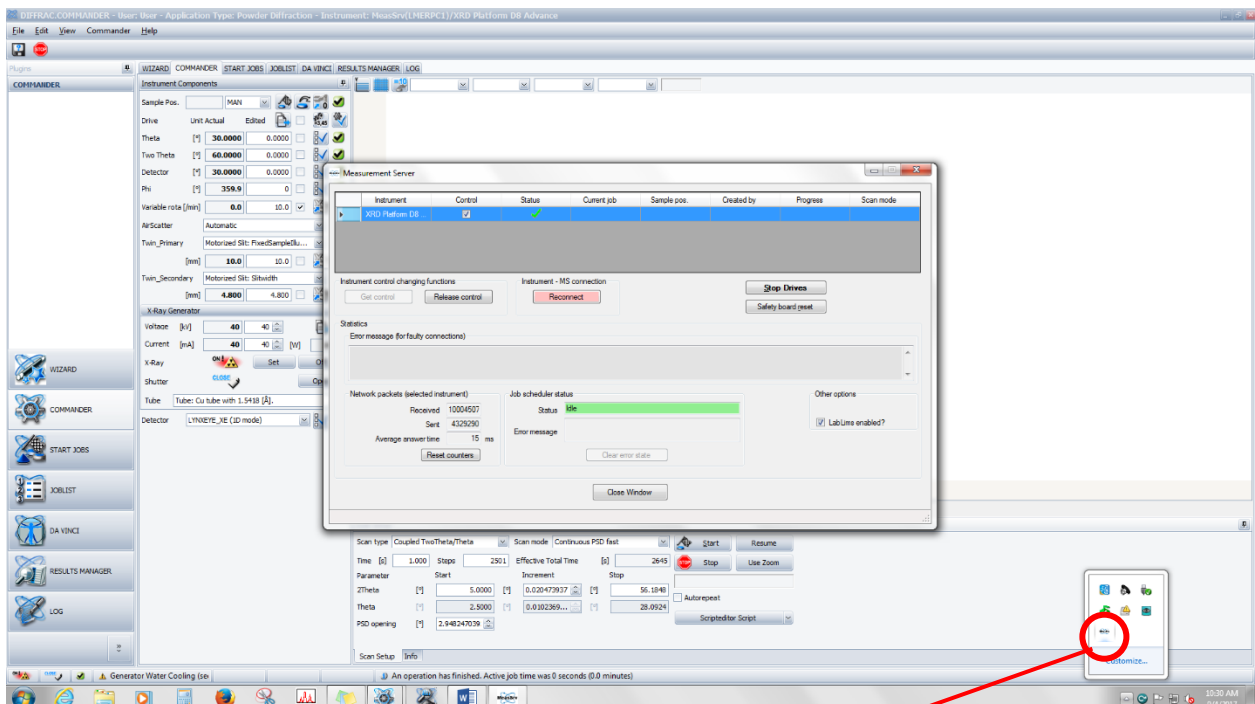
Changes on the goniometer (capillary stage and high temperature furnace) require extra training on demand.

Bear in mind: whatever you place in the beam-path consumes intensity (though often improves resolution).

Things you will/might need on the desktop:



1. Measurement server: needs to be running to, allows PC to communicate with diffractometer. Usually starts automatically upon logging onto Diffrac.
2. Diffrac.Measurement: This is where you control your measurements.
3. Diffrac.Eva: Data visualization, transformation, “search and match” option, and various other preliminary analyses.
4. Topas: Advanced analysis (structure refinement, solution, phase analysis....).
5. File exchange: File format exchanger, can also be done with EVA.
6. PDF: Powder Diffraction File database. Covers inorganics. When doing “search and match” in EVA this database is used.



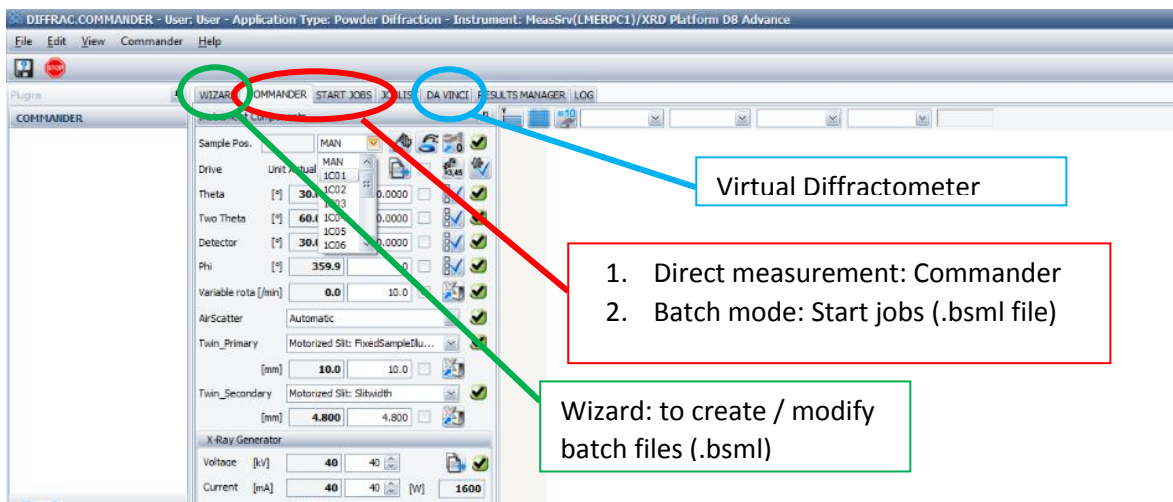
- If the instrument has connection problems, try reconnecting in the measurement server, which can be found in the windows taskbar.

Bragg – Brentano (symmetric, Reflection) Measurement:

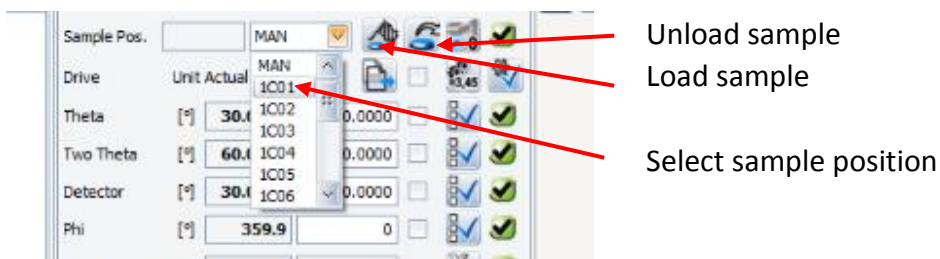
Optics:

Primary: Motorized slits
 Secondary: Motorized slits
 Incoming slit: empty
 Incoming axial Soller slit: loaded
 Nickel Filter: loaded
 Detector: 1D

- **Open Diffrac.Measurement.**
 - Login as User, password User.

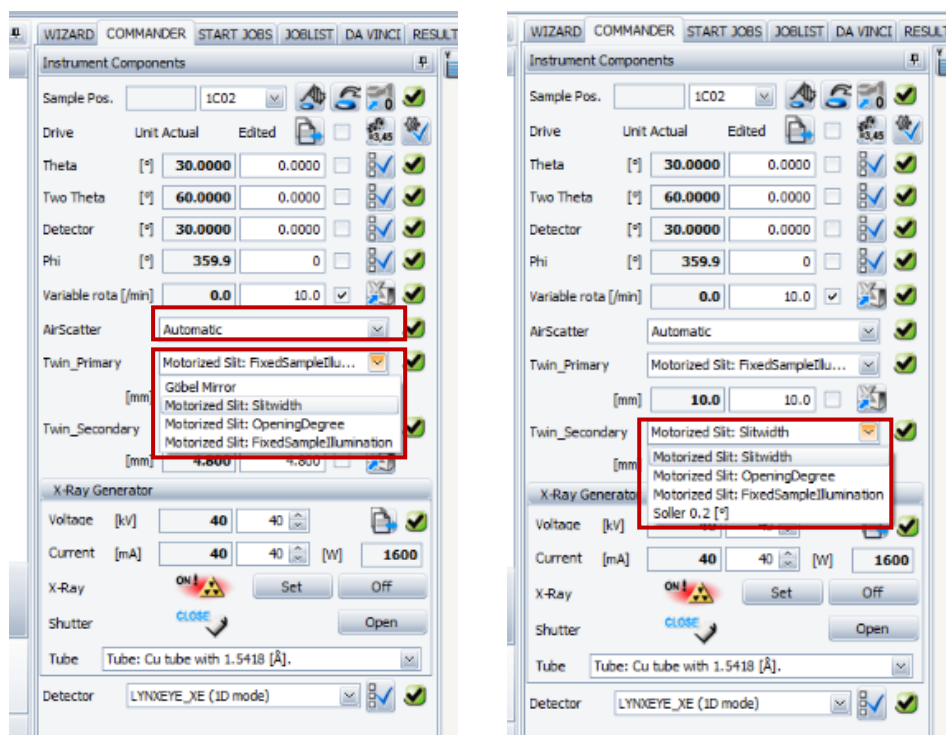


- **Load Sample.**
 - Place sample in respective slot of the changer, select this slot + click the “load” icon.
 - If avoidable, do not use the manual option of sample loading (danger of forgetting sample and causing collision of robot)



- **Check configuration.**

- It is better to check the configuration, the previous user may have been in a different one. .



Air Scatter: Mode depends on scattering of sample. For bad scatterers or low sample amounts it is better to run this in automatic mode (minimizes air scattering).

- **!!!!CAREFUL:** At low angles (MOFs, Zeolites, 2D materials, membranes...) the knife edge will come down very close over the sample (0.6 mm). There is a danger that you lose the sample while rotating.

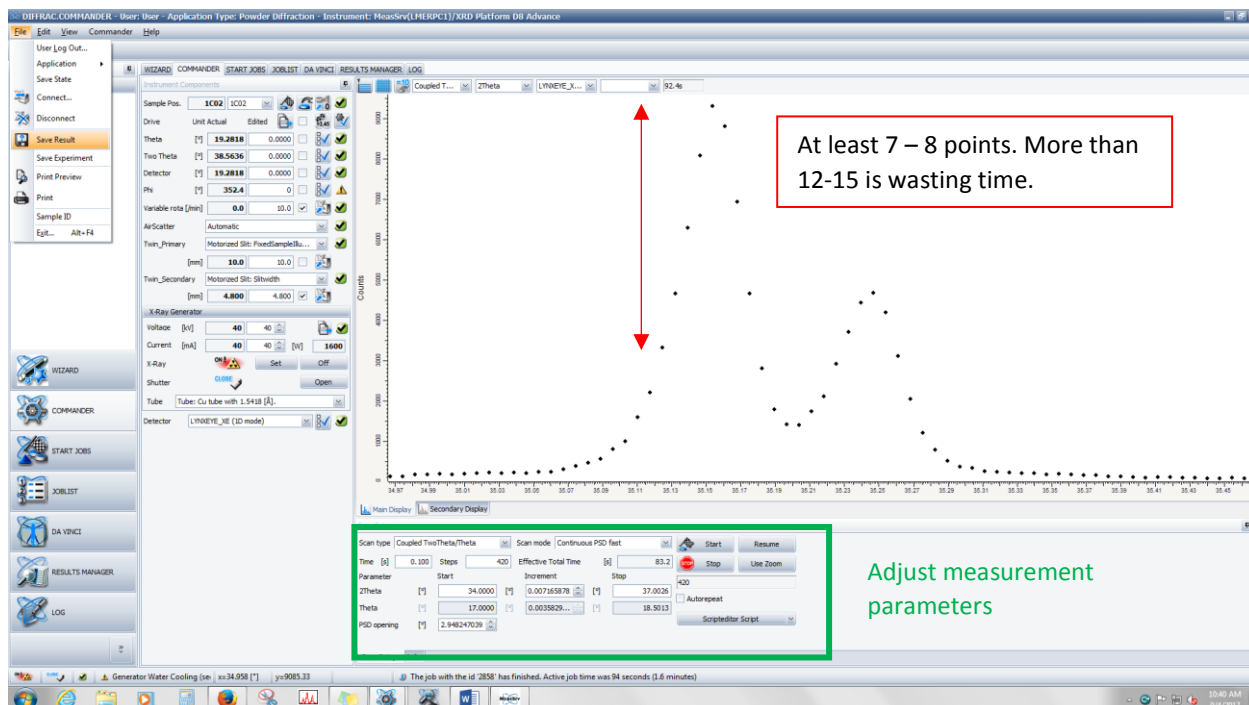
Primary Optics: For BB you use motorized slits. You can (i) set the aperture to a fixed value (in mm or degree) to approximate the assumption of a constant irradiated volume, or you can (ii) set the X-ray footprint on the sample to a fixed area. (ii) is likely better for very thin samples (films, little powder on Si-crystal holders..), e.g. 10 mm for a 1 cm² substrate.

Secondary Optics: For BB you use motorized slits. Different options as on the primary ones. These optics influence instrumental resolution. Closing them will improve angular resolution. In the very most of cases the resolution is sample-limited, meaning worse than the achieved angular instrumental resolution at open slits. You can usually set them close to 5 mm on “Motorized Slit: Slitwidth”. Try around with different slits and decide.

Sample rotation: It is always better to rotate your sample, if you can. A speed of 10 rpm if reasonable. (watch the knife edge at low angles if in automatic mode).

- **Chose measurement conditions.**

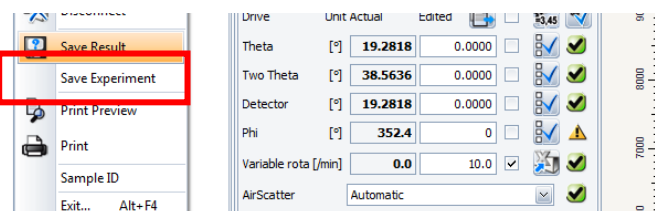
- In order to efficiently use your reserved time it is best to optimize various measurement parameters, especially when you change from one “type” of sample to another.



- **Scan type:** ALWAYS “coupled two theta/theta” for Bragg Brentano measurements on this instrument.
- **Time:** Have a quick look at some intense peaks and adjust the time to get a decent signal/noise ratio.
- **Increment:** It is recommended to choose the 2Theta step such that you obtain at least 7-8 points above the full width at half maximum of the Bragg peak in order to get good peak shapes. This is especially important if you want to model data yourself or have them modelled by someone else (profile fitting, Rietveld refinement...).
- **PSD opening:** (Detector slit) Can be at the maximum, as with the slit, play around for resolution.

- **Start measurement.**

- **Direct:** When measuring directly with the commander you simply click the “Start” button, once you are happy with your config.
 - **Data:** When measuring directly from the Commander neither filename nor path can be specified prior to measurement. You can save data once it is written and the measurement finished (i.e. once you see it on the screen).
 - **All data is saved by default** in the folder “Results”, even if you forget to save.
- **Batch mode:** You can save your measurement conditions for a respective experiment as a Bruker “.bsml” file. This file can then be loaded in the start jobs plugin.



- **Start jobs:** You can save your measurement conditions for a respective experiment as a Bruker “.bsml” file. This file can then be loaded in the start jobs plugin.

Valid	Sample Position	Sample ID	Experiment Name	Result File Name
✓	1C01		..\CRN_man.bsml	..\CRN_man.brml
✓	1C02		..\CRN_man.bsml	..\CRN_man1.brml
✓	1C03		..\CRN_man.bsml	..\CRN_man2.brml
✓	1C04		..\CRN_man.bsml	..\CRN_man3.brml

To run measurements in batch mode you place samples in the sample changer, and specify their position in the interface. You assign an experimental “method” (bsml file) to each sample under “Experiment Name” and specify a filename and path under “Result File name”. You then start all measurements by clicking on Start.

- **Conflicts in batch mode:** It can happen that the current instrumental configuration does not match the configuration specified in your method (bsml). This creates conflicts and the measurement will not start. Common mistakes are:
 - **Ni filter** (was not loaded when creating the bsml file)
 - **PSD opening**
 - **Axial Soller slits**

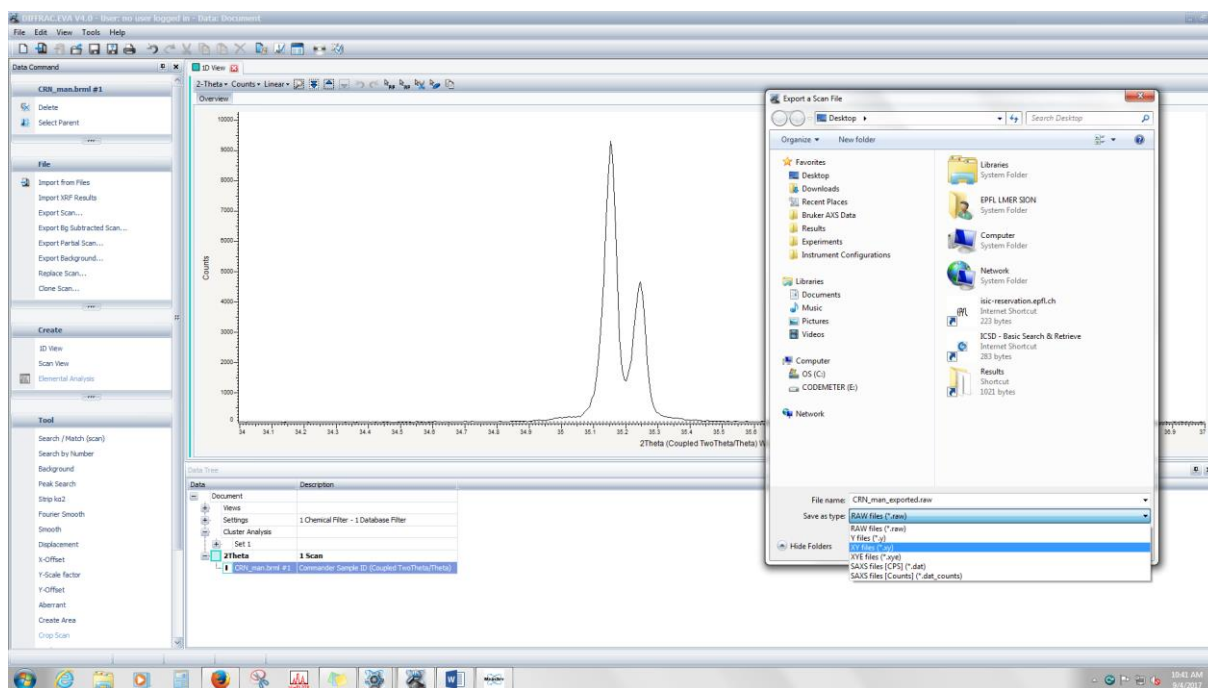
Usually the instrument will try to give you a hint as to what is wrong (appearing as a red warning in the column “Valid” on the left in the Start Jobs plugin). It is worth checking the **Da Vinci** plugin to see the Virtual Diffractometer.

- **Wizard:** The Wizard plugin will allow you to inspect and create bsml files, independently of what you are doing in any other plugin.

- **Accessing data.**
 - **NO EXTERNAL DEVICES on any instrument!**
 - All data are saved by default (see above) in the Results directory of the local PC. If you measured in batch mode you have specified the path previously. All data are synchronized every 15 minutes.
 - Descriptions on how to access them are found on our wiki:

<https://wiki.epfl.ch/xrd>

- **Visualizing data.**
 - Data are written in .raw and .brml format, and need to be transformed prior to working on them with any non-Bruker diffraction software or any data analysis package. You can do this with the file exchanger found on the desktop, or with EVA.
 - **EVA:** Load your results, brml or raw, and simply export them as a format of your choice, xy being the most common one.



Grazing incidence GID (asymmetric, Reflection) Measurement:

Comment: GID is better performed on the D8 Discover. The D8 Advance does not allow for aligning the height nor omega. Additionally the standard sample stage can shadow the beam at low angles.

Optics:

Primary: Mirror

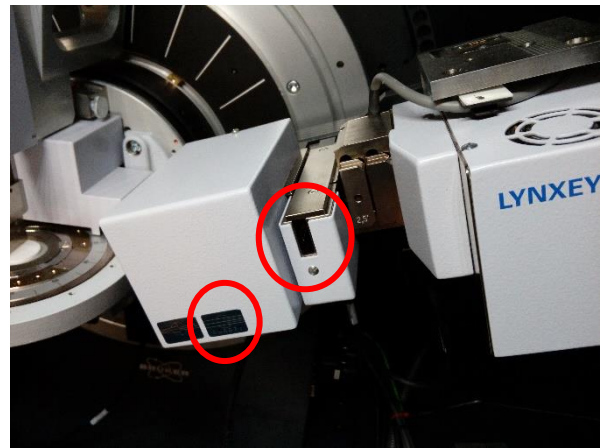
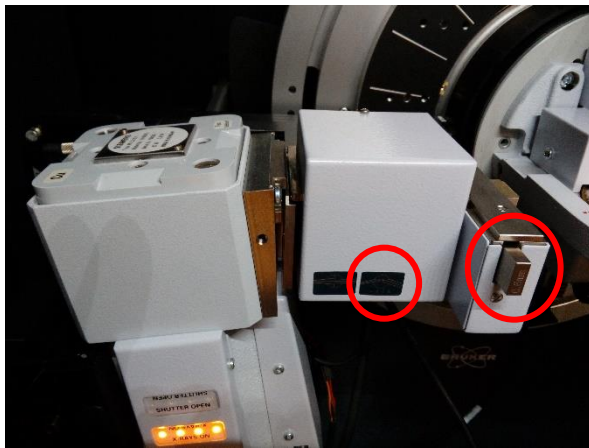
Secondary: Soller

Incoming slit: loaded, calculate to match beam footprint on sample depending on incident angle.

Incoming axial Soller slit: loaded

Nickel Filter: empty (Göbel mirror eliminates K_{β})

Detector: 1D or 0D



• **Configuration:**

Primary Optics:

- Change to the Göbel mirror by mouse click (either in Commander or in Da Vinci).
- The mirror has no integrated slit, you will cut the beam size down to your sample by placing a mechanical slit in the incoming slit holder (see above). The slit size will depend on your sample dimension and your incident angle. The lower the incident angle, the smaller the necessary slit. It is best to calculate the required size. Slits are found inside the enclosure on the left in their storage racks (as well as in the other instrument enclosures). The beam exiting the mirror is approx. 1 mm wide in the direction of the scattering vector.

Secondary Optics:

- You do not need the Nickel filter since you are using the mirror. Remove it to minimize absorption.
- Change from the motorized slit to the equatorial Soller slit in the Commander or in the Da Vinci virtual diffractometer.

Air scatter:

- Match the air scatter screen to cut off as little of the incident beam as possible. This can be done by using the automatic mode.

Sample stage:

- Ensure that you are not shadowing the sample at low angles. Change the phi rotation in the commander if this is the case.

Detector:

- In general a GID measurement is of higher quality in the OD mode of the Lynxeye. Which allows recovering some of the angular resolution lost by the parallel beam. However, this depends largely on the crystallinity of the sample, and the intensity loss due to the OD mode may not be worth a minimal increase in resolution; try both OD and 1D.

- **Chose measurement conditions.**

- Optimize measurement parameters as for the Bragg Brentano scan.
- BEWARE: Due to non-focusing conditions peaks will be a lot broader. This broadening is not due to particle size, but to conditioning of the beam.
 - Adjust your stepsize accordingly!!!

The screenshot displays the Commander software interface. The 'Instrument Components' panel on the left has several settings highlighted with red boxes and red text annotations:

- Theta** is set to 2.0000. Annotation: "Fix source to incidence angle".
- Twin_Primary** is set to Gobel Mirror and **Twin_Secondary** is set to Soller 0.2. Annotation: "Change primary optics to Mirror, secondary to Soller".
- Detector** is set to LYNXEYE_XE (OD mode). Annotation: "Use OD for better resolution (click on the symbol next to the detector spec to open/close the active window)".

The main display shows a 2Theta scan plot with 'Counts' on the y-axis and '2Theta' on the x-axis. The plot shows a broad peak around 6 degrees 2Theta.

The 'Scan Setup' panel at the bottom has the following settings:

- Scan type: TwoTheta
- Scan mode: Continuous
- Time [s]: 10.000
- Steps: 2501
- Effective Total Time [s]: 25010
- Parameter: 2Theta
- Start: 10.0000
- Increment: 0.04
- Stop: 61.0000
- Autorepeat:

Annotation: "2Theta scan, source is fixed in GID!"

- **Scan type:** 2Theta scan.
- **Incident angle of primary beam:** Chose incident angle manually according to sample requirements and desired information (the lower the angle, the more sensitive your are to surface information) and drive the source to this angle. The source does not move in a GID measurement.
- **Time and Increment:** As for the Bragg Brentano scan, a minimum of 7-8 points above the full width at half maximum. Note that absolute intensity registered will be much lower (compared to BB) since you are probing the surface and shaping the beam. Measurement times can be up to many hours.
 - Measure overnight to not penalize other users!
- **Detector opening:** In the very most of cases you can fully open the 0D or 1D detector. Closing them will improve angular resolution but needs to be justified by the crystallinity of your sample.