Standard Instructions for the

Bruker D8 Advance Diffractometer, EPFL Valais

Debye Scherrer geometry (Transmission)

For any questions regarding the X-ray facility, contact:
Pascal Schouwink

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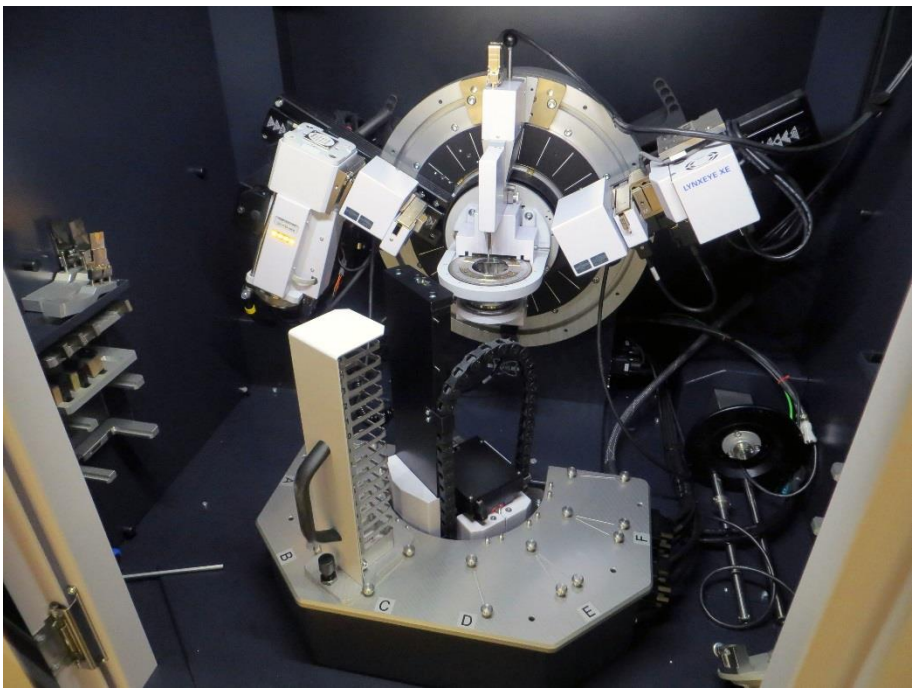
https://isic.epfl.ch/X-Ray



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 measurement is discussed in the following. A sample changer taking up 15 samples is available for reflection measurements. A heating chamber (T > 1000°C) is available for transmission measurements.

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 shot-off (cuts power) by pressing one of the 3 red buttons (see image below).
- No external storage devices on any instrument.

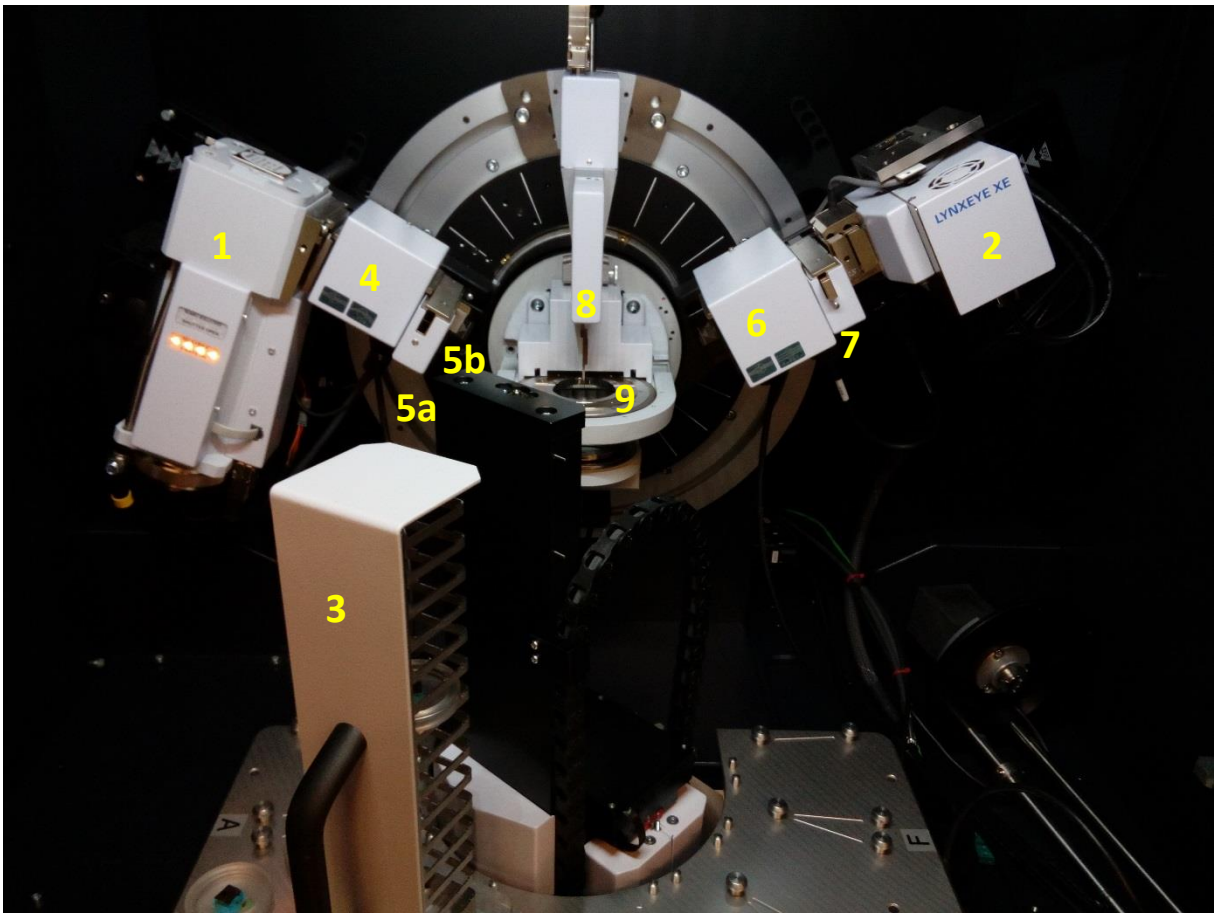
Logins:

Local PC: Bruker
Diffrac: 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:



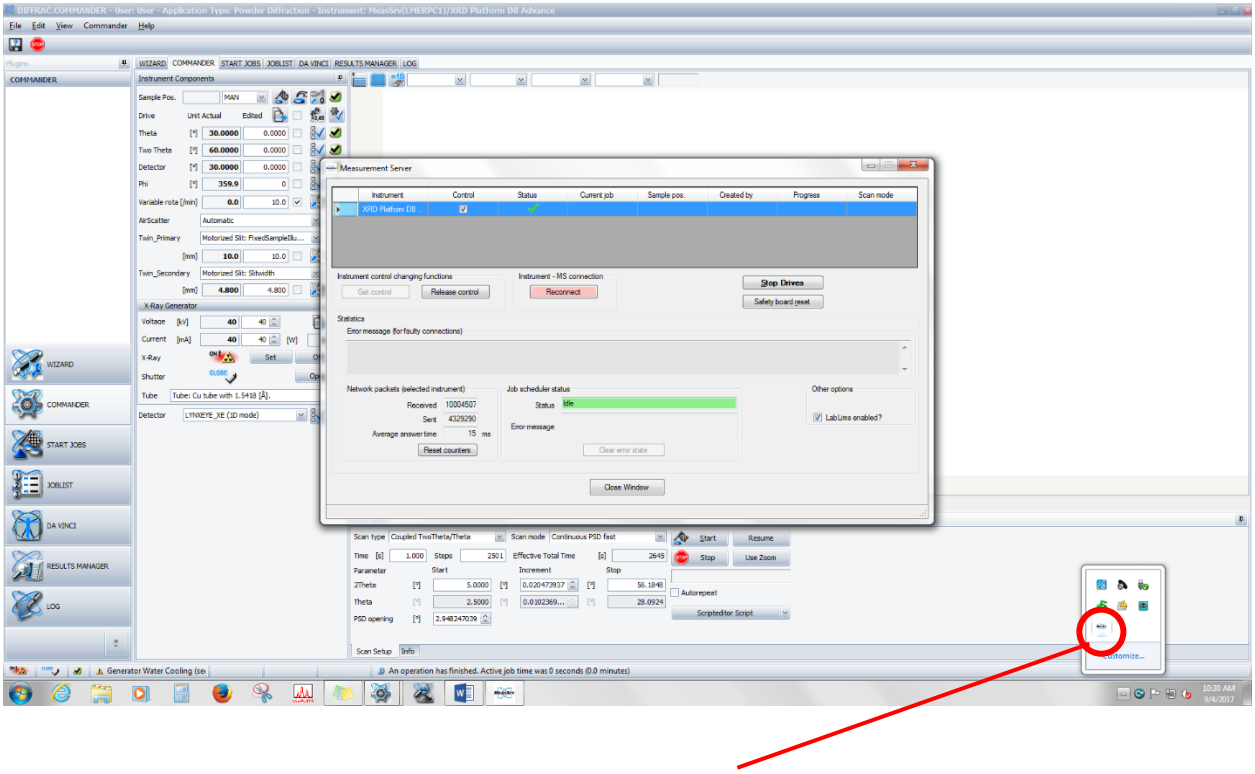
1. X-ray source (line or point focus)
2. X-ray detector (1D or 0D)
3. Sample Changer (15 per rack)
4. Primary Optics (Göbel mirror, motorized slits)
5. a) Incoming slit (empty in image)
   b) Incoming axial Soller slit
6. Secondary Optics (equatorial Soller, motorized slits)
7. Ni filter (K_beta)
8. 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 beampath 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.

Debye – Scherrer (Transmission) Measurement:

Accessible 2Theta range, approx. 137 °. Possible at room temperature or in the furnace (T > 1000 °C). Requires mounting of capillary stages. Contact Pascal Schouwink or perform extra training.

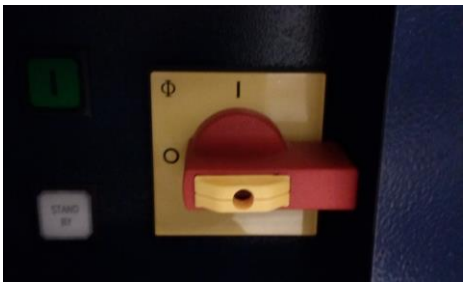
CAREFUL:

When switching from reflection to transmission you will exchange different parts of the instrument.

NEVER leave anything anywhere beneath the X-ray tube and its arm nor the detector and its arm. Risk of collision!!!

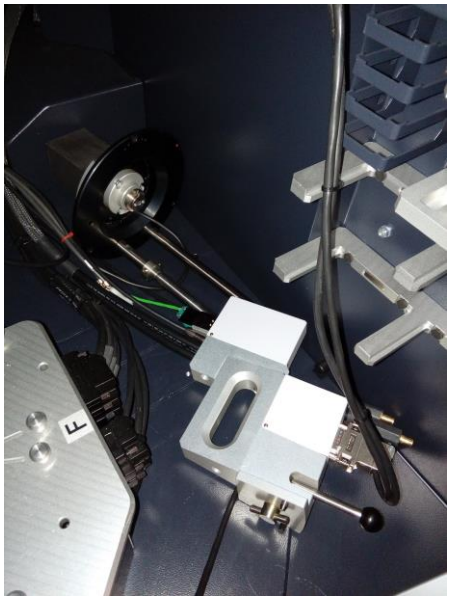
Optics:
Primary: Mirror
Secondary: Motorized slits
Incoming slit: loaded, size should approximately match capillary diameter.
Incoming axial Soller slit: loaded
Nickel Filter: empty (Göbel mirror eliminates K_beta)
Detector: 1D

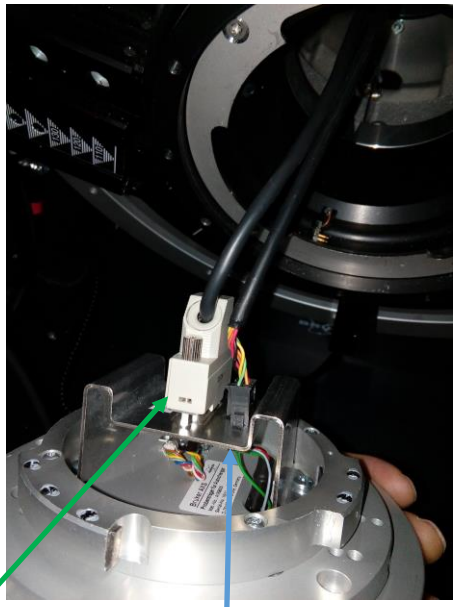
- **Switch of diffractometer at mains.**
  - You will need to remove the standard sample stage and mount the capillary spinner. Both stages uses different communication ports, first step is to switch off the instrument.
  - Ramp tube down to 20 kV / 5 mA in Diffrac.
  - Press generator button and wait that generator ramps down completely.
  - On the left hand side of the enclosure press the white standby button. Wait 10 seconds, and switch off the mains by turning the red knob.



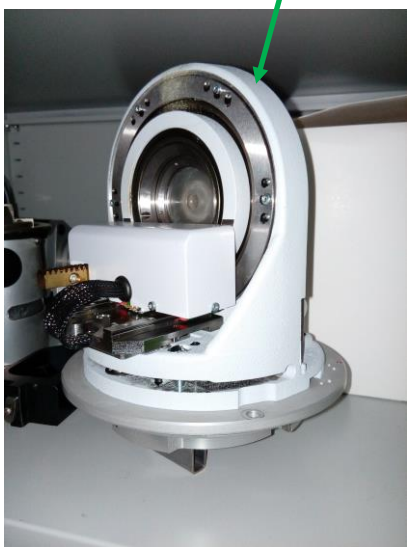
- **Remove motorized anti air scatter knife edge.**
  - The capillary spinner optionally uses a different non-motorized knife edge, you will remove the motorized knife.
  - Unclip the cable attached to the knife edge from the plastic clip on the top inside of the enclosure.

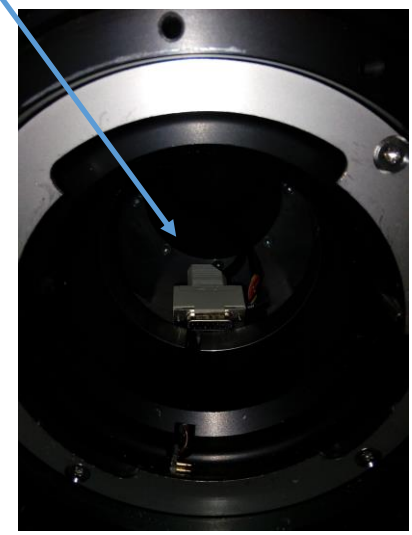
- Push the lever on the knife to vertical position and carefully remove the whole component. Lay it down somewhere on bottom right of the enclosure (**NOT anywhere under the detector**).



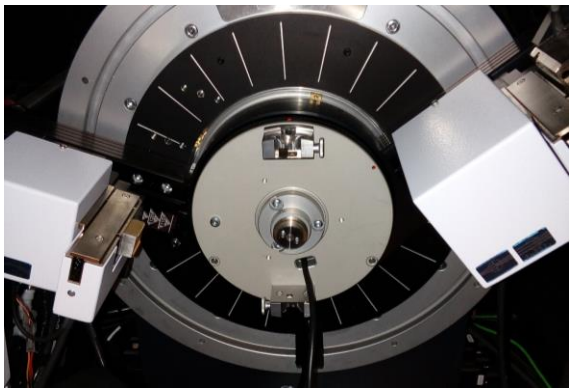


- **Remove standard sample stage.**
  - Remove the standard sample stage by unscrewing 3 screws (1 red on top and 1 further on each side at same radius), and rotating counter-clockwise until the red marks coincide. **Gently** pull the stage slightly out of the goniometer until you can see behind it (**no further!**). **CAREFUL: stage is heavy and has electronics attached behind.**
  - Take a screw driver (can be found in the cupboard on the left) and unscrew the connector to the **com port**, unclip the second cable on the **black clip**.
  - Now you can remove the stage out of the enclosure, place it in the cupboard standing **on the electronics box**. **Push the 2 cables** inside the goniometer somewhere, they are not needed for the capillary stage.





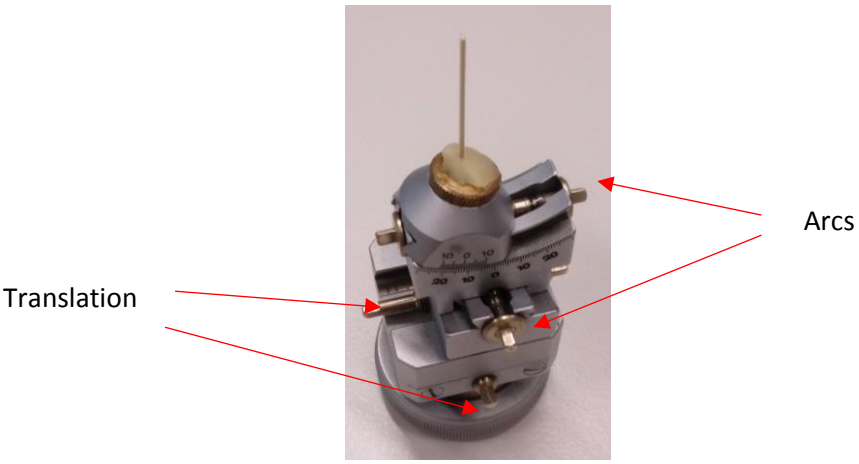
- **Mount capillary stage.**
  - Take the capillary stage (on right hand side in enclosure) and place it into the goniometer, where you just removed the standard stage. The cables of the standard stage should previously have been placed inside the goniometer somewhere. Mount the capillary spinner by matching the red marks, then turn it clockwise to fasten it. Turn it clockwise until it blocks. Place back the 3 screws you took out from the standard stage.



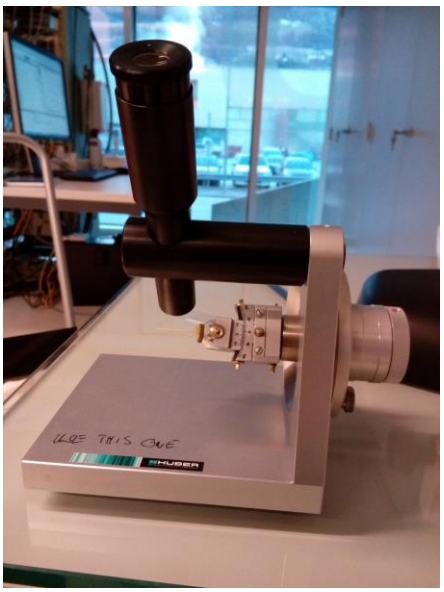
- **Switch on diffractometer at mains.**
  - Turn the mains (red knob) back to “I”.
  - Wait 10 s, then press the green button.
  - Wait until the lights on the left hand side of the door stop blinking. Then press the upper light (at this point white, with an “I” in the center).
  - The generator should be ready now (showing the radiation sign on the top light).
  - If the lower light is white instead of green the instrument is not communicating yet. If Diffrac is open, reconnect (in Diffrac or Measurement server). If Diffrac is closed, open it, and the communication should be established automatically.

- **Open Diffrac.Measurement (if not open already).**
  - Login as User, password User.
  - For brief explanation of the Diffrac interface see the manual on Bragg Brentano and GID measurements

- **Mount capillary on goniometer head.**
  - The loaded capillary needs to be mounted on a goniometer head with 2 arcs, found in the cabinet next to the Diffractometer on the left.

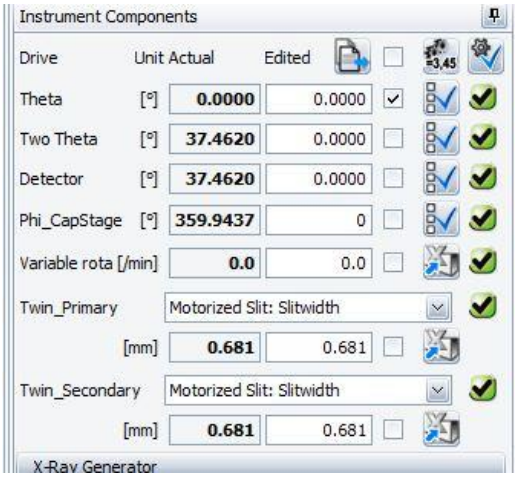


  - Use the **telescope/microscope** to pre-align the capillary. Mount the head and perform rotations of 180 ° bringing the capillary into the centre of the optic by adjusting the two translations of the **rails** with the goniometer tool found in the cabinet. Adjustment are performed when the respective motion is perpendicular to the line of sight (i.e. the optic).

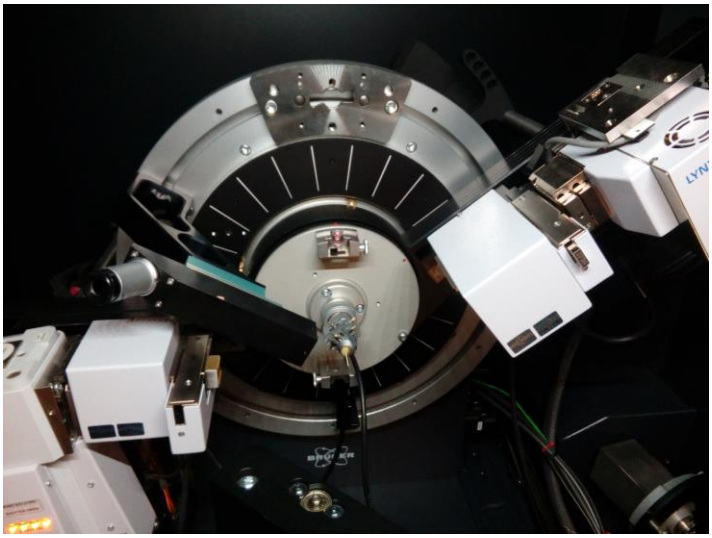


  - When the capillary no longer moves out of the centre too much upon rotation, adjust the inclination (using the goniometer tools on the 2 mutually perpendicular arcs on the goniometer head) to match the horizontal line upon rotation.
  - Iterate the two last steps until you are happy.

- **Mount capillary on the goniometer.**
  - Now carefully mount the capillary on the previously installed capillary sample stage.
  - Check for alignment and improve if necessary. For this you need to mount a microscope (in the cabinet) on the outer track of the goniometer. First you need to make place by moving the tube (theta) to 0°.
    - Check there is absolutely nothing under the tube preventing movement to 0° (other than cables). RISK OF COLISSION!
    - Move source to 0°.



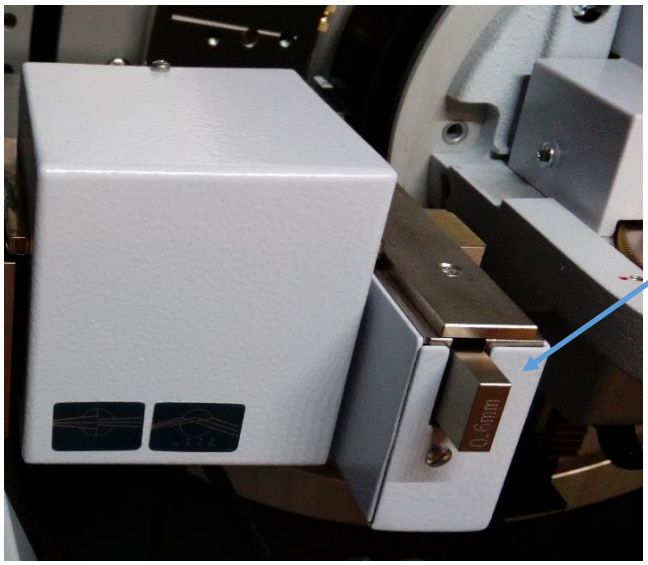
    - Mount **telescope/microscope** somewhere on outer track, close to source



    - Perform same alignment as previously on the capillary until you are happy.

- **NOW UN-MOUNT THE MICROSCOPE – RISK OF COLISSION during measurement!**

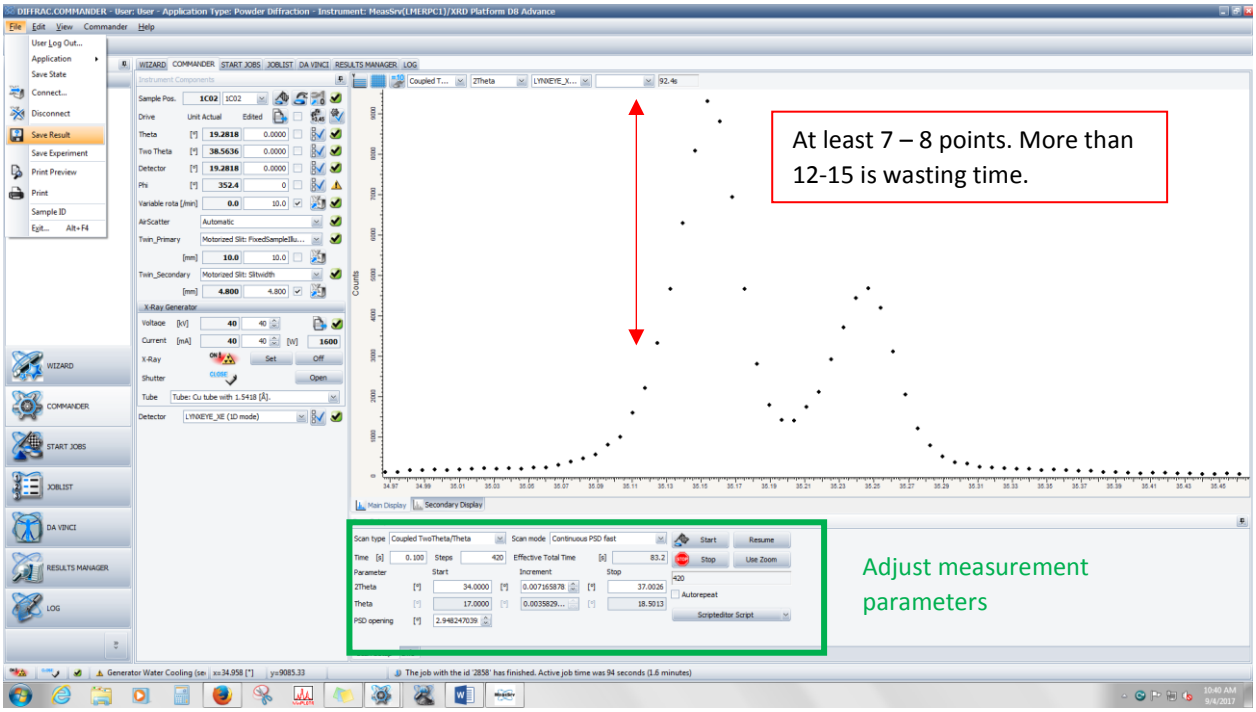
- **Change to parallel beam optics.**
  - Debye-Scherrer measurements require a parallel beam. On the D8 Advance with TWIN/TWIN optics this configuration change is down through the software, either in the Commander or the Da Vinci virtual diffractometer. You need to select the component called “Göbel mirror”.
  - Mount a slit in front of the primary optics box. Choose a slit nor larger than you capillary. Slits are found on the left and right inside the enclosure of this and the other powder diffractometers. If you need to borrow it form another instrument place it back after measurement.



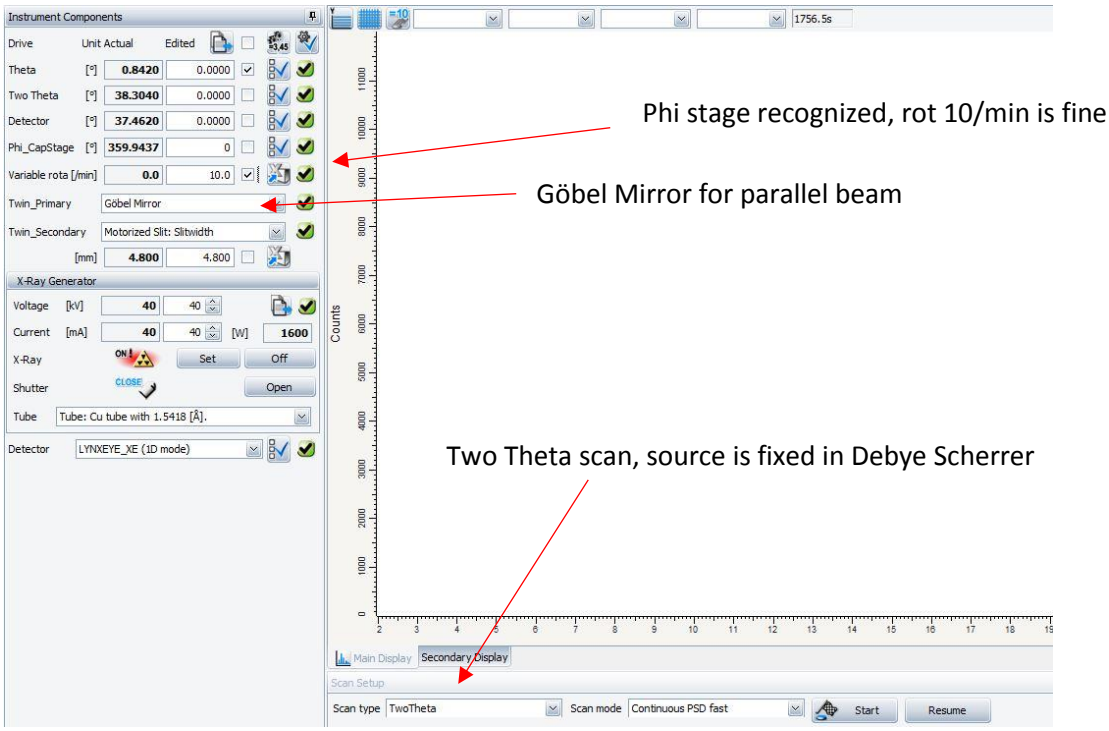
Choose appropriate slit

  - Remove the Nickel filter.
  - **Air Scatter:** Re-mount if you think you need it. Fix it in software above capillary (in fixed mode).

- **Measurement.**
  - **Check that the microscope is no longer mounted on the goniometer.**
  - 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**: The source is commonly not moved in Debye Scherrer measurements. You will choose the scan type “2Theta” in the commander.
  - **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 model data yourself or have them modelled by someone else (profile fitting, Rietveld refinement…). Anything above 12 points is usually a waste of measurement time.



- **PSD opening:** (Detector slit) Can be at the maximum, as with the slit, play around for resolution.
- **Sample rotation:** It is always better to rotate your sample, if you can. A speed of 10 rpm if reasonable.



- **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.

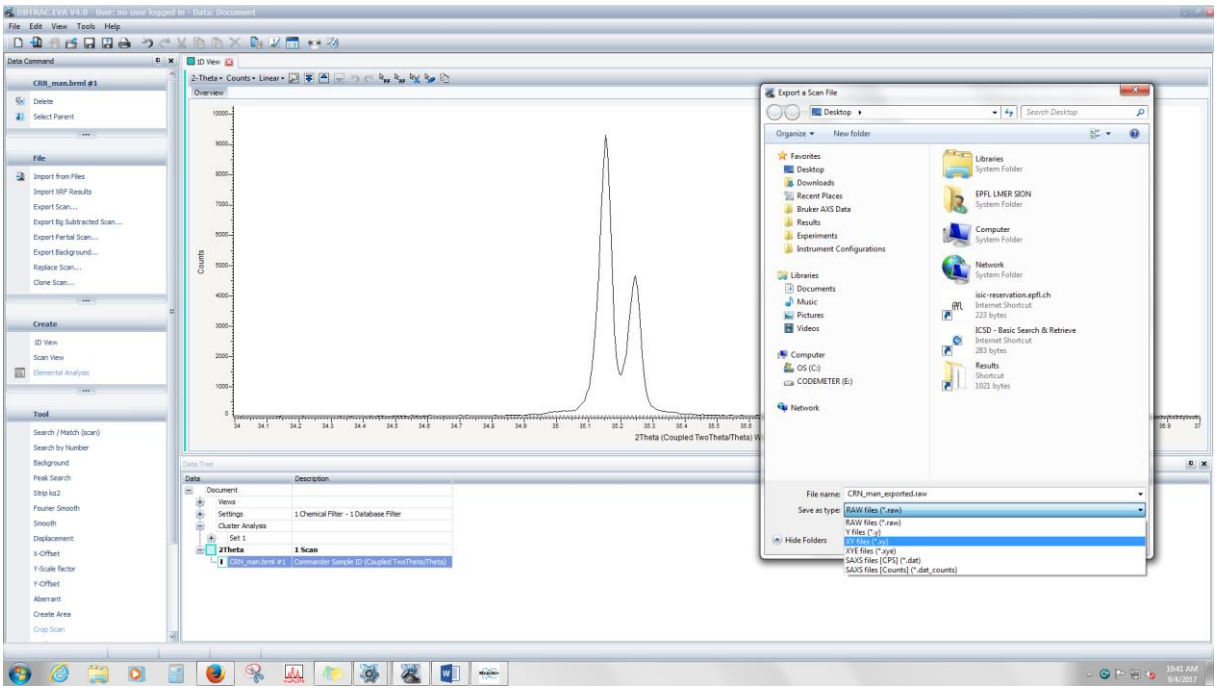
  Batch mode: You can save your measurement conditions for a respective experiment and apply them to future samples. However, one by one in Debye Scherrer mode, there is no sample changer.

- **After capillary measurement.**
  - Reverse all steps (switch of instrument, change sample stage….) to bring the instrument back into Bragg Brentano geometry for the next users.

- **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 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.


Export a Scan File
File name: CRN_man_exported.raw
Save as type: RAW files (*.raw)
RAW files (*.raw)
Y files (*.y)
XY Files (*.xy)
XYE files (*.xye)
SAXS files [CPS] (*.dat)
SAXS files [Counts] (*.dat_counts)
Hide Folders