Empyrean 3 powder diffractometer user manual (Data Collector) 2nd

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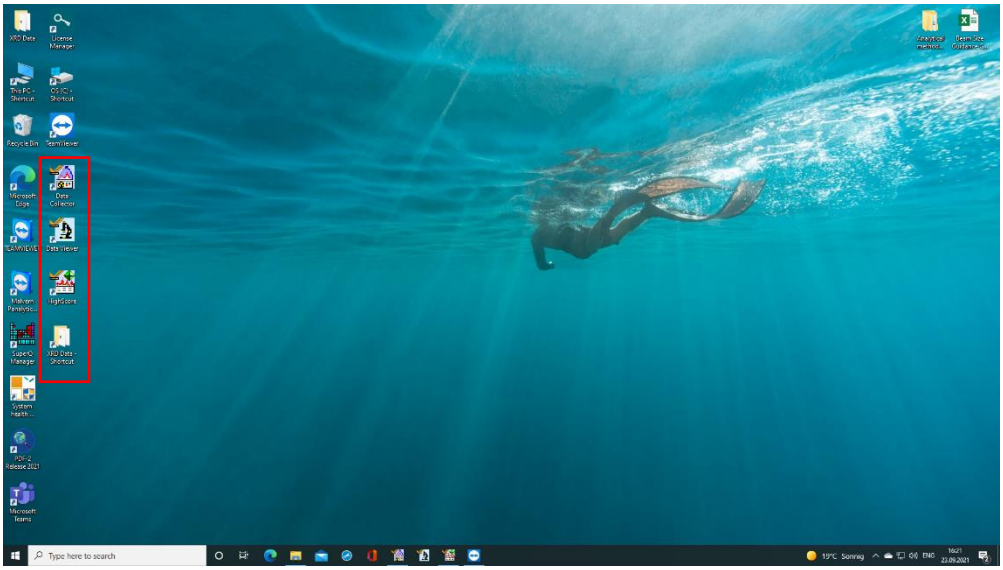
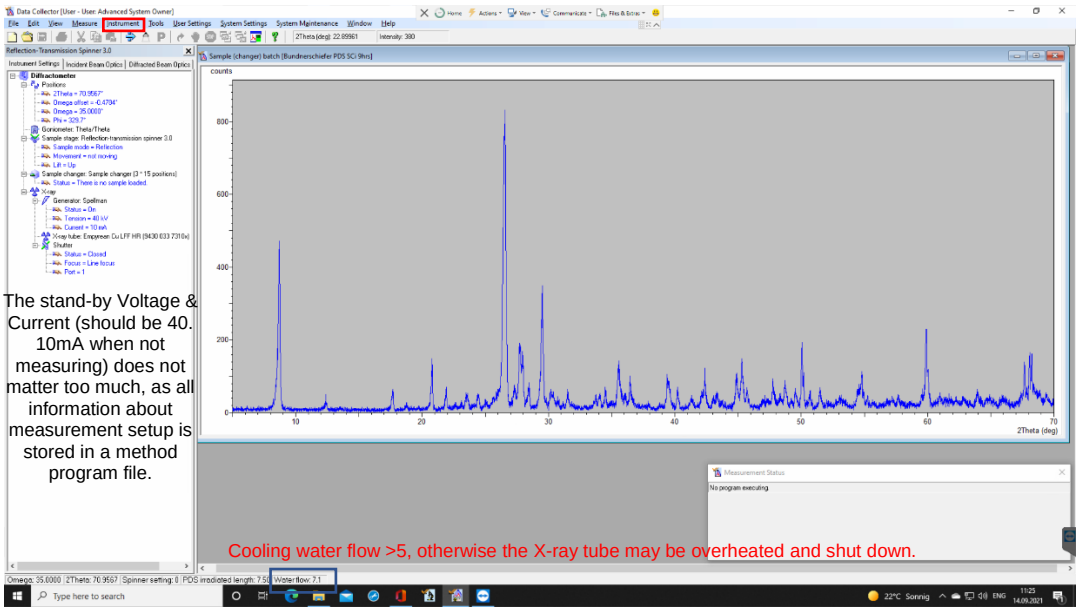
Ning Ma, NW E69, ning.ma@erdw.ethz.ch

Toolkit overview: (**Data Collector**: Taking XRD measurements, **Data viewer**: Quick check/view of results/converting formats, **HighScore**: Data processing software, for phase identification/Rietveld refinement (installed on 3 PCs, so if it takes time, please copy your data to other PC.), **XRD Data folder**: Storage of Method programs& Results). Please create and save data in your own folder.

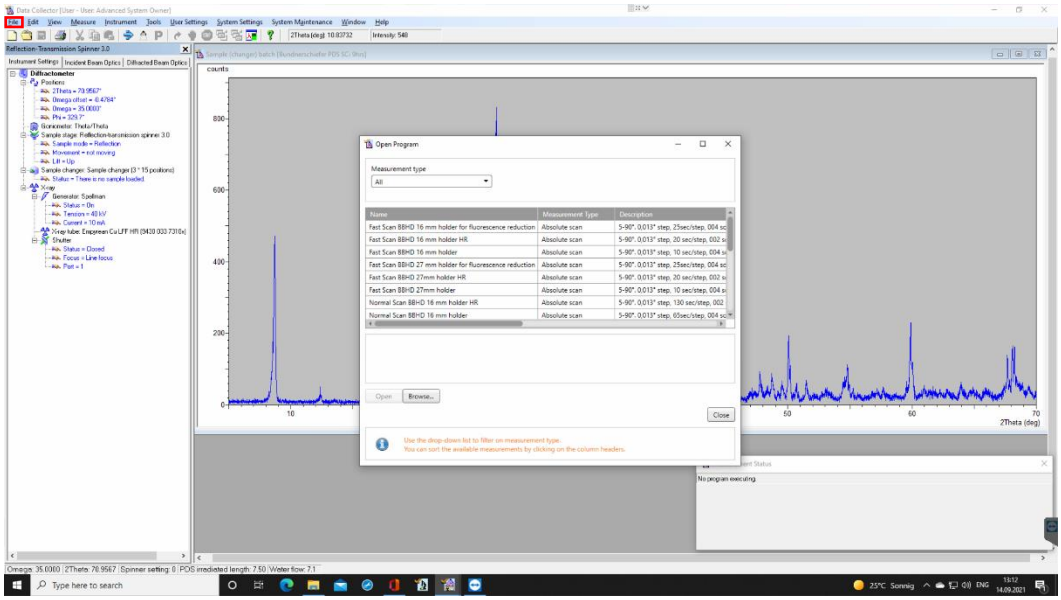


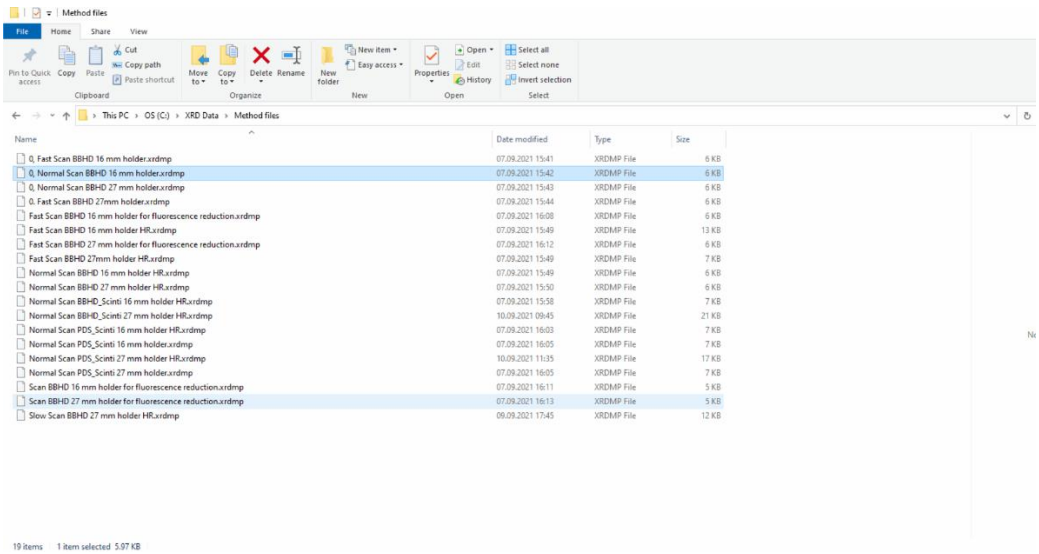
1. Open Data Collector on Desktop, Click Instrument-Connect (instrument with PC, usually already done, needs to repeat after restarting Data Collector/PC). If the system pop up error message 'PRS not found', just click yes then the left panel should appear. In case there is additional error message and fail to connect to the instrument, you need to restart the PC as well as the instrument (please ask us if you are not confident).



2. Users first create/choose a '**Method program**' which specify the conditions/ parameters of the XRD measurements. The user is recommended to copy an existing method file in own folder before further alteration.

For routine analysis one could simply refer to/slightly modify based on one of the existing Method programs by clicking File-Open/create new Program. (Default folder C:\XRD Data\Method files).





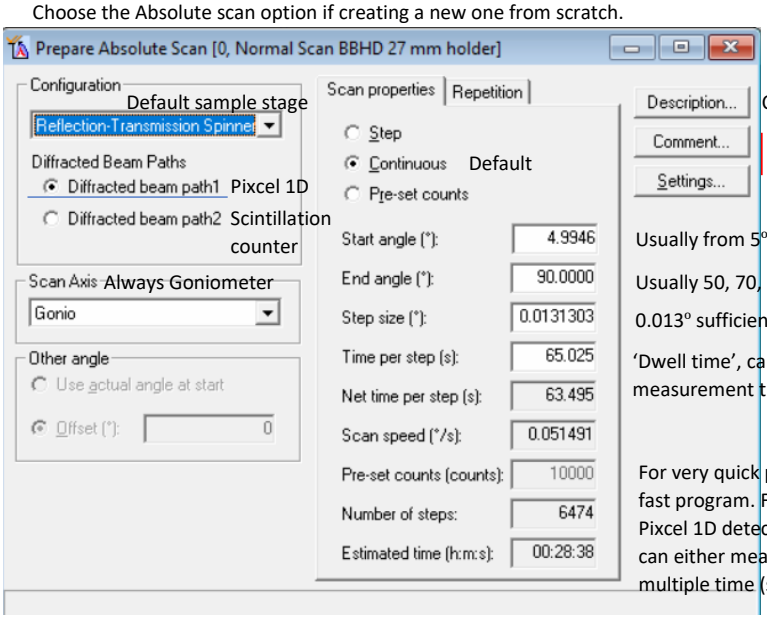
For almost all ordinary (non-fluorescence) samples (i.e. non magnetite/hematite/Fe, Co, Ni-based alloy etc, Fe-bearing silicates like fayalite is not a problem) the standard setting of XRD instrument is to use **BBHD** optical module (Brag-Brentano) coupled with a semiconductor-based **Pixcel 1D** detector (**Diffractometer path 1**).

3. Check/modify a suitable program (.xrdmp files, copy and then save in your own user folder). The most common ones are those that starts with 0. i.e. 0. Normal Scan

(speed, 30 min) BBHD (Optical module with Pixcel detector) 27mm (the largest sample) holder.

Important: the optical parts inside the instrument should match your choice and be replaced manually accordingly. There is an Excel sheet to advice the choice of optics (later). When not familiar with the relationships of optical module or exchanging parts, please ask Ma Ning or Lydia Zehnder for assistance.

Choose the Absolute scan option if creating a new one from scratch.



Can type description of setting

Usually from 5º, high direct X beam background <3º

Usually 50, 70, 90 depending on interest, max 150 º

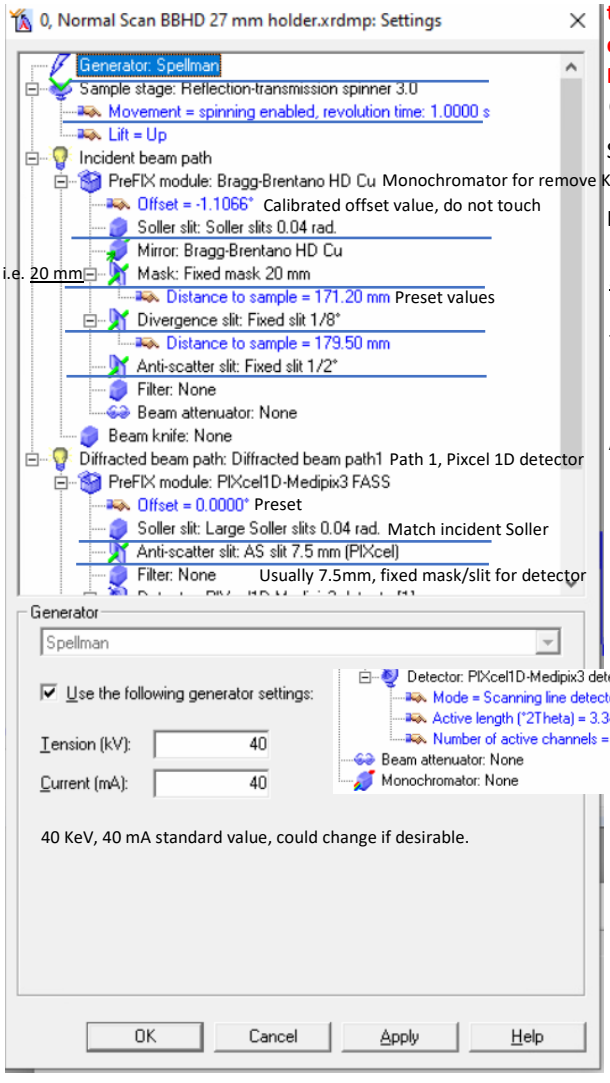
0.013º sufficient, no need to be smaller

'Dwell time', can freely adjust to change total measurement time.

For very quick phase check 5 min enough (use the fast program. For standard quality 30-60 min with Pixcel 1D detector. When higher counts required, can either measure longer or repeat measurements multiple time (see repetition).

When using standard programs, you only need to check that the actual optical parts inside instrument matches the description here (in settings). When in doubt, please ask Ma Ning or Lydia Zehnder for suggestions.

Change only underlined parameters. Do not touch parameters if you don't know what it does.



Mask controls irradiated area, i.e. 20 mm

Cu X-ray tube operated at 40 KeV, 40mA (Default)

Sample spinning (1-8 s/rev), not too fast.

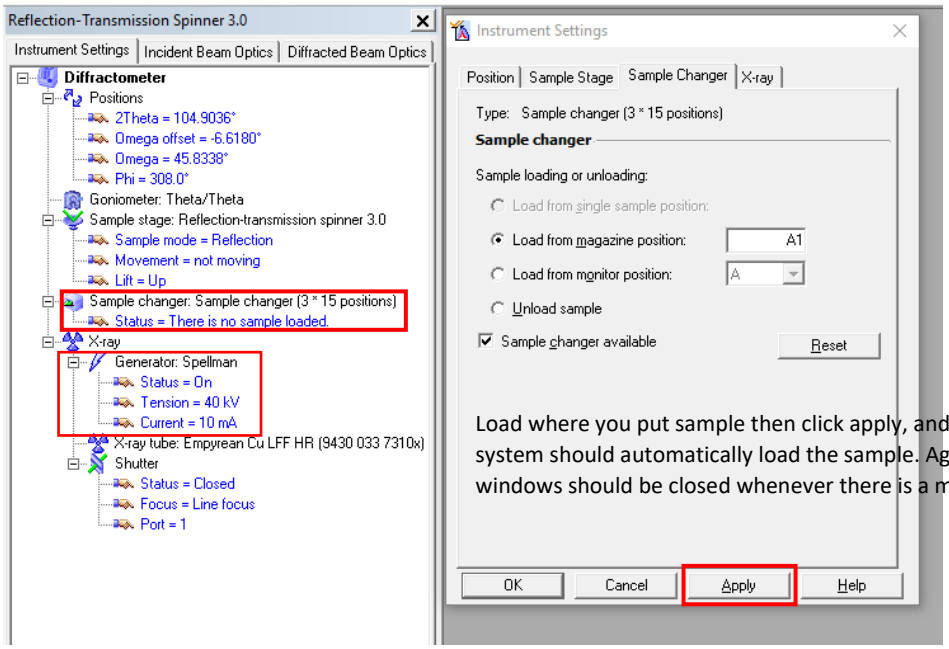
Incident optic module (BBHD, the other available one is PDS)

Soller slit 0.04 (default), restricts horizontal angular divergence of X-ray beam, 0.02 means higher resolution but lower (~half) intensity. The choice of Soller slit in incident arm should be same to diffracted arm (Physically, Soller for incidents arm looks smaller than diff arm.

Divergent slit: i.e. 1/8º controls vertical angular divergence of X-ray.
Anti-scatter slit : 1/2º (4 times larger than F(Fixed)DS)

Pixcel 1D detector, the other detector installed is Scintillation counter (with PDS) usually for fluorescence samples (requiring rather different setup)
Default setting, do not touch.
All chips on same horizontal line correspond to one 2θ point.
The angle width of 2θ segment simultaneously detected.
255 rows , ~50 um wide each.

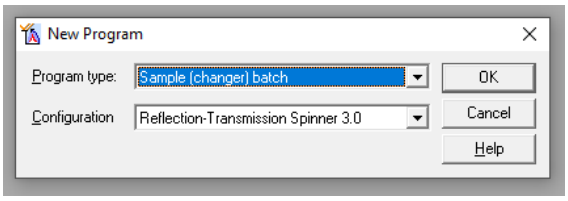
For single measurements



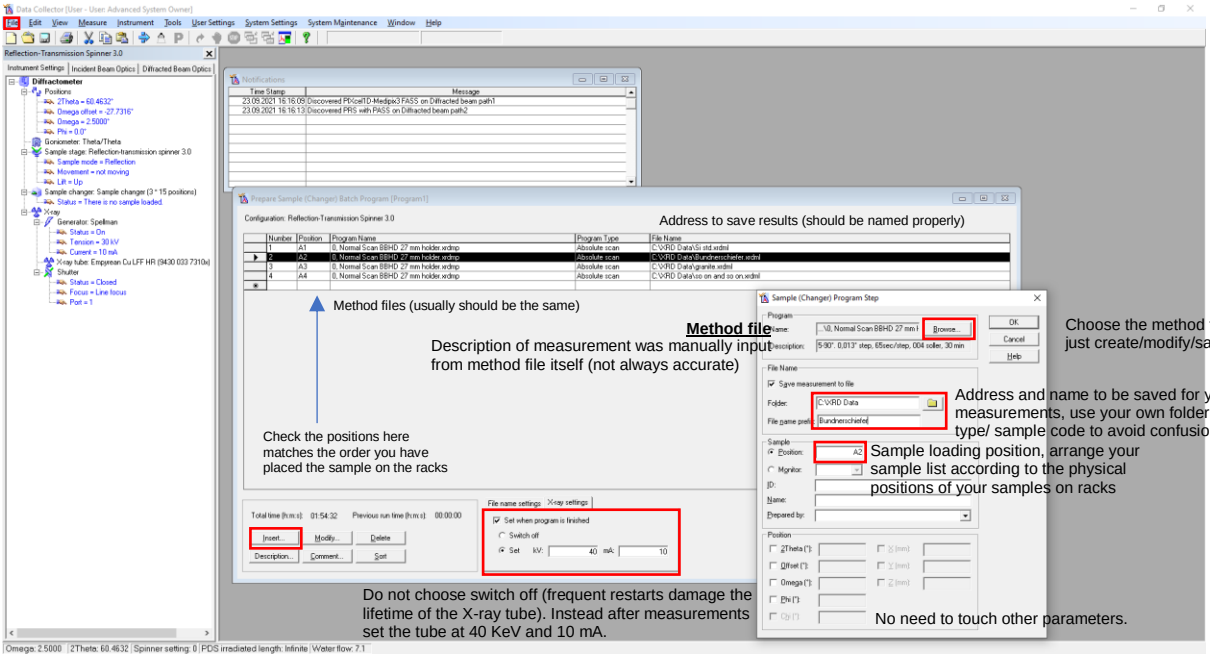
For single measurements, you can manually load the sample here (double click)

Load where you put sample then click apply, and then the system should automatically load the sample. Again windows should be closed whenever there is a movement.

For multiple samples **(recommended)**



File-new program, choices: Absolute scan (single measurement), sample exchange batch (batch file)



4. Check all parameters here and actual ones inside instrument, replace if necessary. Save this method program (in your separate folder if altered) by clicking exit and save. **For single sample measurements, you are almost ready.** Now just click Measure-program-then select the program you just saved, assign sample name and the saving address of your measurements (in your own folder!).
   A disadvantage for this is after measurement, voltage and current will not automatic decrease to stand-by value (i.e. 40 KeV, 10mA), staying at analysis condition (40

KeV, 40mA dwindles the lifetime ofX-ray tube). So we recommend all users to either 1. using this single measurement only if you can come back when finished, and reset voltage, currents back to 40, 10 (double click on left panel (X-ray, change the voltage and currents then apply, make sure the system goes back to standby value before you leave), or 2. Use the general batch (described below) where you can reset voltage and current after last measurement finished (more recommended).

5. **For batch measurements, an additional batch file is needed,** Click File-create new program-sample(exchanger) batch program. In this program, the user needs to **create a list of sample names, their rack positions, the method files (the Method file you just saved), addresses to save the measurements (again in your own folder). When done, save this (batch) program (this will be the program you use later with Measure-program)**
   Theoretically one can use different settings for different samples (by selecting different method files) within the same batch if no parts is to be exchanged, but this sounds and is just stupid, just do it in two batches.
   Repeat for all samples (one could copy the settings of the first sample (Ctrl+c the first line, Ctrl+v on next line) in the following lines and simply change the name/address and loading position.
   In total 3 sample racks (3*15) are referred as A,B,C 1-15 i.e. A1, A2 …A15, B1…B15, C1…C15. Because A1 position is often taken as the default sample unloading position (not in the case of sample exchange batch but often in single measurement),so to avoid potential crash, for batches please always leave the A1 position empty!! Just start from A2 position.
   At the end of the sample list, click the last empty line, set the system to go to 40 keV, 10 mA after the last measurement to proliferate the longevity of Cu X-ray tube.
   Note. Once the method files or the folder has been renamed, the link between batch program and method program is lost, therefore we recommend you to save all your files in a personal user folder. Save you methods before using it!

Now to load the sample(s) in **Racks**, do not try to put sample directly on sample stage!

The critical factors for sample preparation are to ensure a **flat surface and correct sample height** while avoiding possible preferred orientation of crystals (more common than you think).

6. Input sample(s) in order on sample racks (check the racks are positioned correctly), close the instrument window (remember the window must be closed whenever there is any movement inside detector e.g. initializing, sample (un)loading, to avoid potential crash).

7. For single measurement, put sample in A1 position or any other position (make sure nothing is on sample stage right now) and then click on Data Collector (left panel instrumental settings)-Sample exchanger-load sample on stage, click apply. Then monitor the instrument until the sample is loaded on sample stage (do not try to load sample by directly placing it on sample stage). Then click Measure-single measurement-the method file-input the name and address for the result data file. **Irrespective if the optical module written in the method file is not completely the same with last measurements, the instrument would always pop out a warning window asking you to check/replace certain parts. Think about this, check if necessary before clicking yes.**
   After this, you should hear the window of instrument locked (it will be impossible to unlock the door during measurements (namely when the X-ray shutter is open. High V, A is almost always on and is not a problem as shutter is usually closed, so it will be completely stupid-proof), and the instrument starts to position/initializing itself by rotating both arms to the starting position (i.e. $2\theta = 5^{o}$).
   Then you should wait at least until the first data point (within 1 min)/ first peaks to see the instrument runs normally. Some hints: the background (bremsstrahlung) intensity is usually zig-zaged and ranging 400-1500 cps, intensity of the peak can reach >10'000-100'000 (for Pixcel1D detector saturation should be quite rare).
8. For batch measurements, click measure-batch measurements-select your method file click OK. Do the same for the warning message and observe the instrument to initializing. The measurement is saved every minute, so you could already open it with data viewer if desired but usually the display in data collector is sufficient.
9. When measurements are finished, open data file (data viewer as default) for quick view/check, or drag the data file to HighScore for phase identification/Rietveld refinement.

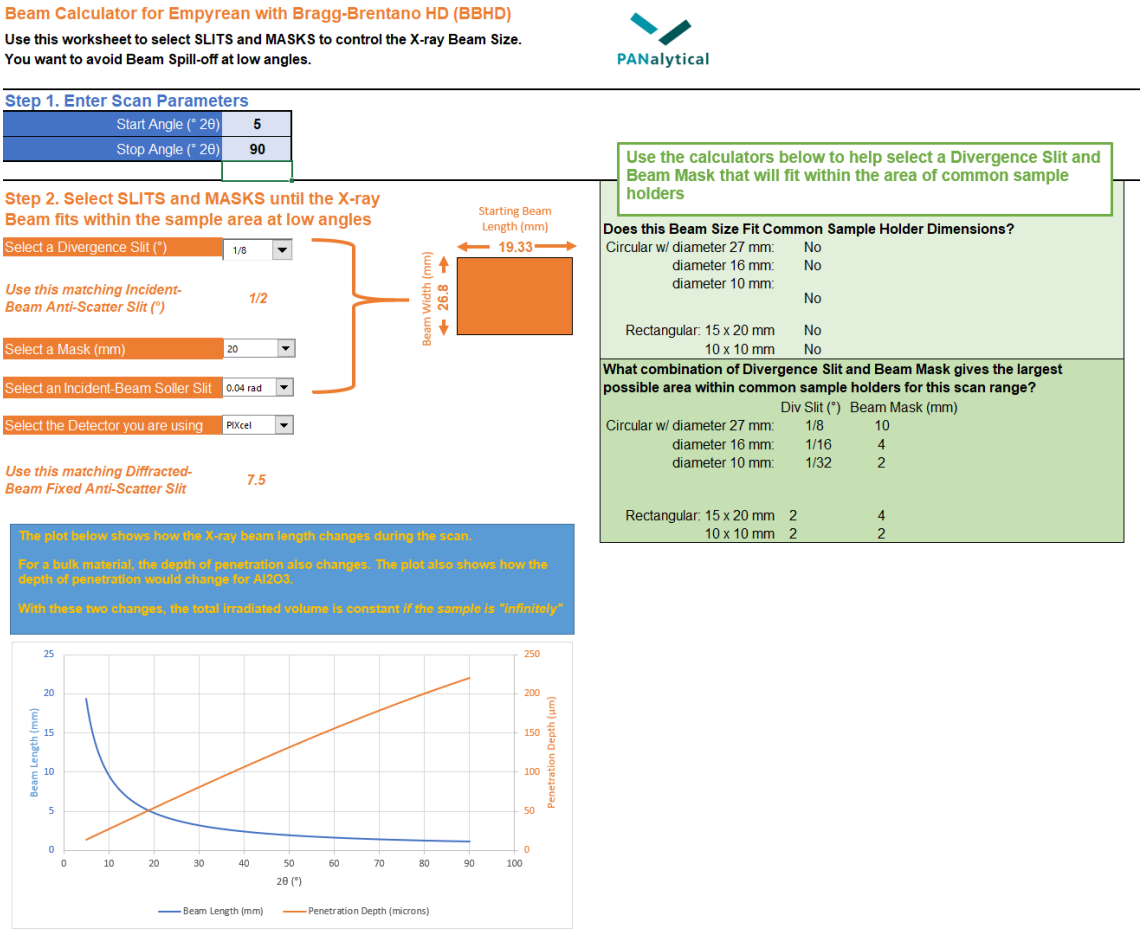
Insert 1. Choice of optical modules

For user interested in understanding the choice and relationship of optical parts, please consult the excel spreadsheet: Beam Size Guidance Calculator for Empyrean (Desktop), first chose the correct optical module (BBHD/PDS) excel sheet, specify 2θ angle range, choose divergence slit/mask/soller/detector by consulting the green shaded areas.

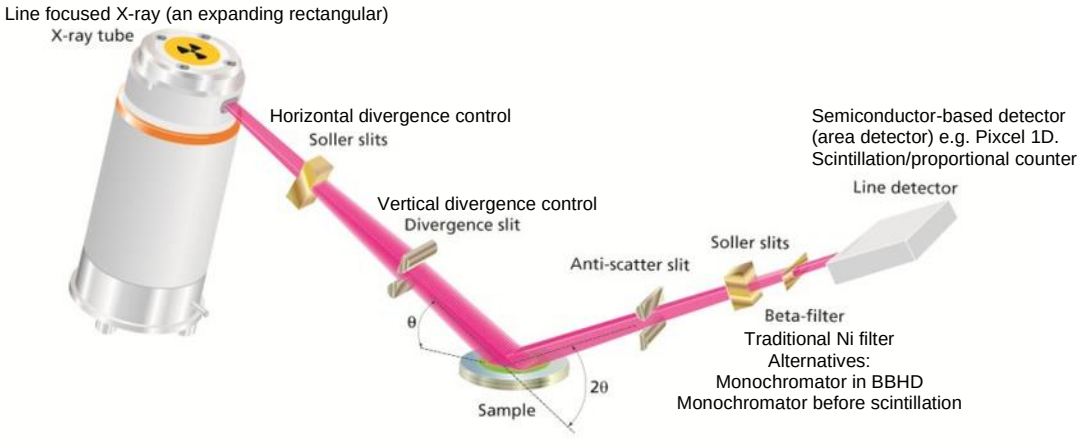
The larger the optical parts, the higher the signal. But this is restricted by the actual dimension your sample and desired resolution. Over-irradiating sample holder will produce intense interference that didn't come from your sample. On the other hand, the smaller the optical parts, the higher the resolution but lower in signal.

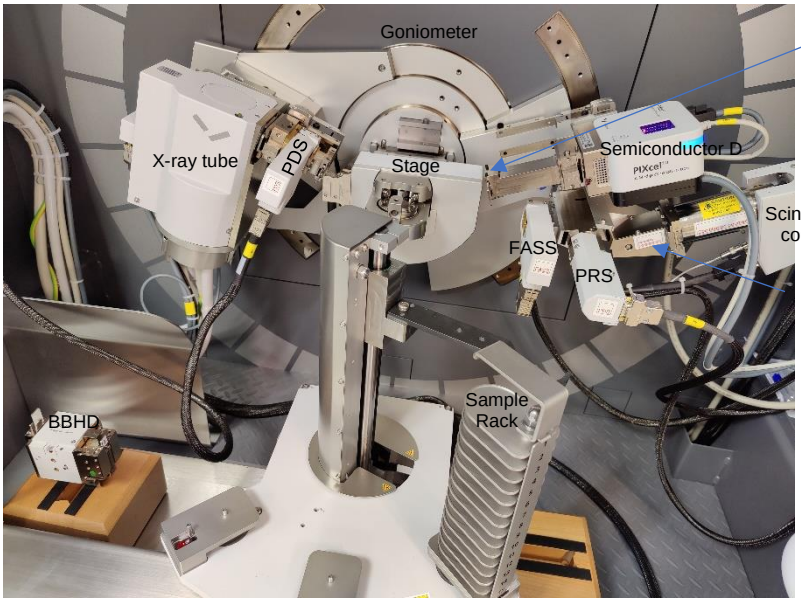
As demonstrated in the figures, for fixed slits the beam width (defined by Soller slits) remains constant over entire scan, but the beam length (the length along X-ray propagating direction) decreases significantly at higher angles. The effective penetration depth (of cause sample-specific) on the other hand increases linearly with angle, reaching ~200 μm at 90º. This means when the low background Si-wafer sample holder is not used, then your sample should exceed this thickness.

At this stage you may have noticed that despite measuring the same sample under the same X-ray, 'the effective volume' probed by X-ray is actually angle specific, therefore the measured intensity does not yet reflect the true 'intensity' or structure factor of your sample, and the relative intensity ratios among different reflections or your measurements vs. reference of database is thus dependent on the condition/ measurement setup. This will become relevant later for Rietveld refinement.



Insert 2. Instrumental design and optical modules

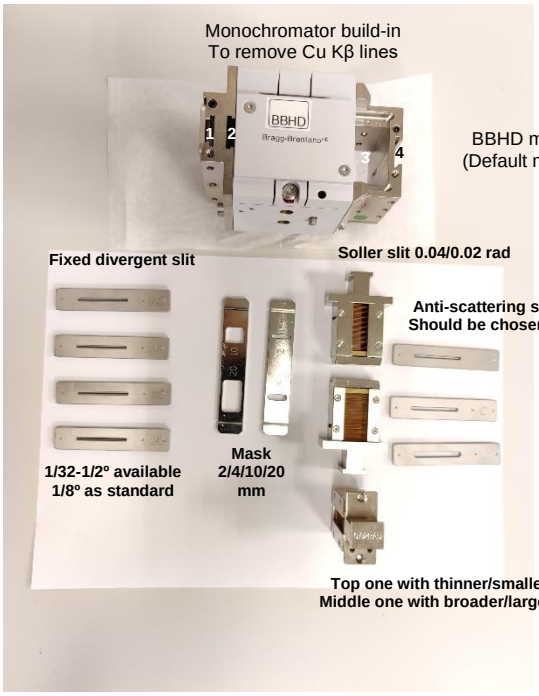




For BBHD one always use fixed slits, while as for PDS the user specify a irradiation area so that during measurement, the automated slits inside PDS is gradually adjusted to sustain a fixed irradiation area.

Optical parts at Standard settings:
1. Fixed divergent slit (1/8º)
2. Mask (20 mm)
3. Soller slit (0.04 rad, for both)
4. Anti-scatter slits (1/2º, 4 times FDS)

On diffracted beam side
Fixed receiving slit: P7.5mm



Quick reminder to a measurement:

0. Instrument-Connect
1. File-method program for single measurements/ File-method program, File-batch program. Check and save.
2. Load sample on racks (you can do this first as well).
3. Measure-single/batch programs. Measurement starts.
4. Check & confirming optical modules, wait for instrumental initialization and first measurement point.