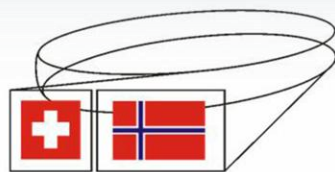


# Powder Diffraction Data Collection and Data Processing

1

PILATUS@SNBL



Swiss-Norwegian Beam Lines  
at ESRF

# Data Collection

2

- Pylatus software on ldo1pilatus should be running together with Camserver. SPEC sessions EXPERIMENT, RHMOTORS, RH\_Musst, RH\_Euro have to be active on Snbla1 computer.
- Set the parameters of data collection
- Set position of the detector
- Start data collection

The screenshot shows the Pylatus software window with the 'Set diffraction experiment' dialog box open. The dialog box has a sidebar with 'User', 'Staff', and 'Guru' tabs. The 'Guru' tab is selected. The dialog box contains the following fields and controls:

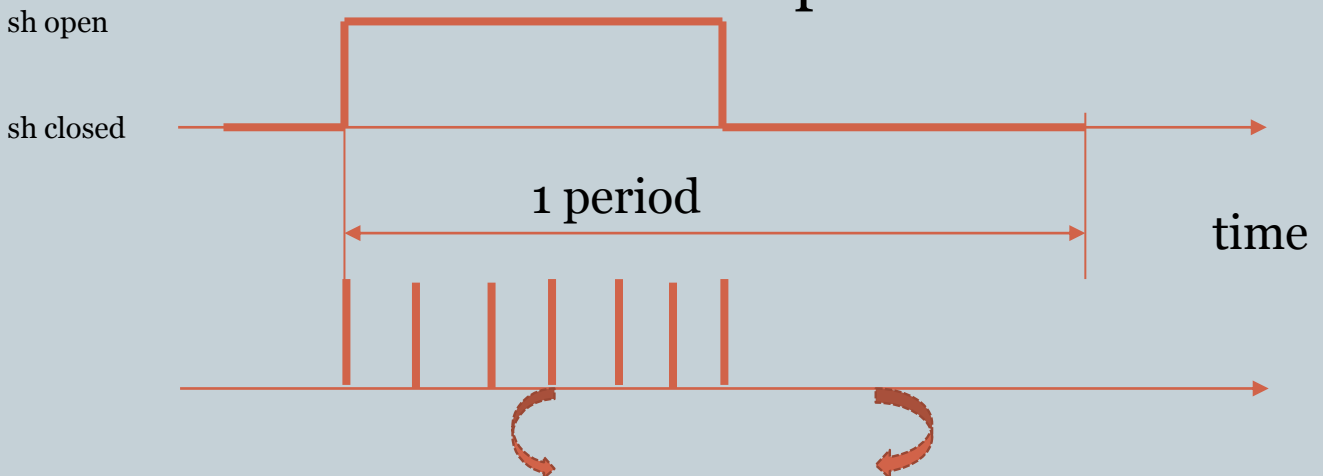
- File base name:** fege
- Folder name:** tests/det
- Exposure time per frame (sec):** 1.0000
- Number of frames per period:** 180
- Number of periods:** 1
- $\Delta \phi$  per image (deg):** 0.5000
- Starting  $\phi_0$  (deg):** 0.0000
- Detector horizontal position:** 0.0000
- Detector tilt:** 0.0000
- Frame horizontal position:** 0.0000
- Detector vertical position:** 0.0000
- Time for the data collection (min):** 3.0
- $2\theta$  at the top of the detector (deg):** 40.5913

At the bottom of the dialog box, there are two buttons: 'Start data collection' and 'Abort data collection'. Below these buttons is a logo for 'Swiss-Norwegian Beam Lines at ESRF' and a green circular indicator with the text 'Experiment is running'.

# Parameters of powder experiment

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- Diffraction experiment is a set of periods - repetitions.
- During 1<sup>st</sup> half of the period  $\varphi$ -axis moves clockwise and detector collects images according to time and step specified.
- During 2<sup>nd</sup> half of the period  $\varphi$ -axis moves anti-clockwise to initial position and the diffractometer prepares to repeat the measurement at the next period



# Recommendations on data collection strategy

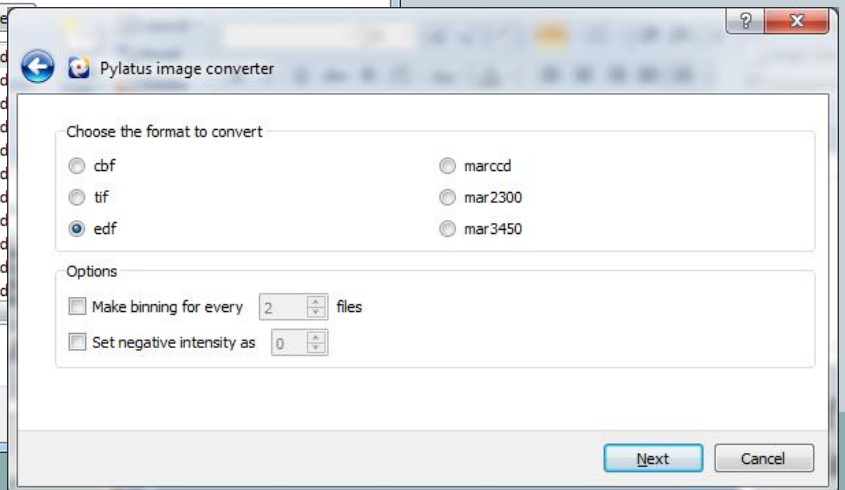
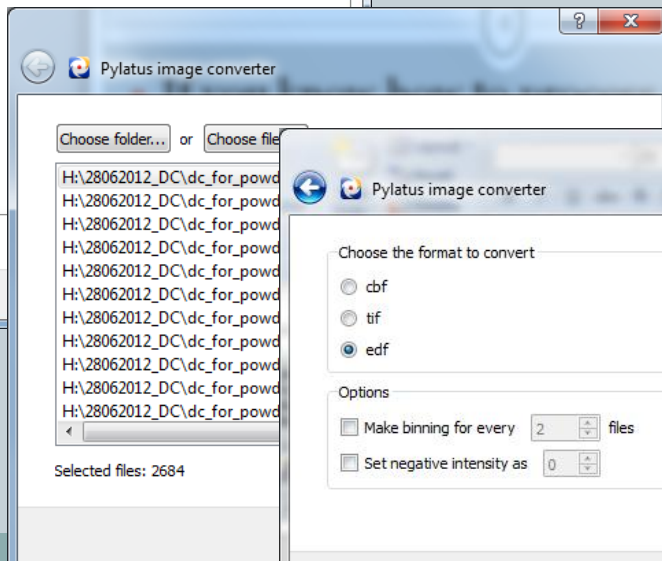
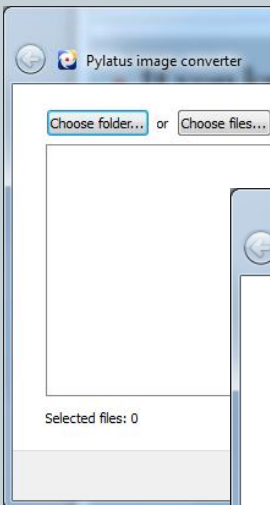
4

- Measure a standard LaB6 sample at a sample to detector distance of your experiment.
- Note  $2\Theta$  value calculated by Pylatus software.
- If you want to have high-angle data – move the detector up vertically.
- If you need better angular resolution – increase sample to detector distance by moving the detector or frame horizontally.
- Before moving diffractometer components be sure that there would be no collisions.
- If you are OK with quality of a still image, set 1 period and ask for the number of images that is equal or bigger than the required number of diffraction patterns.
- If you have to rotate your sample during a temperature ramp – use 1 image per period and ask for the number of periods equal or bigger than the number of diffraction patterns you need.

# Data Conversion

5

- If you know how to process miniCBF data – you do not need any conversion
- If you have to work with Fit2D you need to convert your data to edf or MAR or TIF format.
- We offer users SNBL Tool Box with “Converter” function for this procedure.

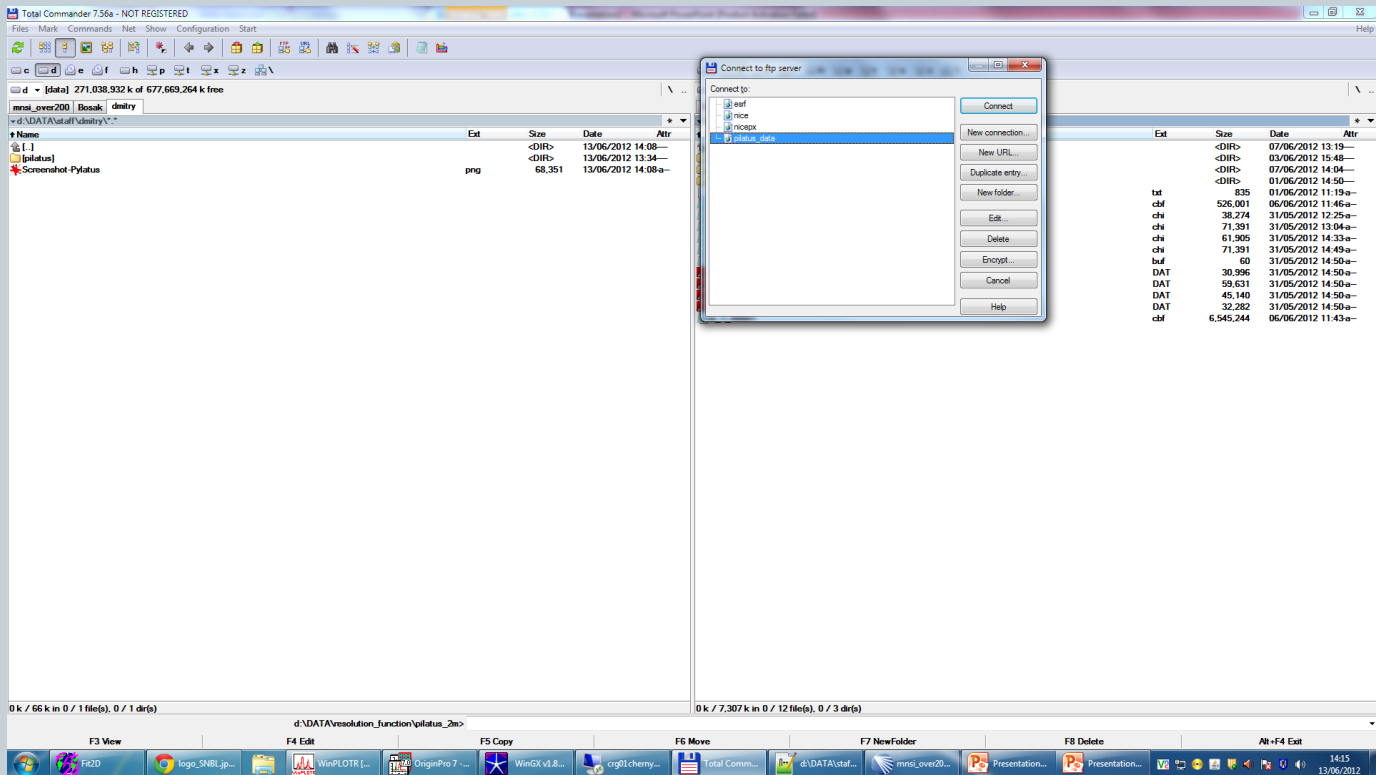


# Data Transfer

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Your data are stored in the folder you have defined setting data collection. You should copy your data to wbm01bern for processing.

To copy the data start Total Commander on wbm01bern and connect to ftp server **pilatus\_data** (net->ftp connect)



Having ftp server **pilatus\_data** connected, find your folder and transfer the data

**NOW THE DIRECT LINK BETWEEN TWO COMPUTERS IS SET. PILATUS PPU is drive Y:**

# Fit2D

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- Calibrate the detector with LaB6. From the main menu of the Fit2D select “Powder diffraction (2D)”, input your LaB6-data, press “calibrant”, “LaB6”, enter good estimates of the distance from sample to detector and wavelength and 172 microns pixel size . Select “refine X-ray wavelength” only if d is <200 mm, otherwise fix it to the known value. In the graphical window define an inner ring and click below to finish. Fit2D will refine the detector parameters.
- Integrate your data. Exit to the main menu without existing Fit2D, click “File series”, “Integrate”, select the first and the last images of the series, choose an increment 1, accept the defaults, define a mask if necessary, accept the calibrated detector parameters and defaults (e.g. apply geometrical and polarization corrections). Answer “yes” to save each integrated scan to file, select “CHILOT” and define “chi” as an output file extension. Wait until the integration is finished.

# Fesi powder

FIT2D GRAPHICS WINDOW

EXPERIMENTAL GEOMETRY

CONTROL FORM

O.K. CANCEL ? HELP INFO

DESCRIPTIONS	VALUES	CHANGE
SIZE OF HORIZONTAL PIXELS (MICRONS)	172.0000	X-PIXEL SIZE
SIZE OF VERTICAL PIXELS (MICRONS)	172.0000	Y-PIXEL SIZE
SAMPLE TO DETECTOR DISTANCE (MM)	233.0000	DISTANCE
WAVELENGTH (ANGSTROMS)	0.704000	WAVELENGTH
X-PIXEL COORDINATE OF DIRECT BEAM	731.6805	X-BEAM CENTRE
Y-PIXEL COORDINATE OF DIRECT BEAM	-3.535250	Y-BEAM CENTRE
ROTATION ANGLE OF TILTING PLANE (DEGREES)	75.87579	TILT ROTATION
ANGLE OF DETECTOR TILT IN PLANE (DEGREES)	0.222505	ANGLE OF TILT

Click on variable to change, or 'O.K.'



# Finalization of data processing

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- With SNBL Tool Box (Sigma Scaler) calculate errors and normalize intensities to the monitor or background values.
- This procedure will also rename you files from \*.chi to \*.dat for Fullprof.

