SAXS_toolbox Tutorial

I. Introduction

This tutorial is based on SAXS data acquired on a Xenocs/Xeuss2.0 device equipped with a Pilatus 1M detector from Dectris. In this tutorial, we will process SAXS data corresponding to Au nanoparticles dispersed in toluene.

The data set contains the transmission and scattering data for :

- Silver behenate, used for detector calibration

- hexane, used for intensity calibrations

- toluene used as reference

- 2 samples of nanoparticles.

Table 1 gives the correspondance between file numbers and type of measurements.

		Transmission	Measure
Detector calibration	empty beam	2	X
	Ag behenate	X	3
Intensity calibration	empty cell hexane	14	15
	hexane	17	18
reference	toluene	99	100
sample	Ech 112	102	103
	Ech 113	105	106

Table 1 : Correspondance between file numbers and type of measurement

II. Procedure

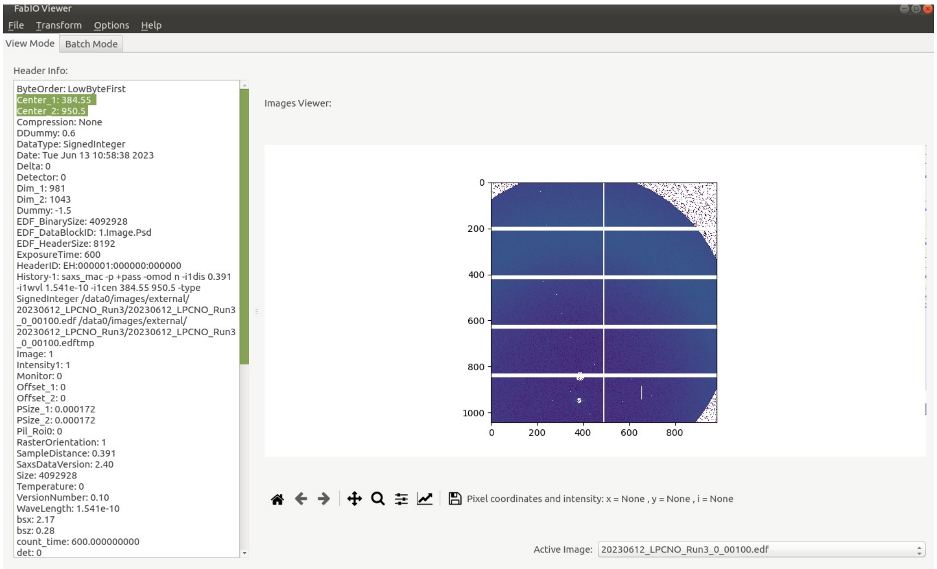
I. Preliminaries

I.1 Set working directory

As a start, use **Menu/set Working Directory** to define the path to your data files. From now on, the program will use this directory as default path.

I.2 Check/perform detector calibration

Before starting the data reduction, it is good practice to check wether the meta data from data file contain the calibration information. To do so, you can open an image using **Inspect Data/Open and view image,** and select a data file from your data set (in our case we use frame 100.edf). Check for the presence/absence of calibration information. If calibration information are present, take note of the text keys giving access to the following items: wavelength, distance, beam center coordinates, frame size, pixel size. Fill the “Experiment Description” tab with the corresponding keys, select an image from your dataset, and click on the “Apply Calibration and generate poni file” button (see Figure 2). Note that default text keys should correspond to most Pilatus detectors (SWING @ SOLEIL, SAXS @ LGC,…)



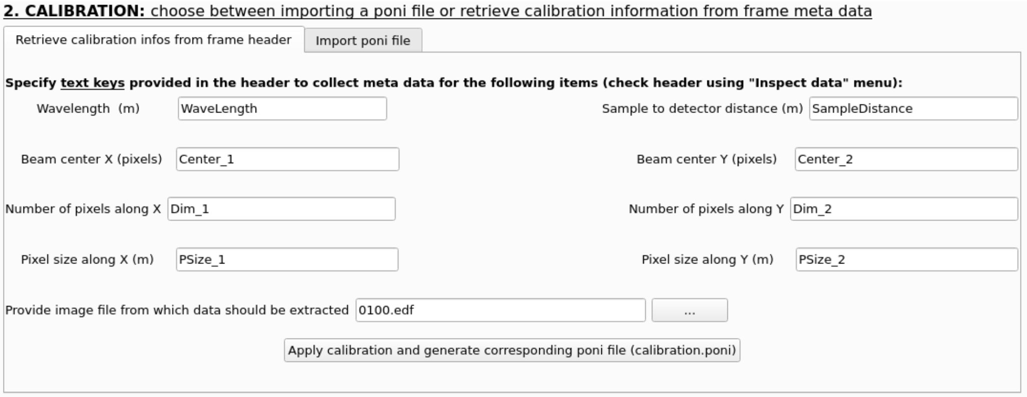
Header Info:

```
ByteOrder: LowByteFirst
Center_1: 384.55
Center_2: 950.5
Compression: None
DDummy: 0.6
DataType: SignedInteger
Date: Tue Jun 13 10:58:38 2023
Delta: 0
Detector: 0
Dim_1: 981
Dim_2: 1043
Dummy: -1.5
EDF_BinarySize: 4092928
EDF_DataBlockID: 1.Image.Psd
EDF_HeaderSize: 8192
ExposureTime: 600
HeaderID: EH:000001:000000:000000
History-1: saxs_mac -p +pass -omod n -i1dis 0.391
-i1wvl 1.541e-10 -i1cen 384.55 950.5 -type
SignedInteger /data0/images/external/
20230612_LPCNO_Run3/20230612_LPCNO_Run3
_0_00100.edf /data0/images/external/
20230612_LPCNO_Run3/20230612_LPCNO_Run3
_0_00100.edftmp
Image: 1
Intensity1: 1
Monitor: 0
Offset_1: 0
Offset_2: 0
PSize_1: 0.000172
PSize_2: 0.000172
Pil_Roi0: 0
RasterOrientation: 1
SampleDistance: 0.391
SaxsDataVersion: 2.40
Size: 4092928
Temperature: 0
VersionNumber: 0.10
WaveLength: 1.541e-10
bsx: 2.17
bsz: 0.28
count_time: 600.000000000
det: 0
```

Pixel coordinates and intensity: x = None , y = None , i = None

Active Image: 20230612_LPCNO_Run3_0_00100.edf

Figure 1 : Fabio_viewer interface and visualization of meta-data


Retrieve calibration infos from frame header
Import poni file
...
Apply calibration and generate corresponding poni file (calibration.poni)

2. CALIBRATION: choose between importing a poni file or retrieve calibration information from frame meta data

Specify text keys provided in the header to collect meta data for the following items (check header using "Inspect data" menu):

Wavelength (m) WaveLength

Sample to detector distance (m) SampleDistance

Beam center X (pixels) Center_1

Beam center Y (pixels) Center_2

Number of pixels along X Dim_1

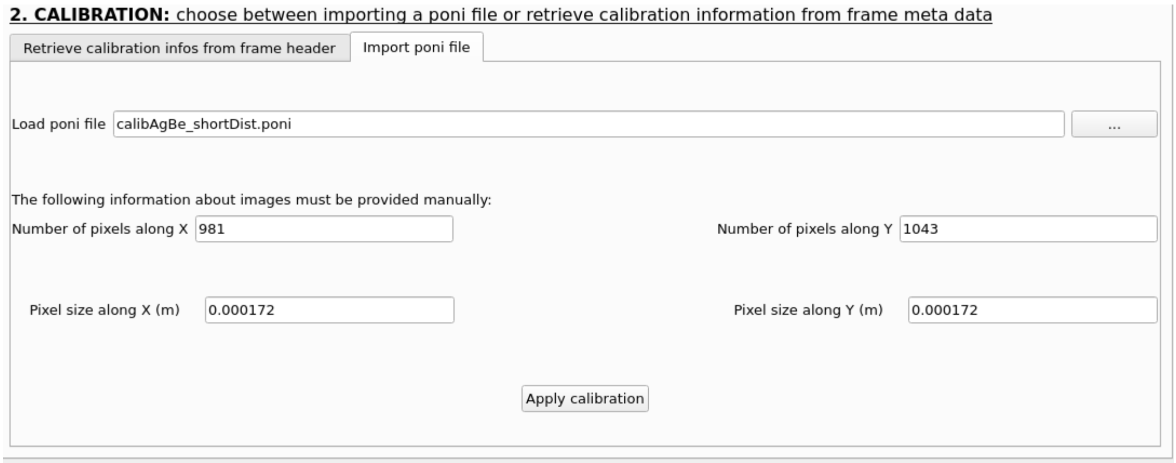
Number of pixels along Y Dim_2

Pixel size along X (m) PSize_1

Pixel size along Y (m) PSize_2

Provide image file from which data should be extracted 0100.edf

Figure 2: Calibration of images with meta data



2. CALIBRATION: choose between importing a poni file or retrieve calibration information from frame meta data

Load poni file calibAgBe_shortDist.poni

The following information about images must be provided manually:

Number of pixels along X 981

Number of pixels along Y 1043

Pixel size along X (m) 0.000172

Pixel size along Y (m) 0.000172

Figure 3: Calibration of data with a poni file

When the calibration information are absent, it is necessary to perform the calibration using **pyFAI/Detector Calibration** (see SAXS_toolbox documentation for more details). Additional information such as pixel sizes and frame dimensions must be provided by the user, as shown in Figure 3.

I.3 Generate mask and load it

A last preliminary step consists in providing a mask file, either using an image file via **pyFAI (pyFAI/Generate mask/using an image file),** either through the conversion of a foxtrot mask file **(pyFAI/Generate mask/convert foxtrot mask file)**.

In this tutorial, no mask file is available. Activate the function **pyFAI/Generate mask/using an image file** and select file 0018.edf (hexane measurement) to generate the mask. Here we select the file that contains a scattering signal as homogeneous as possible, in order to detect easily the bad pixels.

Perform the following steps

- mask below 0 intensity (automatically masks the dead zones of the detector + some of the dead pixels – but not all of them)

- create a circular mask around the beamstop

- use pencil tool to mask additional aeras (remaining bad pixels, shadows,…)

- inspect the frame using zoom tool and seek for bad/dead pixels

- use polygon tool to mask the shadowed areas in the frame corner

- save your mask in edf format by clicking the disk icon and then « Save mask and quit »

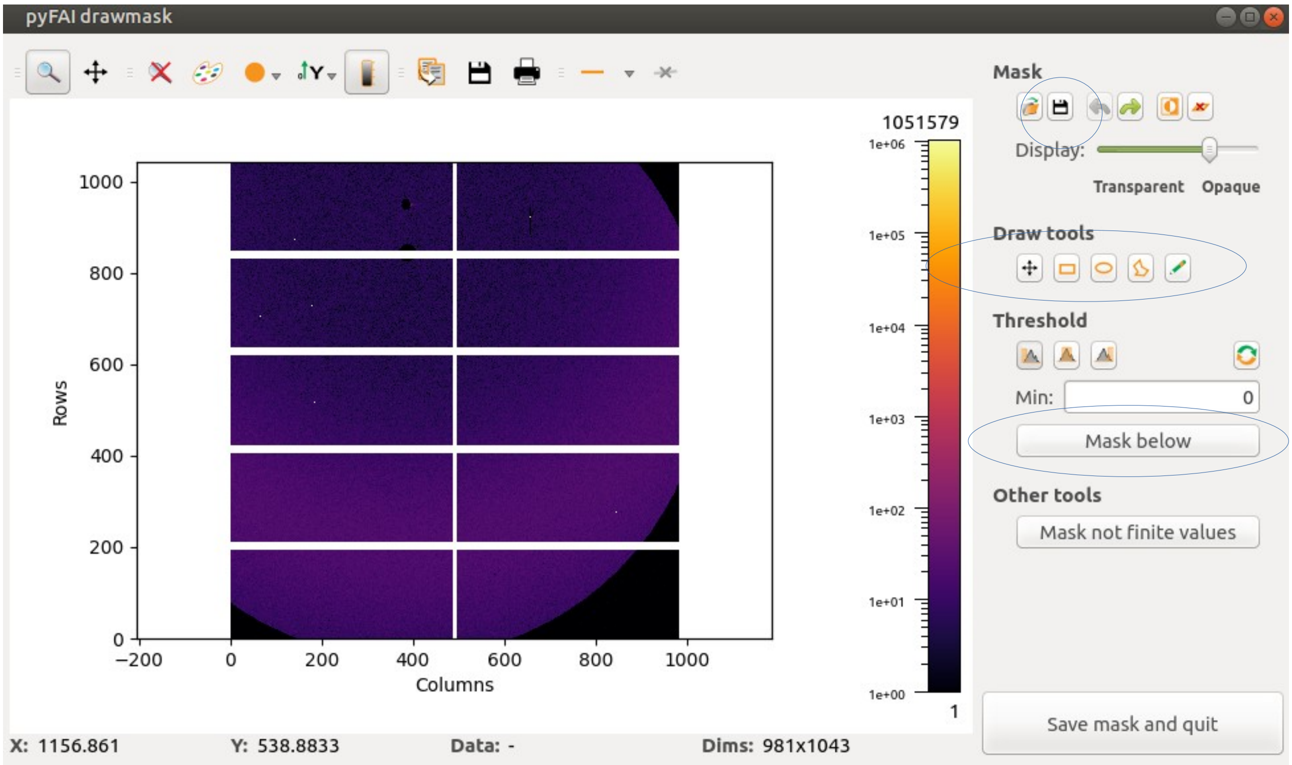


Figure 4 : pyFAI drawmask interface

After edition, the final mask shoud look like the one visible in the figure below :

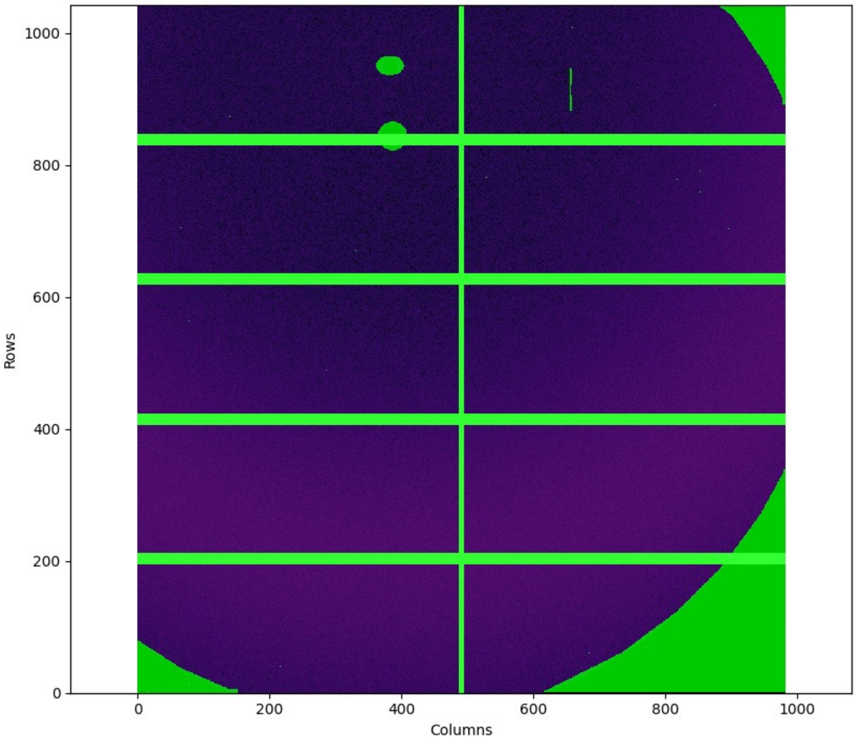
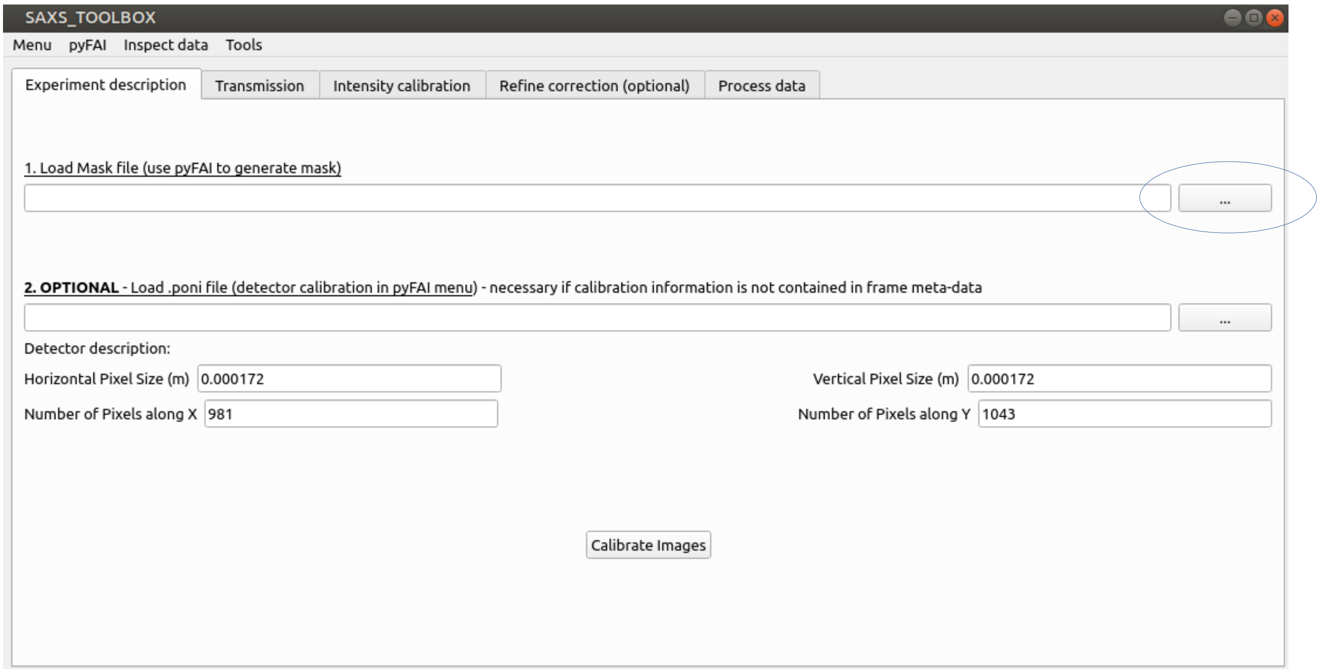


Figure 5 : View of the mask generated with pyFAI

Once the mask file is generated, load the mask file in the tab Experiment Description from SAXS_toolbox interface.



1. Load Mask file (use pyFAI to generate mask)

2. OPTIONAL - Load .poni file (detector calibration in pyFAI menu) - necessary if calibration information is not contained in frame meta-data

Detector description:

Horizontal Pixel Size (m) 0.000172

Vertical Pixel Size (m) 0.000172

Number of Pixels along X 981

Number of Pixels along Y 1043

Figure 6 : Experiment description tab: load mask file + optional .poni file

When the mask is loaded, a confirmation message appears in the console (note that an error message also appears when the mask file is not provided)

II.2 Provide correction information

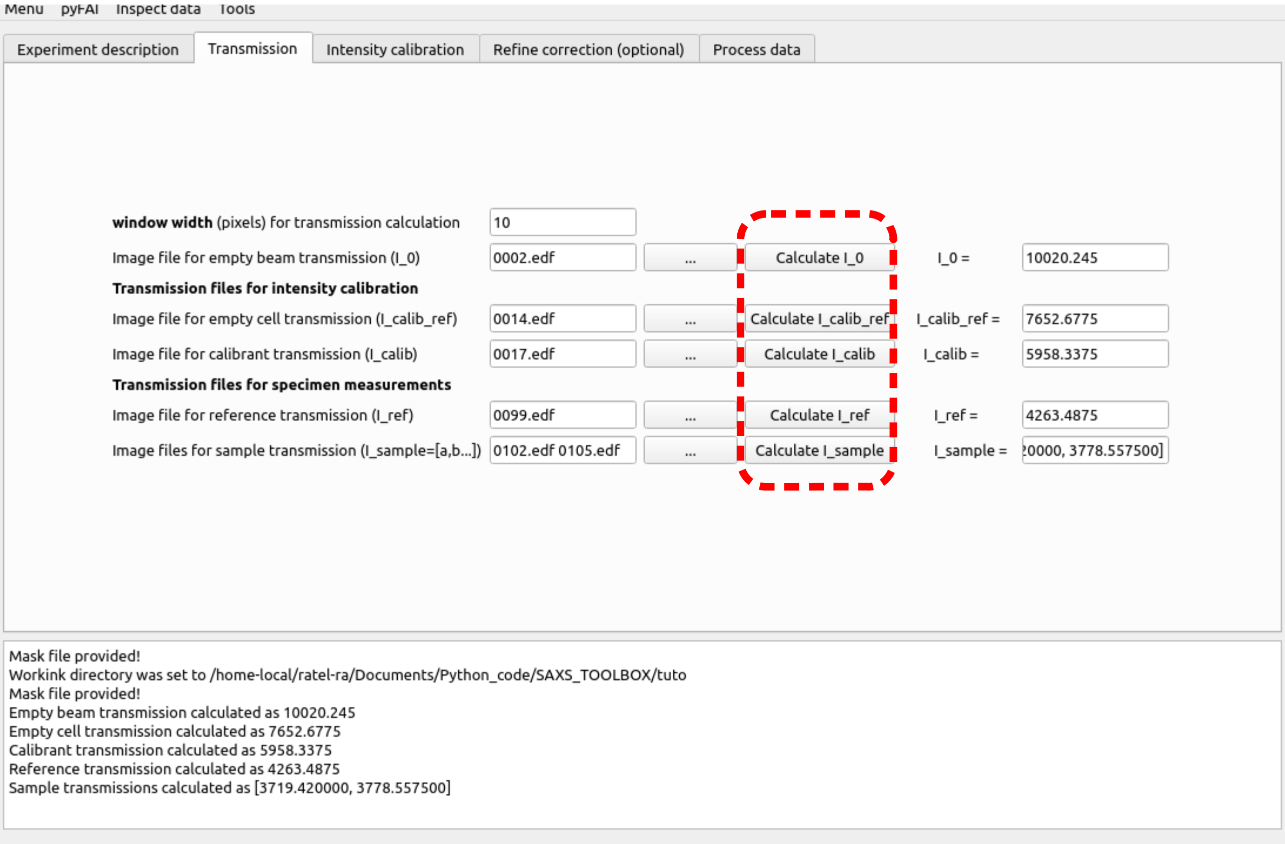
II.2.1 Transmission tab

In the « Transmission » tab, fill the form and select appropriate files according to Table 2

	I_0	I_calib_ref	I_calib	I_ref	I_sample
file	2	14	17	99	102-105

Table 2 : transmission files

Click on the 5 « Calculate » buttons to compute the transmissions. The fields on the right should be updated, as can be seen in Figure 4



window width (pixels) for transmission calculation: 10

Image file for empty beam transmission (I_0): 0002.edf — I_0 = 10020.245

Transmission files for intensity calibration

Image file for empty cell transmission (I_calib_ref): 0014.edf — I_calib_ref = 7652.6775

Image file for calibrant transmission (I_calib): 0017.edf — I_calib = 5958.3375

Transmission files for specimen measurements

Image file for reference transmission (I_ref): 0099.edf — I_ref = 4263.4875

Image files for sample transmission (I_sample=[a,b...]): 0102.edf 0105.edf — I_sample = ?0000, 3778.557500]

```
Mask file provided!
Workink directory was set to /home-local/ratel-ra/Documents/Python_code/SAXS_TOOLBOX/tuto
Mask file provided!
Empty beam transmission calculated as 10020.245
Empty cell transmission calculated as 7652.6775
Calibrant transmission calculated as 5958.3375
Reference transmission calculated as 4263.4875
Sample transmissions calculated as [3719.420000, 3778.557500]
```

Figure 7 : View of the Transmission tab at the end of the procedure

Important note:

Nowadays some detectors include a photodiode in the beamstop, which allows simultaneous transmission and scattering measurements. In these conditions, sample transmissions should be set to 1, by clicking on the “Set sample transmissions to 1” button in the ‘Process data’ tab.

Same procedure applies when considering diffraction data, where transmission corrections are usually ignored.

II.2.2 Intensity calibration

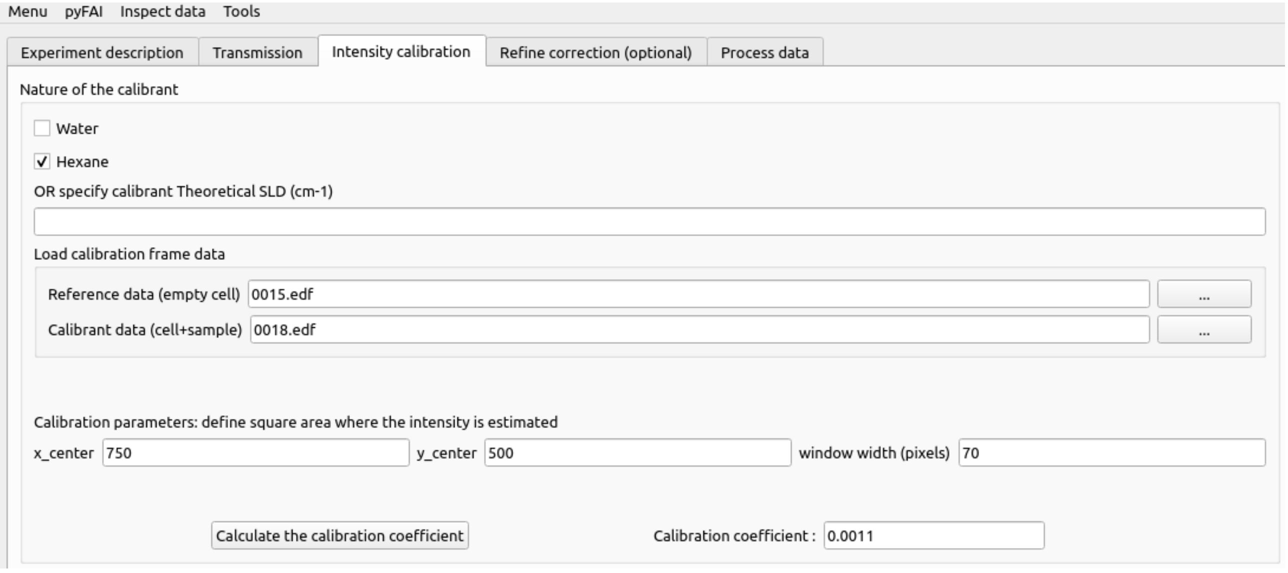
In the intensity calibration tab (see Figure 5), select the hexane as a calibrant by checking the corresponding box.

Load data files corresponding to empty cell (file 0015.edf) and calibrant measurement (file 0018.edf) in the corresponding fields.

You can optionally modify the parameters used for the computation. Those parameters consist in defining a square area in the frame where the intensity is averaged. (see Documentation for more details).

Click on the « Calculate calibration coefficient » button. During the computation, detailed information, such as mean intensity and standard deviations for each frame, are given in the console window (see Figure 6). A small standard deviation (compare to mean value) indicate a good measurement statistics. In the provided example, the standard deviation is rather high due to insufficient exposure of the calibration measurement.

After computation, the calibration coefficient field is updated with the calculated value. Note that if you have already performed the intensity calibration for this experimental setup, the calculated value can be directly typed in this field. In this cas, there is non need to repeat the intensity calibration process, neither to provide transmissions files required by intensity calibration.



Nature of the calibrant

□ Water

☑ Hexane

OR specify calibrant Theoretical SLD (cm-1)

Load calibration frame data

Reference data (empty cell) 0015.edf

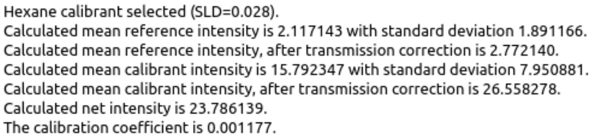
Calibrant data (cell+sample) 0018.edf

Calibration parameters: define square area where the intensity is estimated

x_center 750 y_center 500 window width (pixels) 70

Calibration coefficient : 0.0011

Figure 8: View of the intensity calibration tab



```
Hexane calibrant selected (SLD=0.028).
Calculated mean reference intensity is 2.117143 with standard deviation 1.891166.
Calculated mean reference intensity, after transmission correction is 2.772140.
Calculated mean calibrant intensity is 15.792347 with standard deviation 7.950881.
Calculated mean calibrant intensity, after transmission correction is 26.558278.
Calculated net intensity is 23.786139.
The calibration coefficient is 0.001177.
```

Figure 9: Details of the calculation printed out in the console window

At this stage, data are ready to be processed. However, experience shows that, due to dispersion in capillary sizes (about their nominal size), additional correction may be required. This additional correction can be performed automatically as described below.

II.3 Refine reference correction (optional)

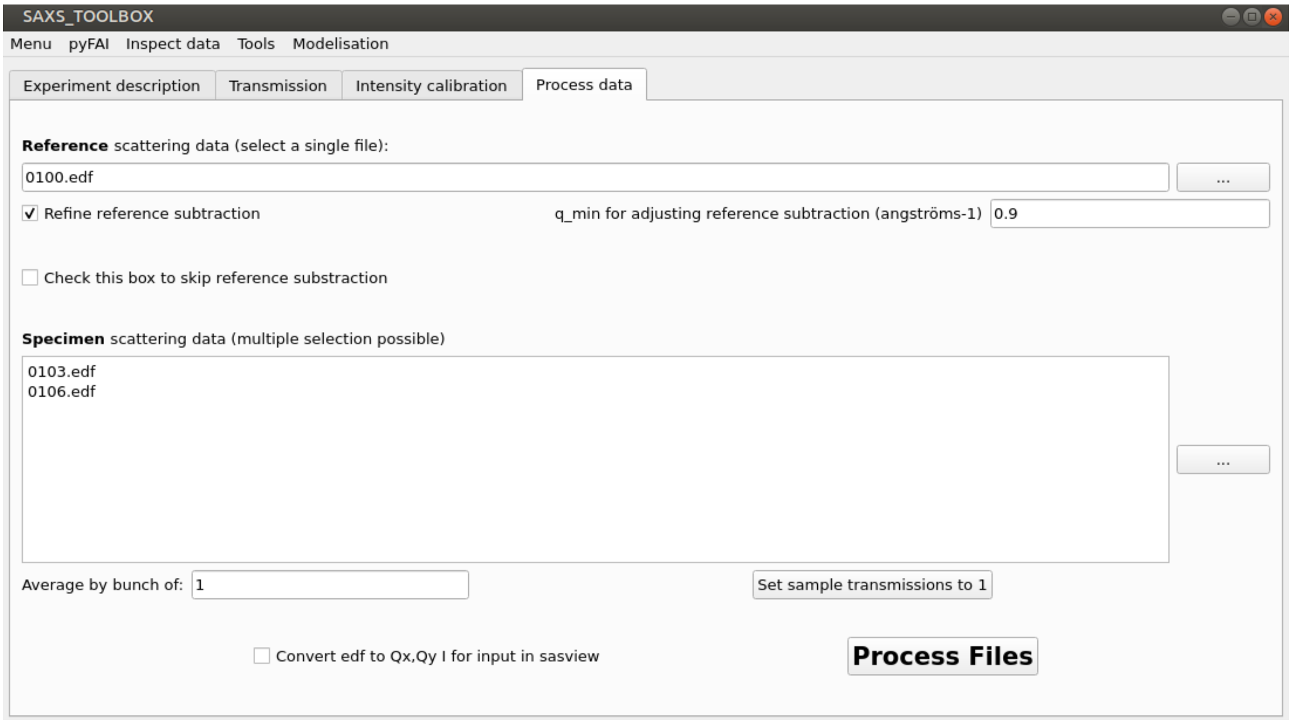
As mentionned in the documentation, the reference substraction can be optimized by checking the box « Refine reference subtraction » in the ‘Process data’ tab. Please counsult the documentaion for more details (section III.2)

II.4 Process data tab

In this tab, the user can specify which files to use as a reference file (file 0100.edf- single file selection is possible), and which file(s) to use as specimen scattering data (files 0103.edf and 0106.edf). Note that an option is available to skip reference substraction. In that case, all transmission values should be set to 1 (click the button « Set sample transmissions to1 »).

If the user wants to generate *.dat files in the Qx,Qy,I format (for input in sasview for instance), the checkbox « Convert edf to Qx,Qy,I for input in sasview » should be checked. This computation is time consuming, and should be performed only when necessary.

To process the data, simply click on « Process data » button. Corrected frames are saved in the destination folder : working_directory/corrected_edf_files, and 2D data files are stored in working_directory/2D_dat_files.



Reference scattering data (select a single file): 0100.edf

☑ Refine reference subtraction

q_min for adjusting reference subtraction (angströms-1) 0.9

□ Check this box to skip reference substraction

Specimen scattering data (multiple selection possible)

0103.edf
0106.edf

Average by bunch of: 1

□ Convert edf to Qx,Qy I for input in sasview

Figure 10: View of the process data tab

II.5 Integrate corrected frames

In the case of isotropic data, you may want to integrate your data to perform the analysis on 1D data. To do so, you can use pyFAI-integrate tool, accessible in pyFAI/Frame integration.

Here you must provide the following items :

- detector calibration *.poni file

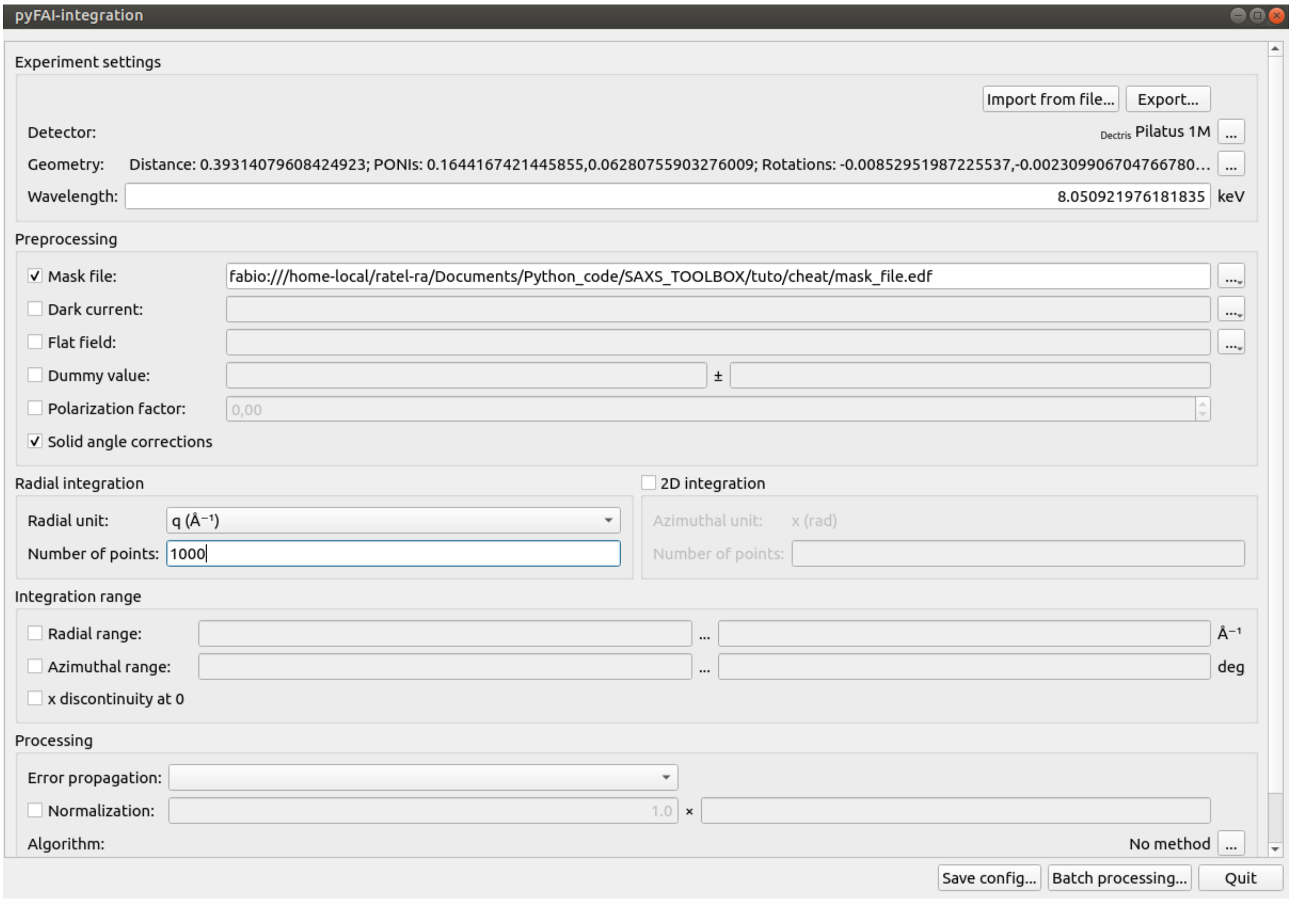
- a mask file (*.edf)

- specify which unit is used for integration (for SAXS select Q (Å^{-1}))

- specify the number of points your 1D data file should contain (here 1000)

When done, clik on the « Batch processing » button to select files to integrate. 1D data files, in *.dat formats are automatically saved in the source directory of the selected images.

Sector integration is also possible by defining the azimuthal range of integration.



Experiment settings

Detector: Dectris Pilatus 1M

Geometry: Distance: 0.39314079608424923; PONIs: 0.1644167421445855,0.06280755903276009; Rotations: -0.00852951987225537,-0.002309906704766780...

Wavelength: 8.050921976181835 keV

Preprocessing

☑ Mask file: fabio:///home-local/ratel-ra/Documents/Python_code/SAXS_TOOLBOX/tuto/cheat/mask_file.edf

☐ Dark current:

☐ Flat field:

☐ Dummy value: ±

☐ Polarization factor: 0,00

☑ Solid angle corrections

Radial integration

Radial unit: q (Å⁻¹)

Number of points: 1000

☐ 2D integration

Azimuthal unit: χ (rad)

Number of points:

Integration range

☐ Radial range: ... Å⁻¹

☐ Azimuthal range: ... deg

☐ χ discontinuity at 0

Processing

Error propagation:

☐ Normalization: 1.0 ×

Algorithm: No method

Figure 14 : view of pyFAI-integrate interface

Integrated data files can be visualized using **Inspect Data/Plot 1D data file(s)/loglog** (or linear).