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,…)

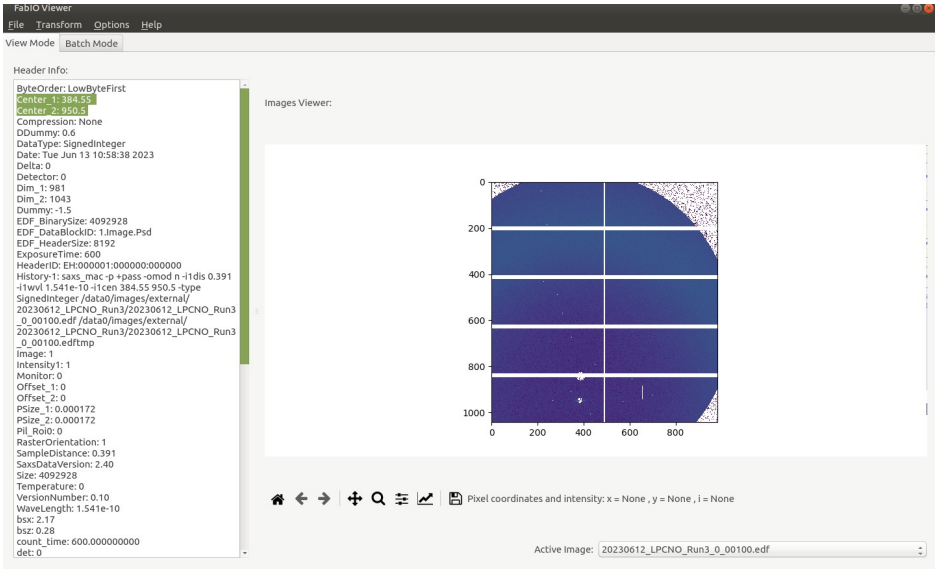

Figure 1 : Fabio_viewer interface and visualization of meta-data

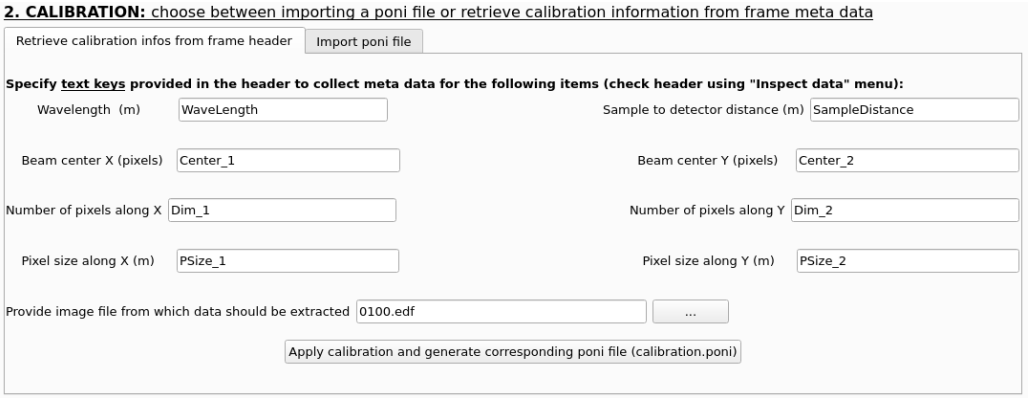

Figure 2: Calibration of images with meta data

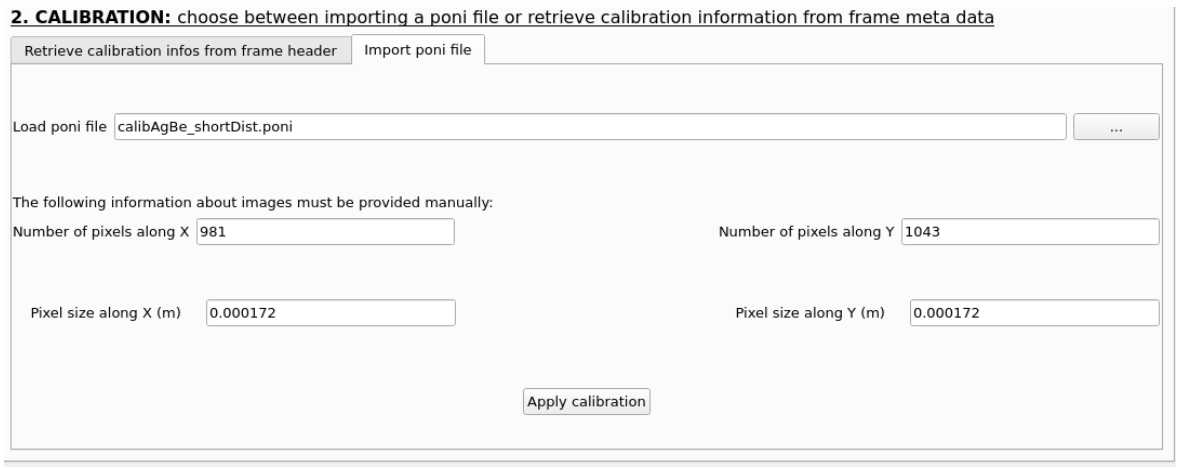

Figure 3: Calibration of data with a poni file

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 »

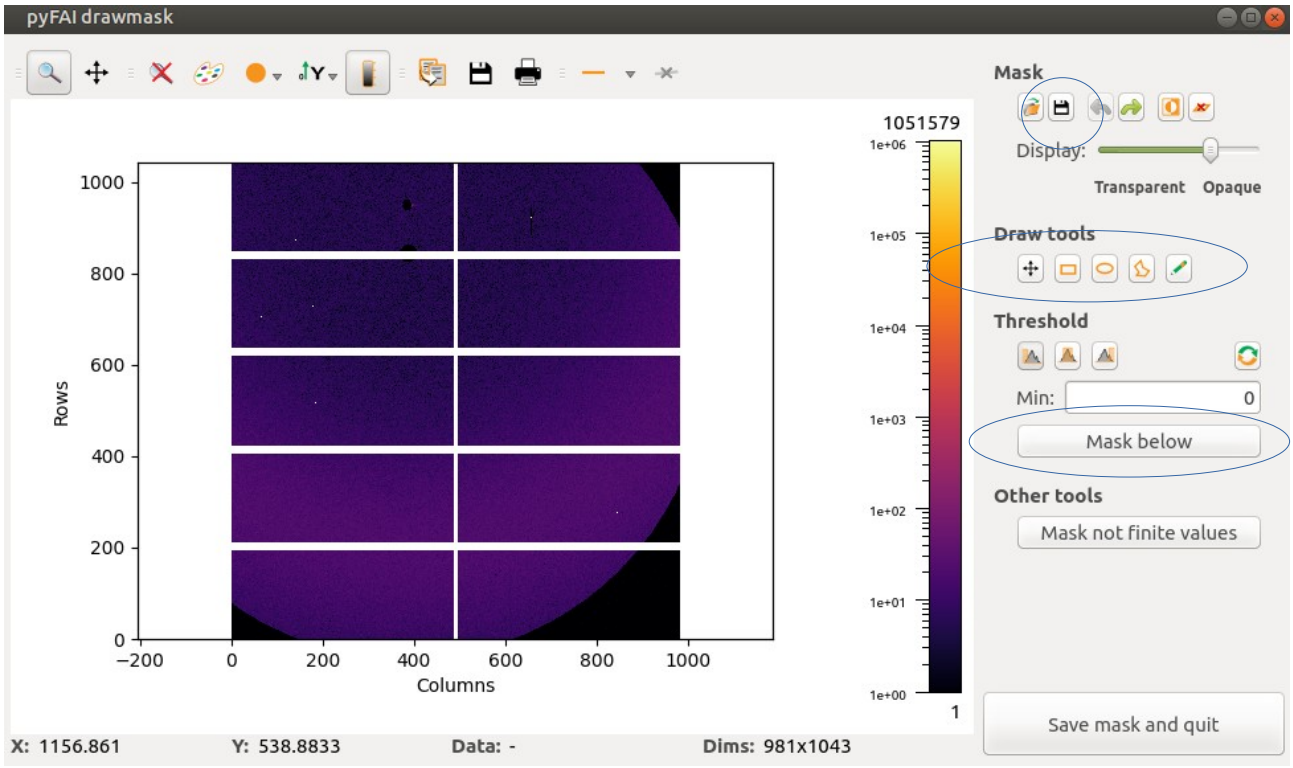

Figure 4 : pyFAI drawmask interface

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

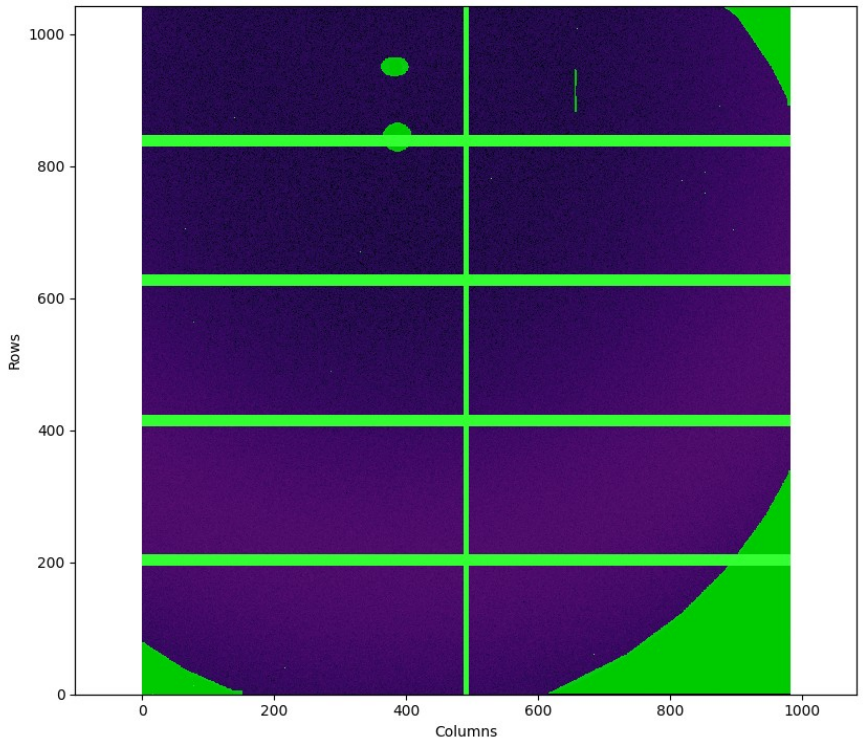

Figure 5 : View of the mask generated with pyFAI

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

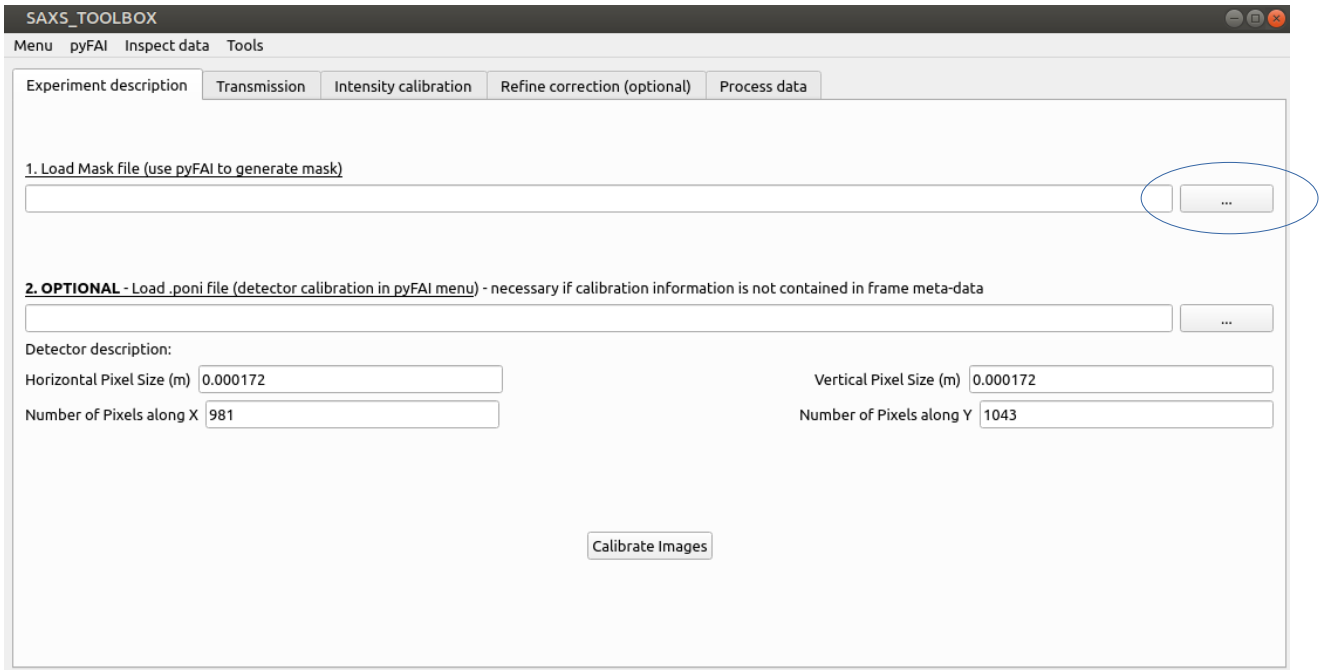

Figure 6 : Experiment description tab: load mask file + optional .poni file

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

	Io	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

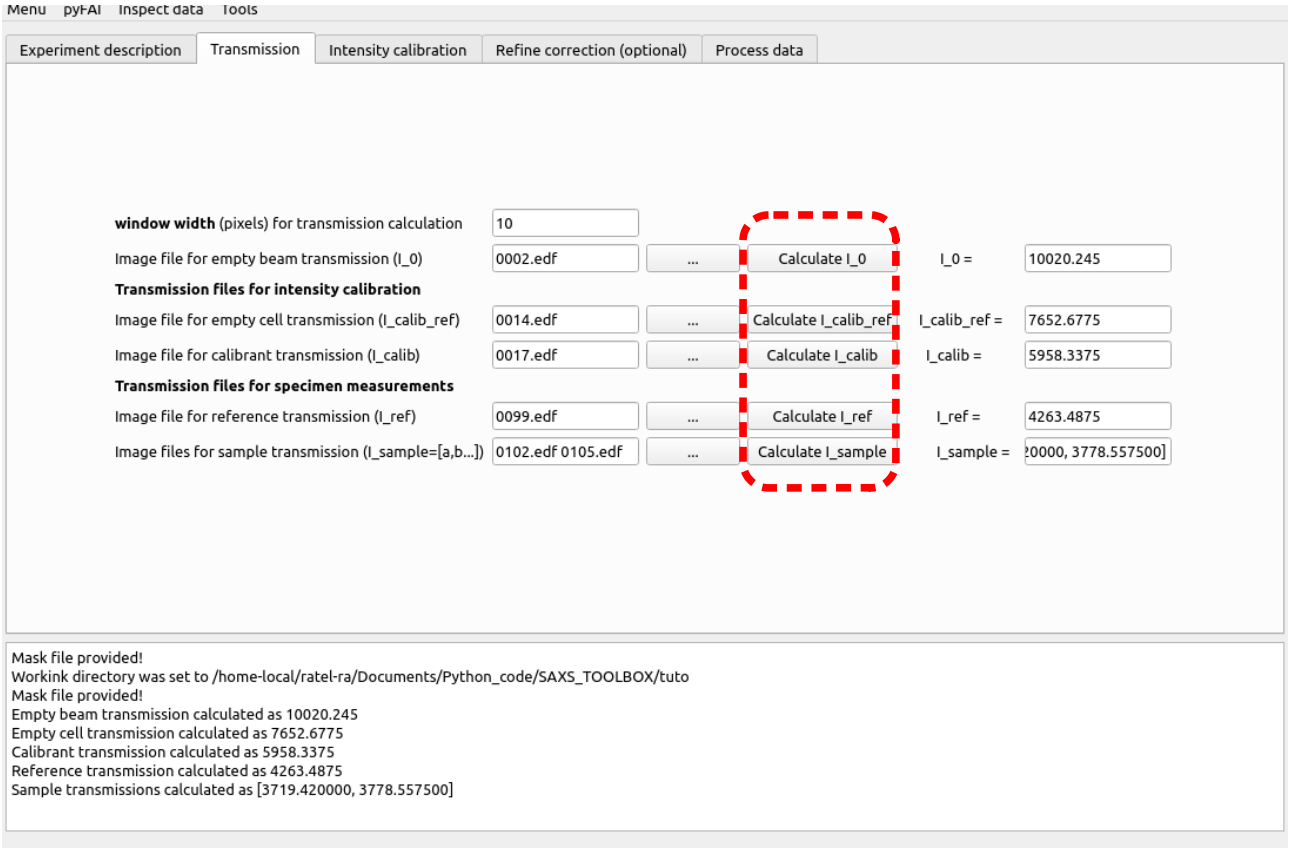

Figure 7 : View of the Transmission tab at the end of the procedure

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.

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 using the “Refine correction tab”.

II.3 Refine correction (optional)

AS mentionned in the documentation, this tool requires the use of 1D data files (so far we have used 2D data frames) corresponding to reference (file 0100.edf) and sample measurement (files 0103.edf and 0106.edf). For the refinement, only one data file is requested for speciment scattering (in our case we will use 0103.edf file).

The first thing to do is tu integrate the 0100.edf and 0103.edf frames using **pyFAI/Frame Integration** in the menu (see Figure 7). To perform the integration, the following items must be provided :

- a detector calibration (*.ponoi file), can be obtanied with **pyFAI/detector calibration**

- a mask file (obtained with **pyFAI/Generate Mask**

- specify integration axis (here q/$Å^{-1}$)

- specify the number of data points in the output 1D file

When all the fileds are completed, click on the « Batch processing » button to select the frames to integrate.

Note that there are many other options availble such as azimuthal (phi) integration within a user-defined q (2θ) range

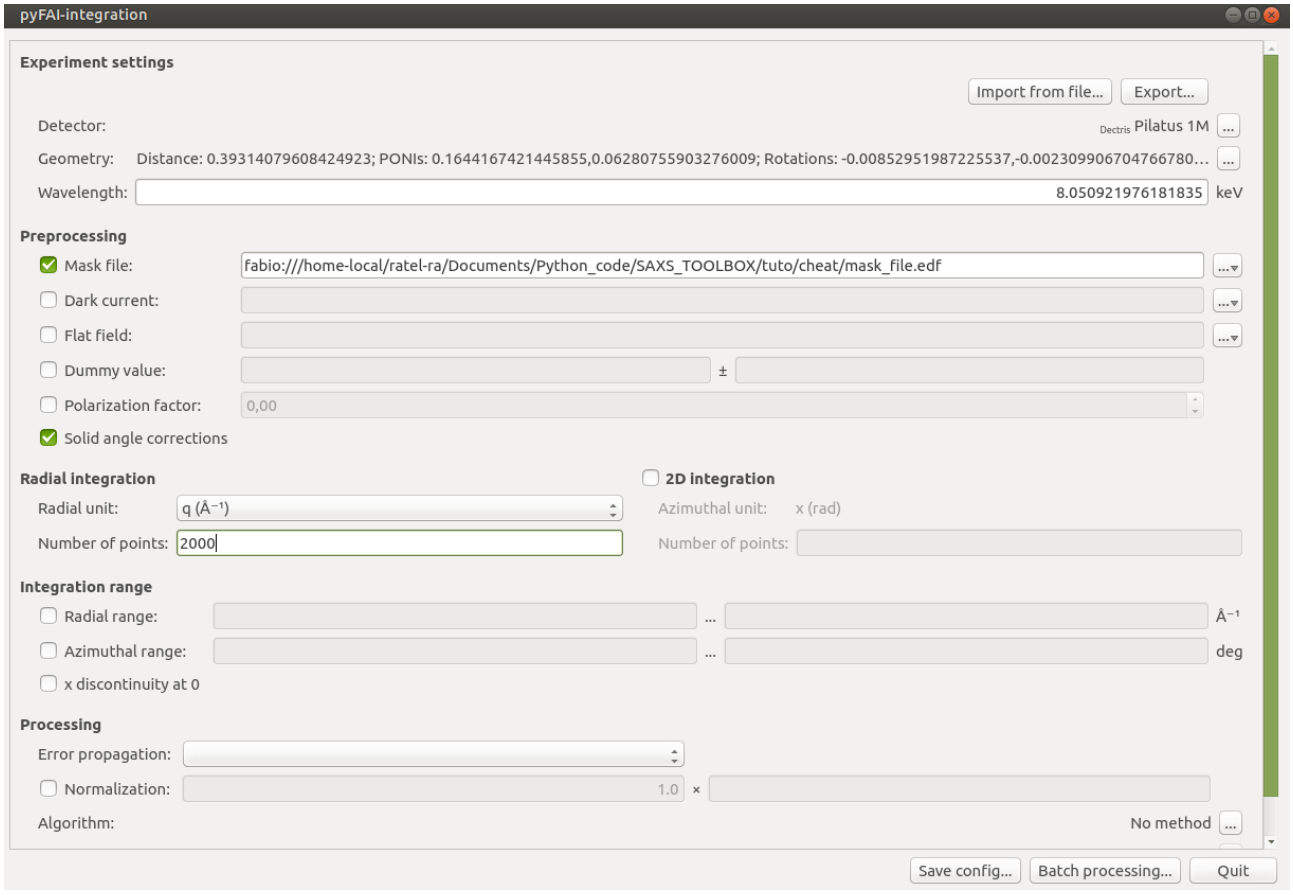

Figure 10 : View of the pyFAI-integrate window

The 1D data files are stored in the same directory as original frames.

Once the 1D data files are generated, simply load them in the « Refine correction » tab of SAXS-toolbox using the corresponding buttons « Browse for reference file (1D) » and « Browse for specimen data », as shown in Figure 8.

The field below is given to provide the transmission value corrresponding to specimen data. In our case, that transmission value corresponds to that obtained for file 0102.edf, i.e. the first value in the sample transmission array (since the 0102 file was the first one in the sample list). This value (3719,42) must be entered manually by the user in the « specimen transmission » field.

Clicking on the « Estimate coefficient » button launches the computation of the coefficient.

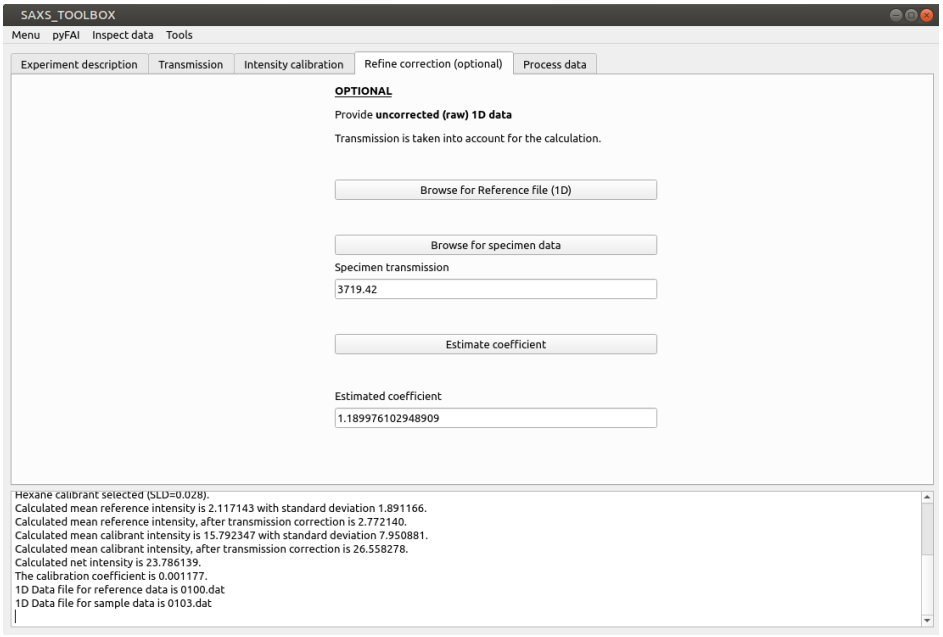

Figure 11 : View of the Refine correction tab

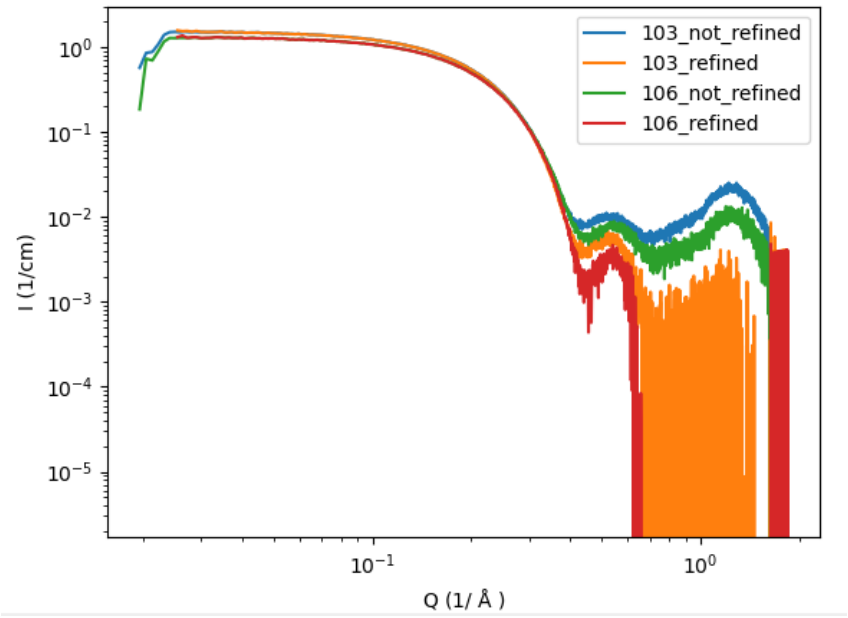

Figure 12 : Influence of refinement correction on processed data

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.

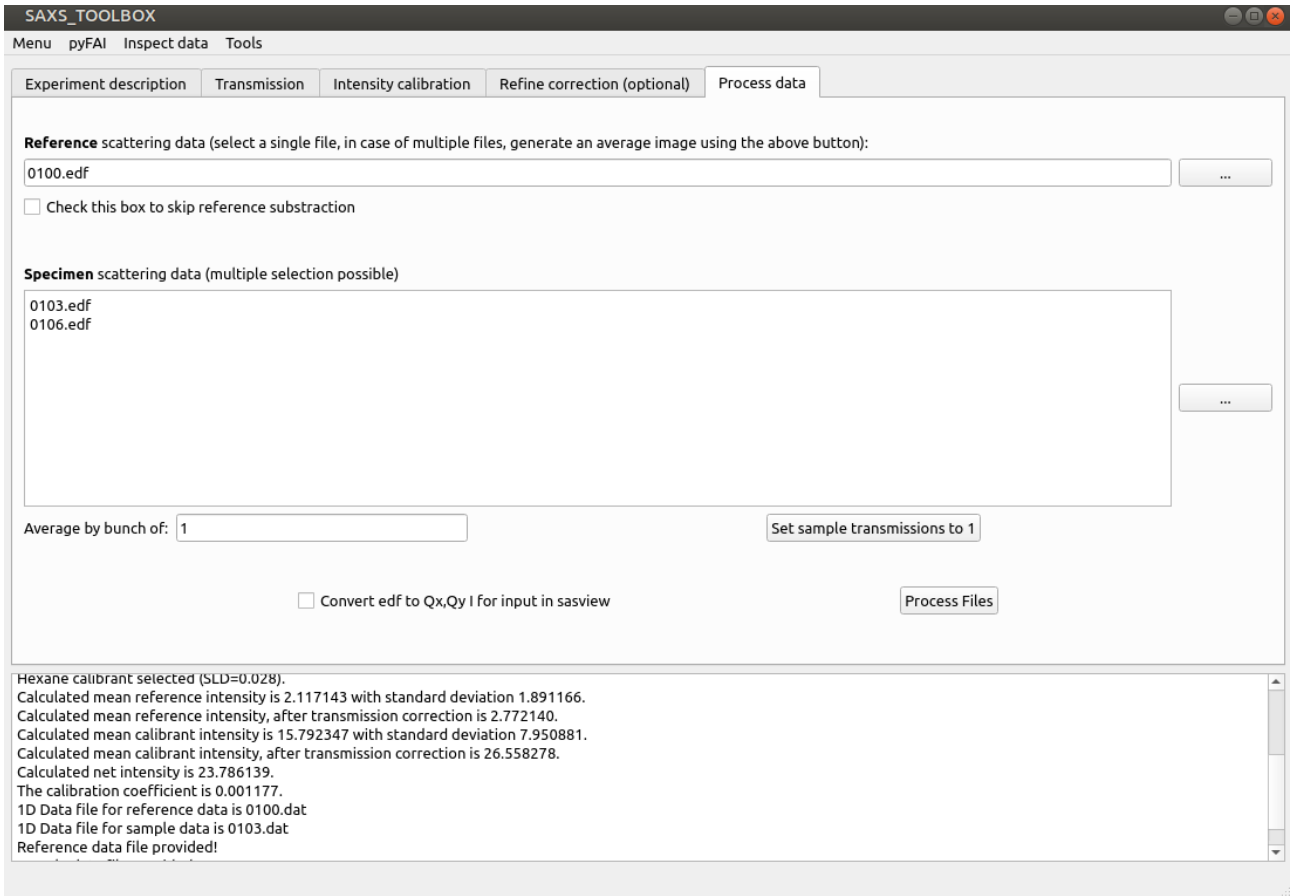

Figure 13: View of the process data tab

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.

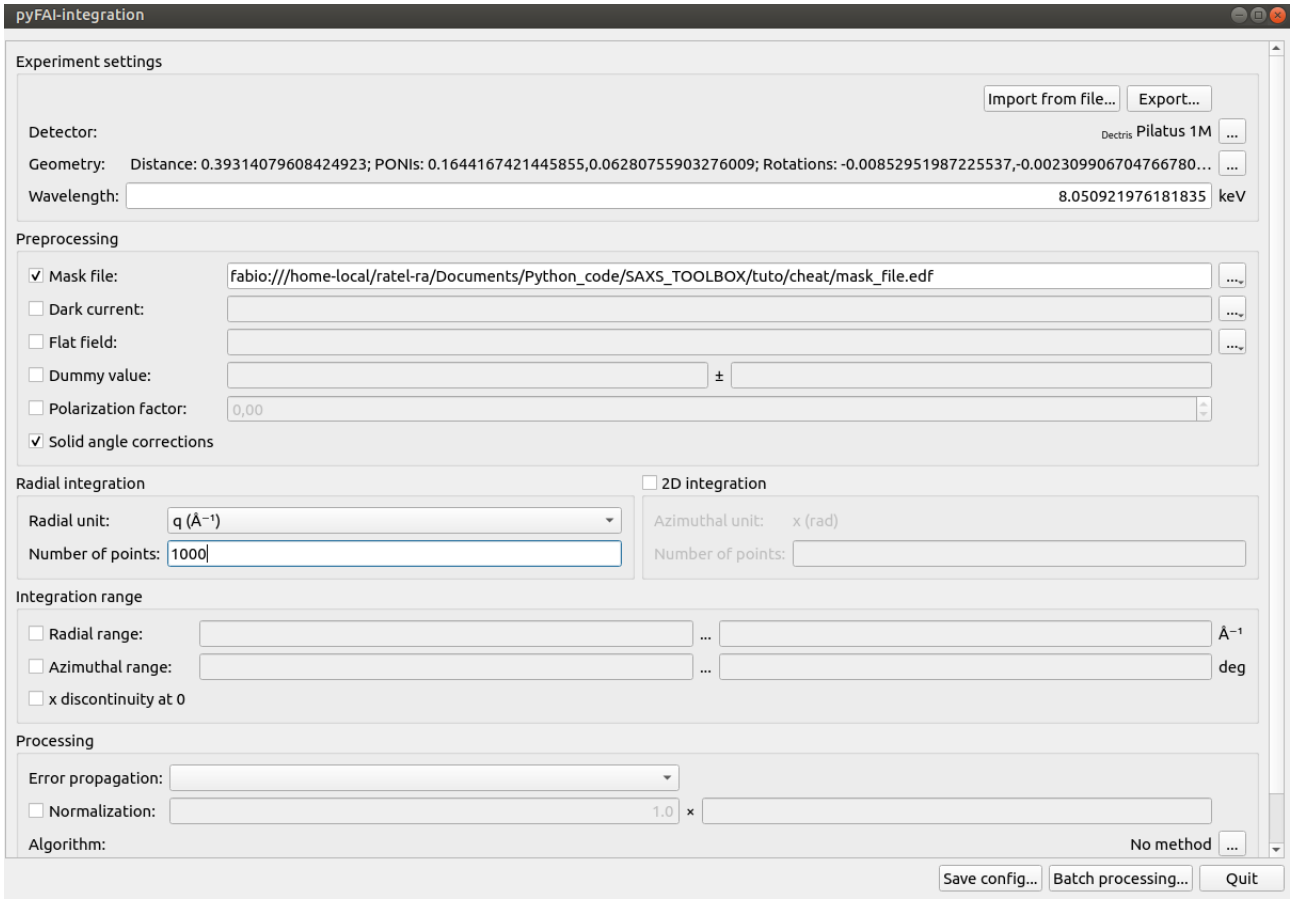

Figure 14 : view of pyFAI-integrate interface

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