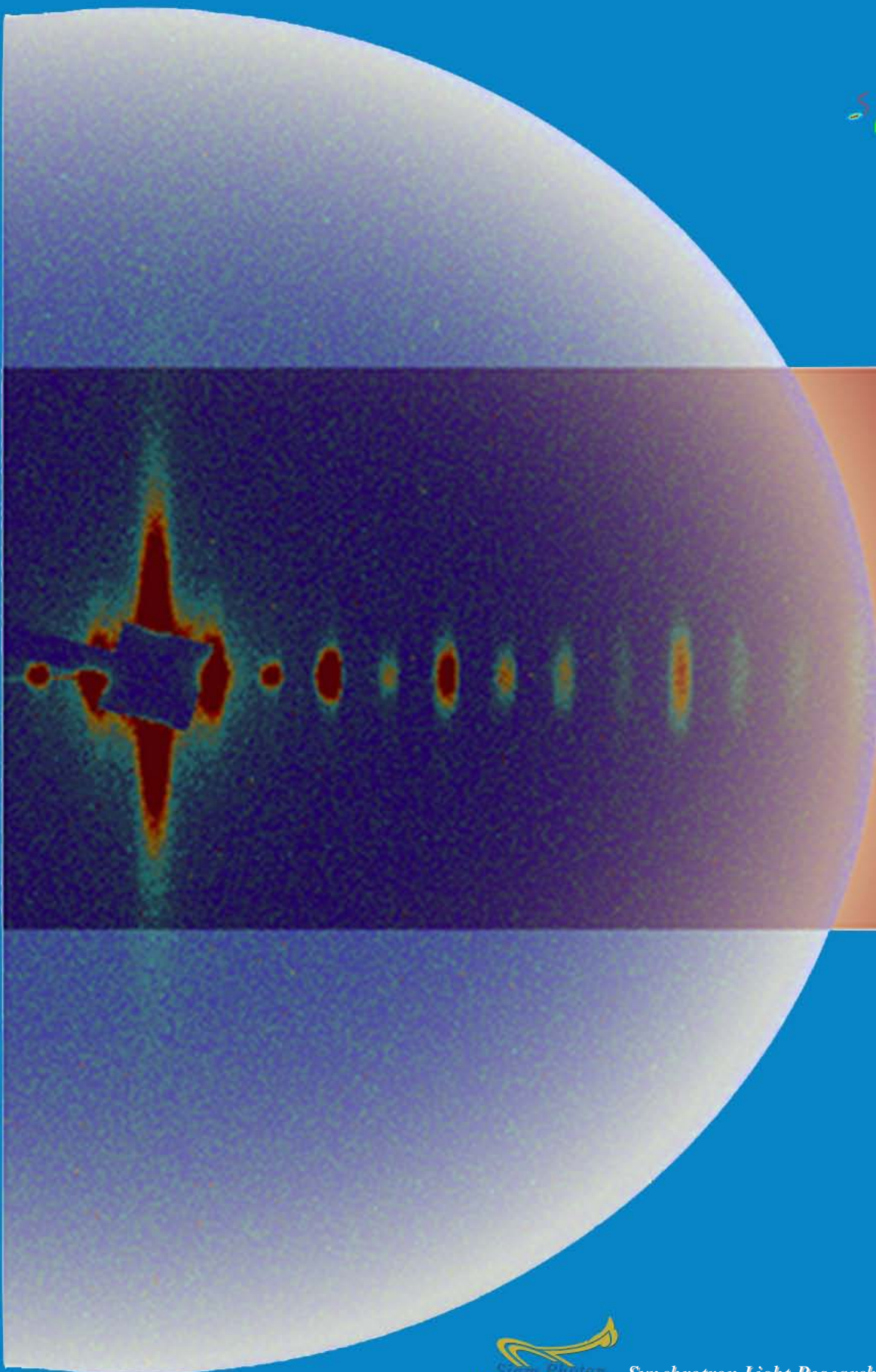
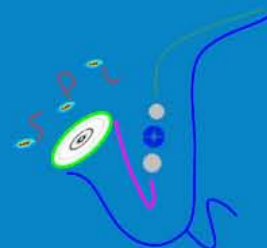


# SAXSIT MANUAL

Manual for SAXS/WAXS data processing using SAXSIT



Synchrotron Light Research Institute (Public Organization)

คู่มือโปรแกรมสัคดีสิทธิ์

SAXSIT Manual

Version 3.66 : December 2013

SAXS/WAXS Station

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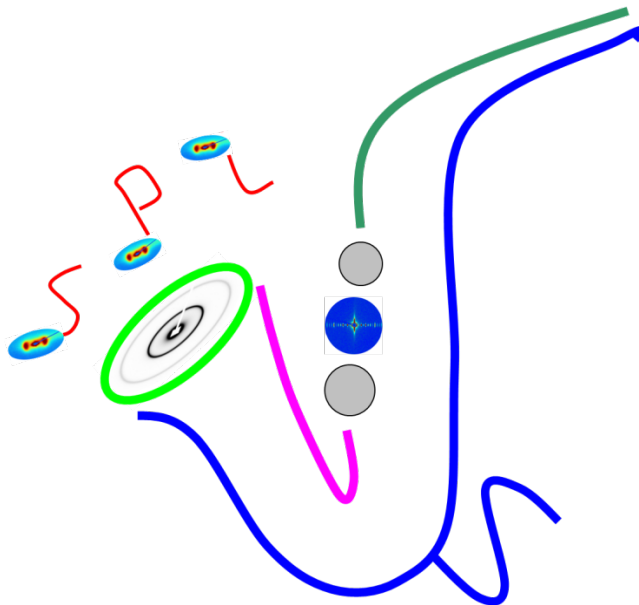
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<http://www.slri.or.th/th/beamlines/SAXS>

# *S*mall *A*ngle *X*-ray *S*cattering *I*mage *T*ool



A program for SAXS/WAXS data processing



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# SAXSIT

Version 3.66, December 2013

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## Introduction

SAXSIT (Small Angle X-ray Scattering Image Tool) is a SAXS data processing program written with Matlab. The program is compiled into executable modules which can be run without Matlab. However, although it is not necessary to have Matlab installed, the Matlab Component Runtime Library (the file MCRIInstaller.exe) must be run once in the computer. The MCRIInstaller.exe is freely available and can be downloaded from Mathwork website. It is also provided in the SETUP file. Please note that the file is a little large (>300 MB).

SAXSIT is mainly intended to be used for processing Tiff image file, including the output .mccd file from Mar CCD. Most of other image file formats (i.e. those supported by Malab) are also expected to be valid. Some unsupported image file format, such as that from Mar345 Image Plate will have to be converted to 16 bit Tiff file before processing with SAXSIT.

Please note that SAXSIT is, and is expected to always be, ‘under construction’ and will most certainly contain some bugs. Any problems or comments please kindly send a message to [supagorn@slri.or.th](mailto:supagorn@slri.or.th) or [siriwat@slri.or.th](mailto:siriwat@slri.or.th).

## Modules

SAXSIT3.65 is structured into modules as the following :

Menu	Module	Function
Main menu	saxsit3p66	The module to start the program. It can be opened by running (double-clicking) the program saxsit3p66.exe.
Read measurement parameters	mmreadmeaspara	Read in measurement parameters. The most convenient way is to read the parameters from an excel file. The parameters are read in specific order. Users should use the given file MeasPara.xls as a template. Alternatively, the parameters may be entered via the interface window.
Analyze standard	mmstd	Find center of scattering pattern, i.e. the position of the primary beam, from pattern of a standard (such as

		silver behenate). The module calculates the pixel shift values relative to a chosen coordinate on the pattern which will be used to align scattering patterns of the samples. This module also calculates sample-detector distance from the known peaks of the standard.
Load pattern / BG subtraction	mmsubbg	Loading patterns and perform background subtraction.
Calculate scattering profile	mmscatprofile	Calculate scattering profile from a scattering pattern and do circular averaging of the profile.
Radial integration / orientation factor	mmradint_hermans	Look at the pattern and examine its appearance. Range of angles to be circularly averaged can also be examined with this module. The module can also adjust intensity and save the pattern as a JPEG file. This module also allows you to do radial integration of the pattern to obtain integrated intensity as a function of azimuthal angle. This is useful in case of microfibril angle measurements and to find the azimuthal angle of the peak intensity. The module also performs calculation of Hermans orientation factor.
Line profile analyses	Mmlineprofile4	Perform various profile analyses, such as baseline removal, curve smoothing and peak search. It can also perform peak function fitting of the profile for Gaussian, Lorentzian, Pseudo Voigt and Thompson-Cox-Hastings functions. However, the peak function fitting is intended to be preliminary and not very effective. Other more effective peak fitting modules are available in the Fitting menu.
Merge data sets	mmmergedata	Merge two data sets, such as SAXS profiles of the same sample from different sample-detector distances.
Normalize data by area under curve	mmnormarea	Normalize data by scaling the data sets to the same value of integrated intensity within a chosen range.
Mask pattern	mmmask	Perform median masking of a pattern to get rid of cosmic ray spikes.
Fitting	mmfit	Contains menu for available fitting

		modules (listed in the next table).
Old modules	mmoldmodules	This menu contains modules which are not expected to be used for data processing. Some modules in there might still be useful, such as intensity adjusting module, image cropping module, image converting module image adding/averaging module.
Estimate q-range	mmqrange	Calculate q-range which can be measured given a specific setup.
Integrate data	integrate_arb	This is just an auxiliary module which allows you to integrate the data.

## Available fitting modules

Guinier and Porod fit	mmguinier_multidat	A product $q^m I$ , where $m=0,1,2,3,4$ versus $q^n I$ , where $n=1,2,3,4$ can be plotted on linear, semilog or loglog scale. Various fittings such as radii of gyration or straight line can be done for multiple data sets.
Nanosphere	mmnanosphere	Fitting nano particle profile for size and size distribution
Paracrystalline1	mmparacrystalline4	Fitting profile to paracrystalline model (lamellar formfactor*paracrystalline structure factor) for polymer and fiber samples. The fitting gives period size and size distribution (Gaussian) and crystalline size.
Paracrystalline2	mmparacrystalline2	Similar to Paracrystalline1, with alternative background terms.
Exponential-power law	mmexpopower	Fitting unified Guinier-power law model of Beaucage for nano particle (single size level only).
Rod & Debye-Bueche	mmroddebye	Fitting crosssectional Guinier term + Debye-Bueche aggregation model

Broken rod & Debye-Bueche	mmbrokenrod	Fitting Rod form factor + Debye-Bueche aggregation model.
Sphere micelles	mmspheremicelles	Fitting sphere+micelles model
Core-shell	mmcoreshell	Fitting sphere+shell model
Peak Pseudo Voigt	mmpeakpv2	Fitting Pseudo Voigt peak functions and calculate areas under curves for crystallinity calculation.
Peak Pearson VII	mmpeakpsvii	Fitting Pearson VII peak functions and calculate areas under curves for crystallinity calculation.

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# Setting up SAXSIT

1. Extract the compressed files SAXSIT3p66\_64bit.zip and SAXSIT\_SETUP64bit.zip for Windows 64 bit, or SAXSIT3p66\_32bit.zip and SAXSIT\_SETUP32bit.zip for Window 32 bit.
2. Run MCRInstaller.exe once (by double clicking it).
3. In the folder SAXSIT3p66 find saxsit3p66.exe, make a shortcut to the desktop. You can now run the program by double-clicking the shortcut.

# Start using SAXSIT

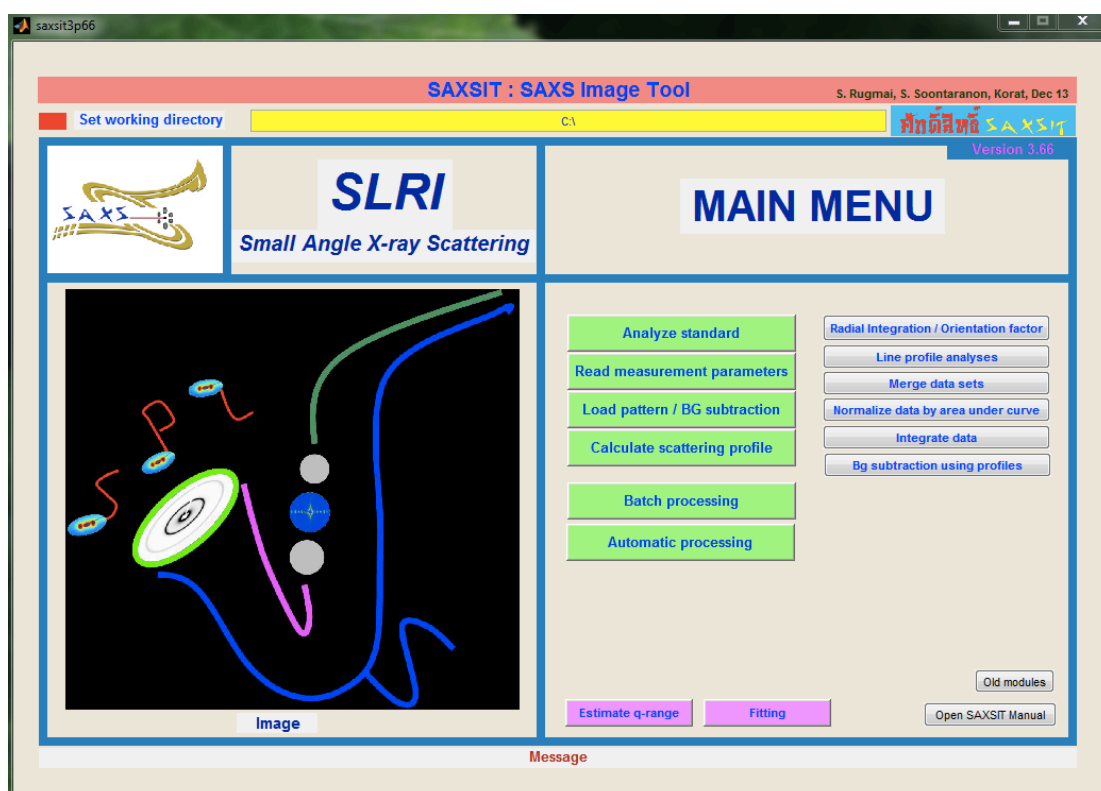
First, it is recommended that you create a data folder and put scattering patterns in there then copy the file MeasPara.xls from the folder SAXSIT3p66 into your data folder. Next, do the following,

1. Open the excel file MeasPara.xls and put in all the parameters in the second column, then save the file. The filename can be changed. Note that not all inputs are necessary, depending on what you want to do. As a matter of fact, in many cases, e.g. when any normalization and background subtraction are not to be carried out, the whole file is not even necessary. However, it is recommended that you use this file and put in the measurement parameters as much as you can. This will nevertheless give consistency during the data processing.

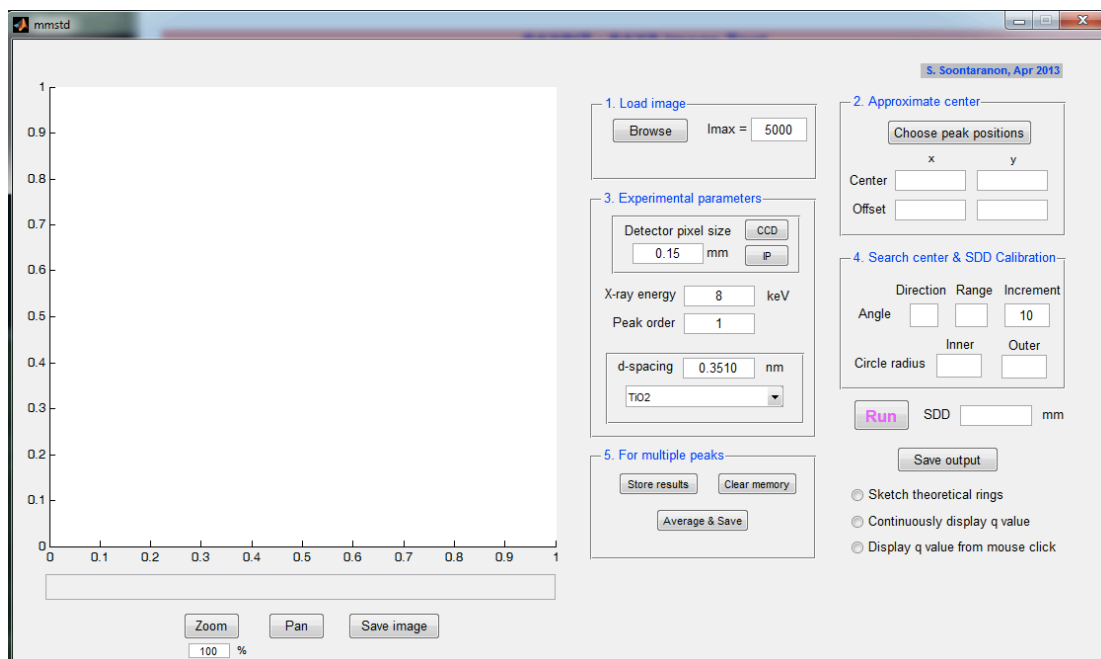
Note that some values in this file will be obtained later after analyzing the standards.

2. Run saxsit3p66.exe
3. Copy the directory path of your data folder into the 'Set working directory' box. All inputs and outputs will be read from and written into this folder.
4. Choose what to do from the MAIN MENU.

Note that this Manual can also be opened from the SAXSIT window by clicking the button 'Open SAXSIT Manual' in the bottom right corner.



## Analyze standard



The module is used to calculate vertical and horizontal pixel shifts to be used for pattern alignment, i.e. to shift the pattern so that its actual center is at the primary beam position. The module uses a pattern of some standard material which gives circular scattering pattern, e.g. silver behenate. The scattering profiles are calculated from the pattern at three points and fitted with Gaussian to find peak positions. The program will then estimate the radius and scan along the circular path. The peak positions are then used to find the center of the circle.

The center of the circular pattern is assumed to be the primary beam position. After the center is found, the vertical and horizontal pixel distances will be calculated between this center and a specified origin of coordinate. When doing scattering profile calculation the scattering angles will then be calculated from the distances with regard to the obtained origin.

1. Load standard pattern by clicking 'Browse'. The program will ask the user to choose a file from the working directory.
2. Choose three points on the middle of the circular pattern by clicking on the pattern picture. When choosing each point, at the beginning, the pointer will act as a zoom and the user will have 5 seconds to zoom in, after 5 seconds the pointer will then change and the user can click to choose the position. These three points will be used to estimate the radius of the circle.
3. Input the detector pixel size (clicking the detector type will automatically give corresponding pixel size) and d-spacing value of the peak (choosing standard from drop-down menu will automatically give corresponding d-spacing of the first order peak).
4. The range of scan will be calculated from the chosen 3 points and displayed in the box number 4. These values can be changed if desired. The 'Circle radius' values indicate the default range of radius for the intensity to be calculated. These radius range can be changed. The range should give the intensity covering the peak to obtain sensible Gaussian peak fit.

5. Click 'Run' button. The program will then start scanning and fitting for peak positions. The circle center, and the sample-detector distance (SDD) will then be calculated. After finishing, the 'Run' button can be clicked again to refine the results. The difference between the current and previous runs will be displayed in the message box. The run should be repeated many times to get a converged SDD value (i.e. when the difference between runs is small enough, e.g. in the order of  $1e-3$ ).
6. Click 'Save output'. The calculated results from each run will be saved in the file 'alignstandard.txt'.

Example of using the Analyze standard' module can be found in Example 1 at the end of this manual.

## Read measurement parameters

This module reads in measurement parameters from an excel file. The parameters must be input in a specific order in the second column of the excel file. Users are recommended to use a template file MeasPara.xls given with the SAXSIT package to make the input file. Not all parameters are necessary, depending on what to be done. For some cases this module, and thus the input file, may not even be necessary. However if the module is used, at least the sample filename must be given in the input file, and the corresponding pattern must be present in the folder.

1	Experimental condition				
2	Title	KC			
3	X-ray energy (keV)	8			
4					
5	Dark current				
6	Dark current pattern filename				
7	Accumulated time (sec)				
8					
9	Reference				
10	Parameters for reference pattern (text file)	20120922_empty.0233.txt			
11					
12	Sample				
13	Sample pattern filename	20120922_0.5KC_n.0237.mccd			
14	Parameter for sample pattern (text file)	20120922_0.5KC_n.0237.txt			
15	Median mask (Y/N)	Y			
16					
17	Background				
18	Background pattern filename	20120922_water.0235.mccd			
19	Parameter for background pattern (text file)	20120922_water.0235.txt			
20	Median mask (Y/N)	Y			
21					
22	Parameters obtained from standard sample				
23	Standard pattern filename	20120922_AgBH.0198.mccd			
24	Center offset X (pixels)	40.9043			
25	Center offset Y (pixels)	-26.4945			
26	Sample to detector distance (mm)	1517.65			
27					
28	Detector parameters				
29	Pixel size (mm)	0.07959	CCD = 0.07959, IP = 0.15		
30	Detector radius (mm)	82.5	CCD = 82.5, IP = 172.5		
31					
32	Profile calculation parameters				
33	Normalize beam current (Y/N)	Y			
34	Normalize exposure time (Y/N)	N			
35	Subtract dark current pattern (Y/N)	N			
36	Normallize transmission (Y/N)	Y			
37	Sample concentration	0	Put 0 if concentration is unknown		
38	Direction to calculate profile (deg)	90			
39	Averaging range (deg)	10			
40	Step size (deg)	0.25			

### Parameters in MeasPara.xls file

In the MeasPara.xls file, the inputs in the section 'Parameters obtained from standard sample' are calculated when the standard pattern is analyzed with 'Analyze standard' module.

The text files (filenames with .txt above) contain values of integrated intensity readings from ionization chamber and photodiode. These values are used to normalize

the scattering intensities. They may also be entered in the interface window of the 'Read measurement parameters' menu (in the table at the bottom) instead. If no normalization is required, these values are not necessary and should be set to 1.

When clicking 'Save parameters' the program will read in from the currently displayed values. The values in the window may therefore be modified after reading from the file.

Working directory : C:\

SAXSIT ver 3.5

**Read parameters**

Read from excel file Save parameters

Experiment title

X-ray energy (keV)

Dark current pattern file

Accumulated time (sec)

Reference parameters file

Sample pattern file

Sample parameters file

Sample median mask (Y/N)

Background pattern file

Background parameters file

Background median mask (Y/N)

Standard pattern file

Center offset X (pixels)

Center offset Y (pixels)

Sample-Detector distance (mm)

Normalize integrated beam intensity (Y/N)

Normalize exposure time (Y/N)

Subtract dark current (Y/N)

Normalize transmission (Y/N)

Sample concentration

Angle to cal profile (deg)

Range of angle for circular averaging (deg)

Angular step for averaging (deg)

Pixel size (mm)

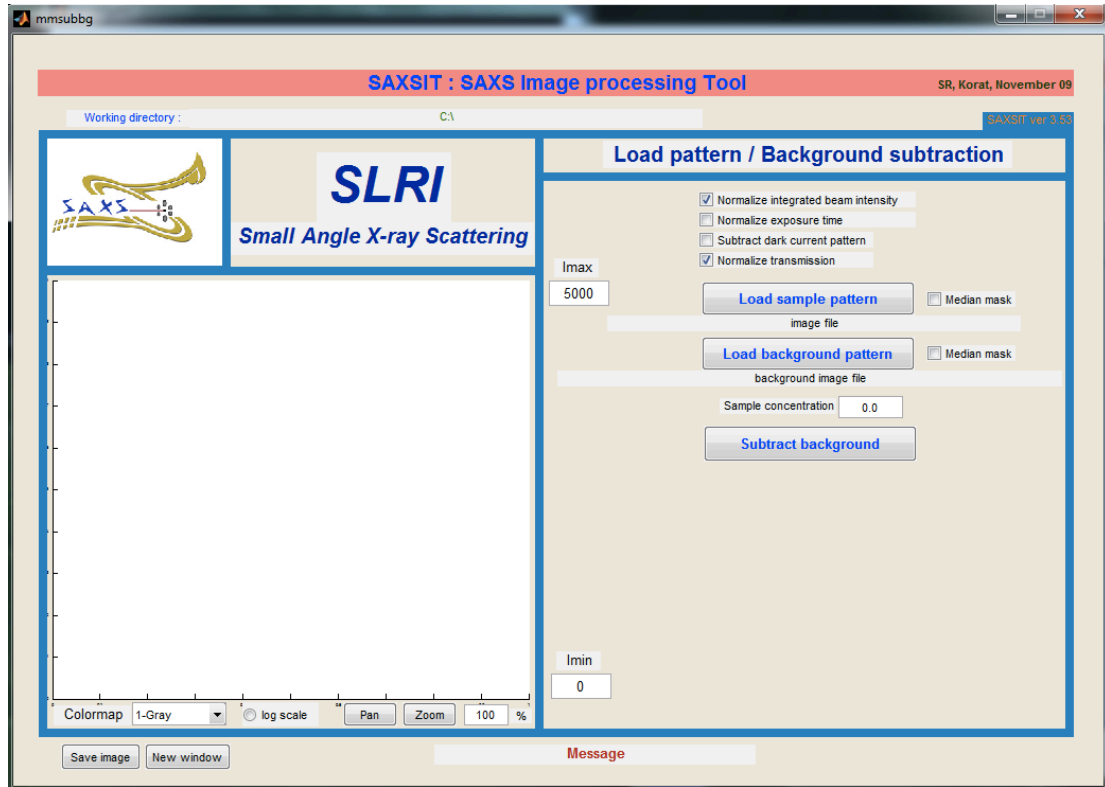
Detector Radius (mm)

	Exposure time (sec)	IC (count)	Integrated IC (count)	PD (count)	Integrated PD (count)	Transmission
Reference	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
Sample	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
Background	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>

Message

1. Click the button 'Read from excel file'. The program will ask you to specify the excel file containing measurement parameters. It will then read in the parameters and display them.
2. Click 'Save parameters'. All parameters will be saved and ready to be used in other modules. The program will also display an image of the pattern specified as the 'Sample filename' in the parameter file. In some cases the shown pattern may look a little odd due to the way intensity is displayed. This should not cause alarm. If in doubt, the pattern may be examined using the module 'Radial integration / Orientation factor'.

## Load pattern / BG subtraction



This module is intended to be used for background subtraction, especially for scattering of particles in solution. However, it can also be used to load sample or background patterns without doing background subtraction. The program will load patterns specified in the parameter file. Four check boxes are options which can be chosen for normalization procedure. The background subtracted pattern is calculated from

$$I_{subbg} = \tilde{I}_{sam} - (1 - c)\tilde{I}_{bg}$$

Where

$$\tilde{I}_{sam} = \frac{(\bar{I}_{sam} - \bar{I}_{dark})}{T_{sam} \cdot i_{sam,IC}},$$

$$\tilde{I}_{bg} = \frac{(\bar{I}_{bg} - \bar{I}_{dark})}{T_{bg} \cdot i_{bg,IC}},$$

$$\bar{I}_{sam} = \frac{I_{sam}}{t_{sam}} \equiv \text{sample pattern divided by sample exposure time } t_{sam},$$

$$\bar{I}_{bg} = \frac{I_{bg}}{t_{bg}} \equiv \text{background pattern divided by exposure time } t_{bg},$$

$$\bar{I}_{dark} = \frac{I_{dark}}{t_{dark}} \equiv \text{dark current pattern divided by accumulated time } t_{dark},$$

$i_{sam,IC}$  and  $i_{bg,IC}$  are x-ray intensity, measured by the ion chamber, integrated over the period of the measurements of sample and background, respectively.

$c \equiv$  sample concentration ( $0 \leq c \leq 1$ ). The default value is 0.0 meaning the background will not be scaled.

$$T_{sam} = \frac{i_{sam,PD} / i_{sam,IC}}{i_{cell,PD} / i_{cell,IC}} \equiv \text{sample transmission, where } i_{sam,PD} \text{ is beam intensity (measured}$$

by a photodiode at the beamstop), integrated over the measurement period of the sample, and  $i_{cell,PD}$  is that for the empty sample cell

$$T_{bg} = \frac{i_{bg,PD} / i_{bg,IC}}{i_{cell,PD} / i_{cell,IC}} \equiv \text{background transmission, where } i_{bg,PD} \text{ is beam intensity}$$

(measured by a photodiode at the beamstop), integrated over the measurement period of the solvent.

If the box ‘Normalize integrated beam intensity’ is unchecked,  $i_{sam,IC}$  and  $i_{bg,IC}$  will be set to one.

If the box ‘Normalize exposure time’ is unchecked,  $t_{sam}$ ,  $t_{bg}$  and  $t_{dark}$  are set to one.

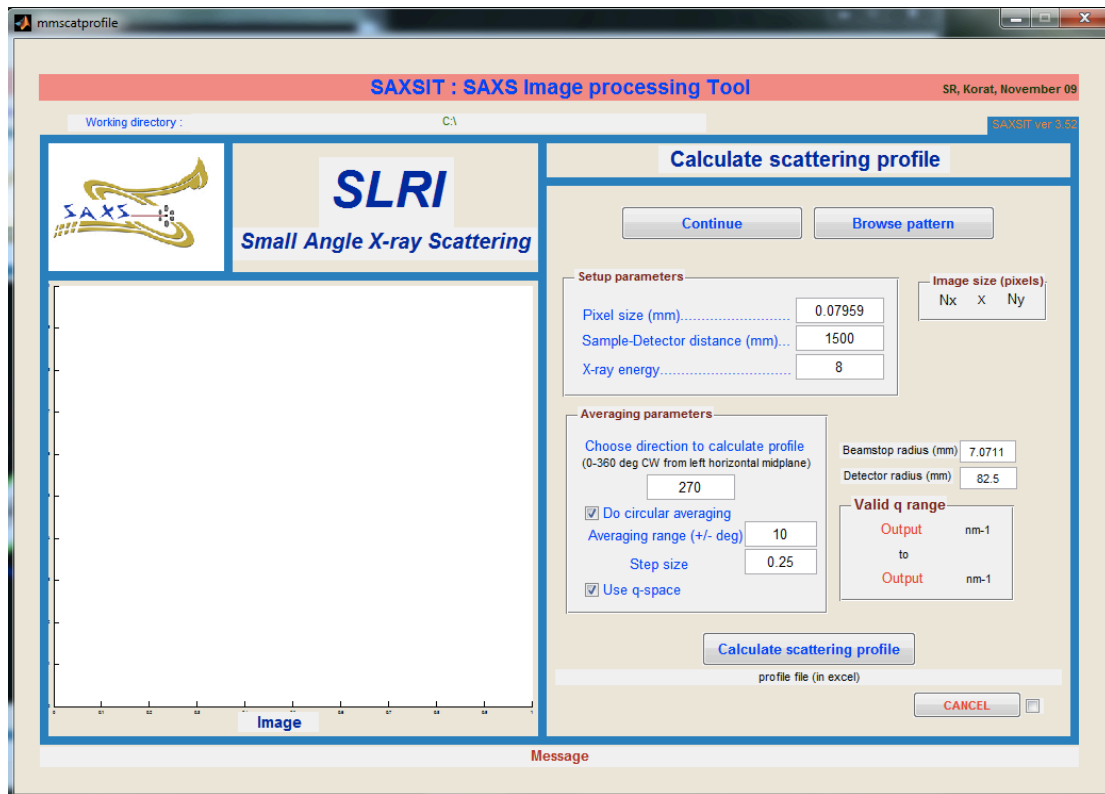
If the box ‘Subtract dark current pattern’ is unchecked  $\bar{I}_{dark}$  will be set to zero.

If the box ‘Normalize transmission’ is unchecked  $T_{sam}$  and  $T_{bg}$  will be set to one.

These normalization boxes will be checked following the input from the MeasPara.xls file. They can also be done at the interface window of this menu.

1. Clicking ‘Load sample pattern’ will put the sample pattern (given by the sample filename in the parameter file) as an active pattern and pass on  $\tilde{I}_{sam}$  for further processing.
2. Clicking ‘Load background pattern’ will put the background pattern (given by the background filename in the parameter file) as an active pattern and pass on  $\tilde{I}_{bg}$  for further processing.
3. Clicking ‘Subtract background’ will calculate  $I_{subbg}$  and pass it on as an active pattern for further processing.

## Calculate scattering profile



A scattering profile is calculated from the scattering pattern along a radial path starting from the specified origin. The intensity values are obtained from each pixel lied in the path. The number of points which the intensity is obtained is approximately the number of pixels in that path. The radial distance from the origin to a point on the detector plane is converted into the scattering angle  $\theta$  and then to a scattering vector  $q$  in the unit of  $\text{nm}^{-1}$  using the input 'Sample-Detector distance' (in mm), the 'Pixel size' (in mm) and the 'X-ray energy' (in ke V). The scattering angle is given by

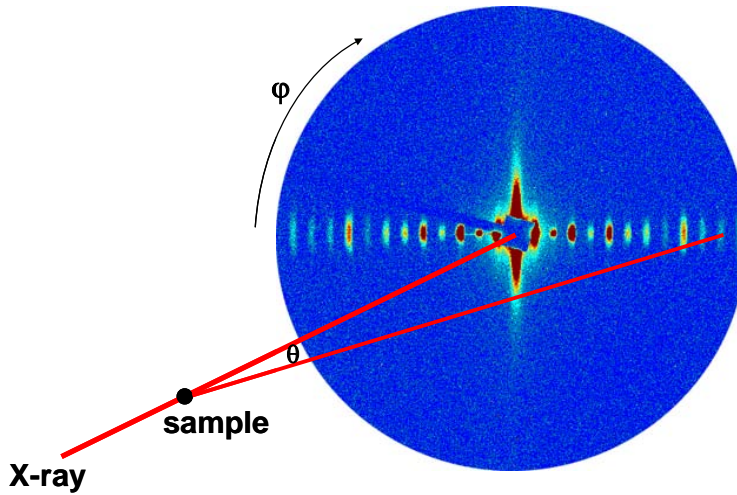
$$2\theta = \tan^{-1}\left(\frac{D}{L}\right),$$

where  $D$  is the distance from the origin to the point on the detector plane, calculated using the pixel size, and  $L$  is the sample-detector distance. The scattering vector is given by

$$q = \frac{4\pi}{\lambda} \sin \theta,$$

where  $\lambda$  is the x-ray wavelength calculated from the specified x-ray energy.

In the 'Choose direction to calculate profile' box, the user is required to enter the azimuthal angle  $\phi$  in which the profile is calculated. The azimuthal angle value is referenced from the left-hand-side horizontal midplane of the pattern (the direction from the center horizontally to the left is 0 degree) and increases in the clockwise direction..



If circular averaging is to be performed the user gives the angular range and angular stepsize for the averaging. For example, if the 'Direction to calculate profile' is 90 deg., the 'Averaging range' is 10 deg. the 'Step size' is 0.25 deg. the profile will then be calculated from 80 deg. to 100 deg. with 0.25 deg. increment. The averaging is performed by integration of the profile,

$$I_{ave}(q) = \frac{\int_{\varphi_{min}}^{\varphi_{max}} I(q, \varphi) d\varphi}{\int_{\varphi_{min}}^{\varphi_{max}} d\varphi}.$$

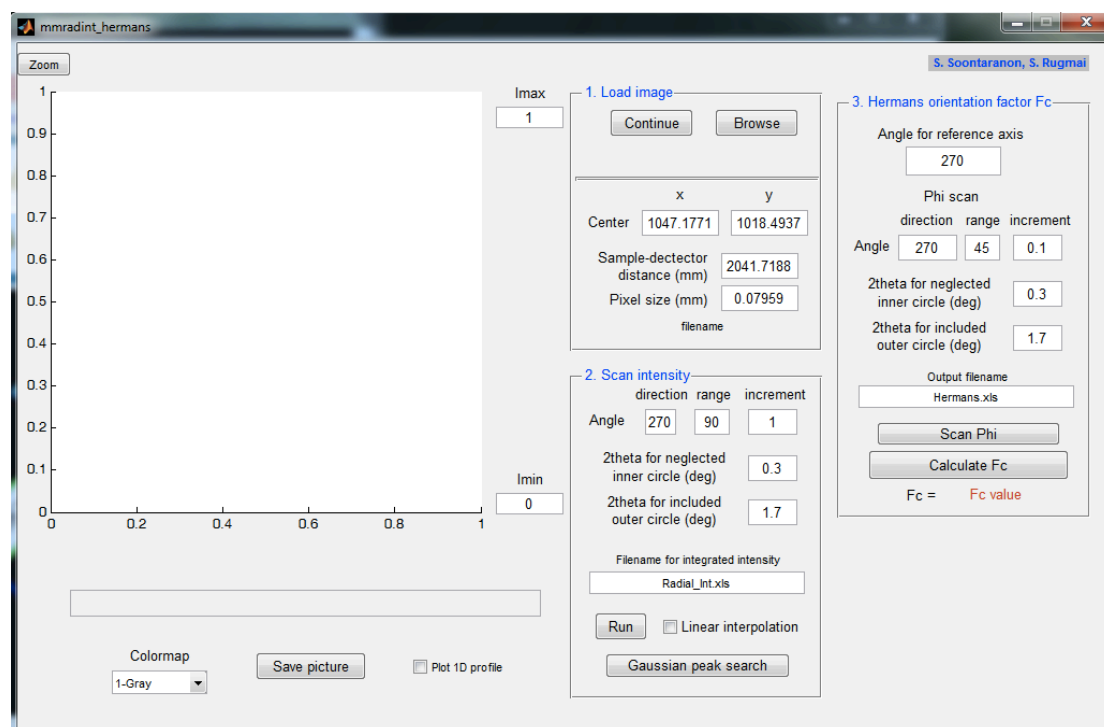
A valid q-range is also calculated just for information when the user provides the beamstop and detector radii. This is the q-range which falls within the distance between the edge of the beamstop and the detector edge.

The outputs are given in an excel file having the filename the same as the pattern filename with a suffix '\_SP\_Xdeg.xls', where X is the profile central angle. The output excel file contains 4 worksheets: 'Averaged I(2theta)' containing circularly averaged intensity profile as a function of 2θ, 'Non-averaged I(2theta)' containing the profile calculated at the central angle as a function of 2θ, 'Averaged I(q)' containing circularly averaged intensity profile as a function of the scattering vector q and 'Averaged I(q) in valid q-range' containing the same data as the worksheet 'Averaged I(q)' but only those in the 'Valid q-range' (this worksheet is given just for convenient, since the data in this range are already contained in the other worksheet anyway). Graphical plots of the profiles are also saved as tiff files.

1. Click either 'Continue' or 'Browse pattern'. Clicking 'Continue' will use the current active pattern while clicking 'Browse pattern' will ask the user to choose a pattern file from the directory.
2. Enter all the values in the boxes.
3. Check 'Do circular averaging' box if circular averaging is to be performed. If this box is unchecked the profile will be calculated for the central angle only.
4. Check 'Use q-space' box (the box is checked by default) to calculate the profile as a function of the scattering vector q. If this box is unchecked the profile will only be given as a function of 2θ in deg.
5. Click 'Calculate scattering profile'.

6. The profile calculation can be stopped by clicking the 'Cancel' button.  
Note that if the 'Read measurement parameters' module is run prior to this module.  
The relevant parameters read from the MeasPara.xls file will automatically be put in  
the boxes of this module.

## Radial integration / Orientation factor



This module can be used to examine the pattern. The intensity can be adjusted by entering the maximum displayed intensity in the 'Imax' box. The center of the pattern can also be estimated by entering the center values in the 'Center' box to move the pattern center. The button 'Save picture' under the displayed image will save the displayed pattern into a JPG file.

The 'Scan intensity' box is used to perform radial integration of the pattern. The program will radially integrate the intensity at each azimuthal angle, starting from the angle ['direction'-'range'] degree to ['direction'+ 'range'] degree with the step of 'increment' degree. The radial integration will be performed in the range between 'Radius of neglected inner circle' and 'Radius of included outer circle'. Clicking 'Run' button will calculate the radially integrated intensity. The results will be written into an Excel file with the specified filename.

The 'Gaussian peak search' button is used to search the peak position. This gives the position of the maximum intensity, which can be used as the reference axis for the calculation of Hermans orientation factor.

The peak search result will be put in the 'Angle for reference axis' box in the 'Hermans orientation factor' calculation box (it can also be entered manually). This reference angle will be the  $\phi=0$  angle in the calculation of the Hermans orientation factor. Clicking 'Scan Phi' button will then radially integrated intensity between the  $\phi=0$  and  $\phi=90$  degrees. Clicking the 'Calculate Fc' button will then calculate the Hermans orientation factor, defined as

$$f_c = \frac{3\langle \cos^2 \theta \rangle - 1}{2}$$

where

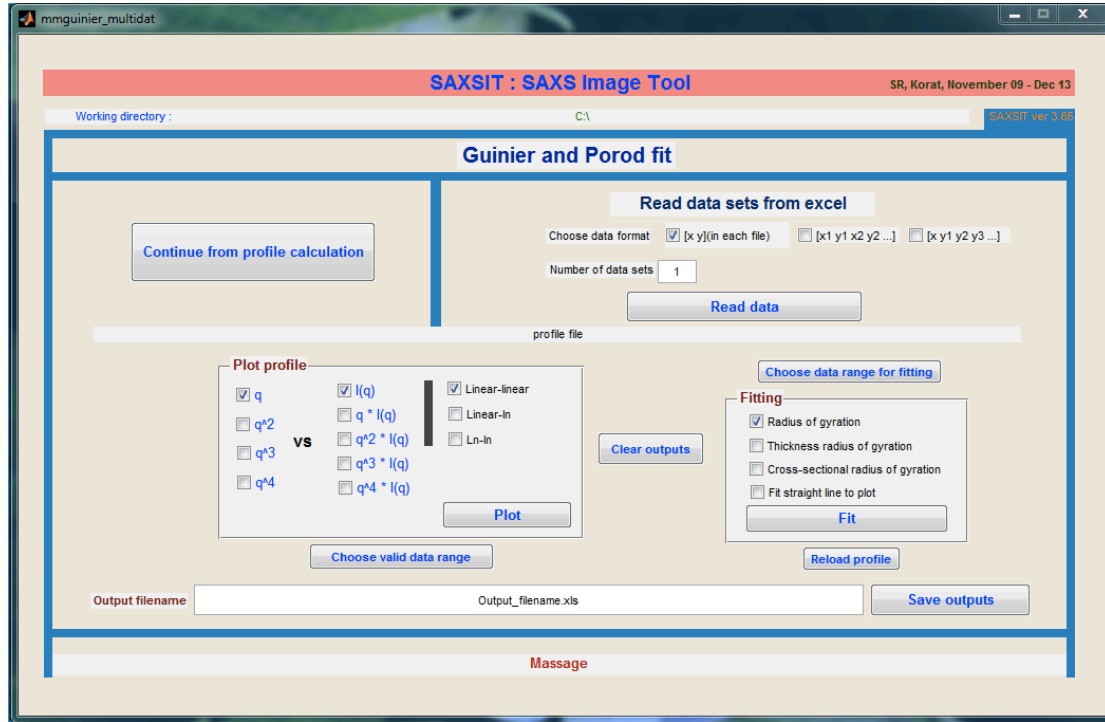
$$\langle \cos^2 \varnothing \rangle = \frac{\int_0^{\pi/2} I(\varnothing) \cos^2 \varnothing \sin \varnothing d\varnothing}{\int_0^{\pi/2} I(\varnothing) \sin \varnothing d\varnothing}$$

And where

$$I(\varnothing) = \int_{0.4}^1 I(2\theta, \varnothing) d(2\theta)$$

is the radially integrated intensity obtained from the ‘Scan phi’ button above. The calculations will be automatically saved in the file with the filename specified in the ‘Output filename’ box.

# Guinier and Porod fit



This module is provided to examine behaviors of the calculated scattering profile. The profile can be plotted as  $q^m$  vs  $(q^n)I(q)$ , where  $m=1,2,3,4$  and  $n=0,1,2,3,4$ , on a linear-linear, linear- ln or ln- ln scale. This can be useful for investigation of dimensionality of the particles via Guinier approximation or mass fractals. The module can also do fitting to estimate slope or various radii of gyration. The module can process multiple data sets simultaneously.

The radii of gyration are defined as follows:

Cross-sectional radius of gyration  $R_c$

$$I(q) \propto \frac{1}{q} \exp(-q^2 \frac{R_c^2}{2})$$

$$R_c = \sqrt{-2 \cdot \text{Slope}[\ln(qI) \text{ vs } q^2]}$$

Thickness radius of gyration  $R_t$

$$I(q) \propto \frac{1}{q^2} \exp(-q^2 R_t^2)$$

$$R_t = \sqrt{-\text{Slope}[\ln(q^2 I) \text{ vs } q^2]}$$

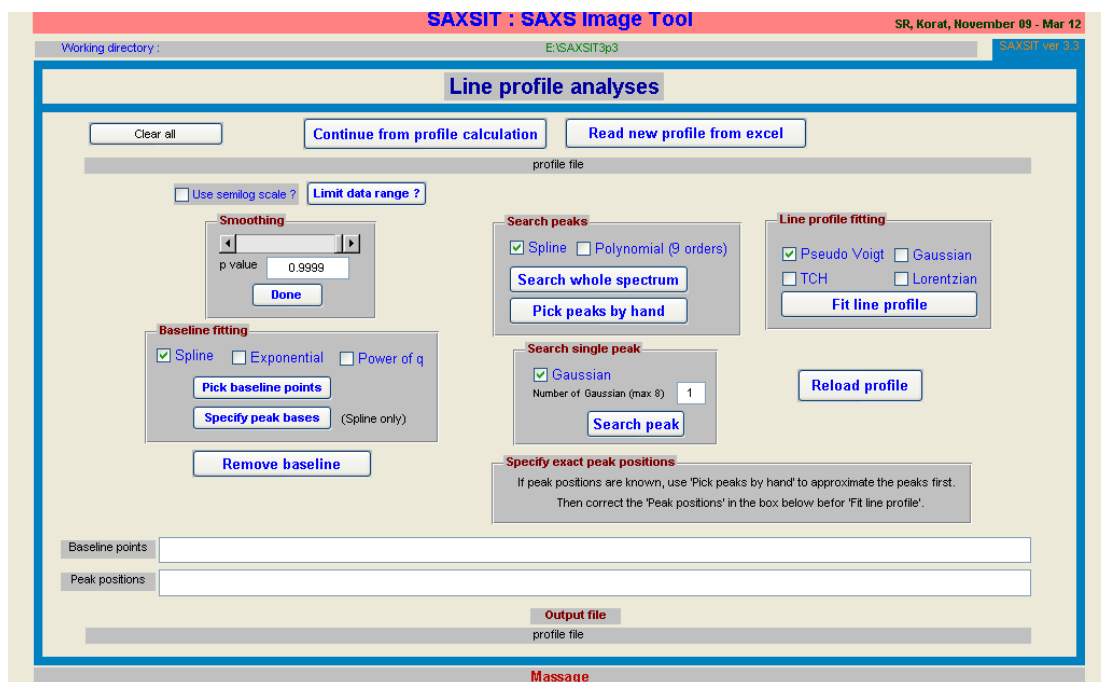
Radius of gyration  $R_G$

$$I(q) \propto \exp(-q^2 \frac{R_G^2}{3})$$

$$R_G = \sqrt{-3 \cdot \text{Slope}[\ln(I) \text{ vs } q^2]}$$

1. If you were processing the measured pattern and just want to continue then click 'Continue from profile calculation'. Clicking 'Continue from profile calculation' will just use the scattering profile from the previous processed pattern. If you want to process data sets from Excel then go to 'Read data sets from excel' block on the right. In this case, multiple data sets can be read in with various formats as indicated with the check boxes. Clicking 'Read data' will ask the user to choose an excel file from the working directory and choose the data to be used. The data must be in the format chosen with the check boxes.
2. Choose the plot type by checking the boxes in the 'Plot profile' area and click 'Plot'. This can be repeated for other plot types.
3. The plotting range can be narrowed by clicking 'Choose valid data range'. This will ask the user to click on two points on the plot to define the plotting range. If the narrowing is overdone the starting profile can be reloaded by clicking 'Reload profile'.
4. Clicking 'Choose data for fitting' will ask the user to click on two points on the plot to define the range of data to be fitted for a slope.
5. Choose the type of radius of gyration to be calculated by checking the boxes in the 'Fitting' area and click 'Fit'. Note that the three types of radii of gyration can be chosen and calculated simultaneously. Note that the three types of radius of gyration have specific definitions. The fitting for the radii of gyration will not depend on the plotting choice chosen in step 2. However, the option 'Fit straight line to plot' will fit for a slope of the chosen plot type. It is also noted that for the radii of gyration the fitted data are supposed to be in the region of Guinier approximation. The value of the fitted radii of gyration will therefore be dependent on the defined range of data.
6. 'Clear outputs' buttons is provided to remove the fit results in case you want to redo the fitting.
7. 'Save outputs' will save the fitting options and fit results to the specified excel file.

## Line profile analyses



This is another assistant module. The module provides basic line profile fitting procedure. It can do baseline removal, data smoothing, peak searching and decomposition of the peaks.

### *Clear all*

In the case that you want to process more than one set of data this button will clear the existing data from the memory.

### *Continue from profile calculation / Read new profile from excel*

The module has two choices to import the data to be analyzed. Clicking 'Continue from profile calculation' will import the latest active data which has been processed earlier. Clicking 'Read new profile from excel' will open a dialog box asking you to choose an Excel file containing the data. The Excel file will then be opened for you to choose the data to be imported. The data must be in two consecutive columns: the first column for the variable (e.g.  $q$  values) and the second column for intensity.

### *Limit data range*

The button 'Limit data range' allows the user to choose the range of data to be analyzed (e.g. to get rid of the beginning and end parts of the data which are probably not good data). 'Use semilog scale' check box is also provided in case some users might want to work in semilog scale.

### *Smoothing*

The smoothing uses cubic Spline smoothing procedure. The 'p value' is the smoothing parameter which runs from 1 to 0. For  $p=1$ , the smoothed result is a natural cubic Spline interpolant of the data, while for  $p=0$  the result will be a least-square straight line fit to the data. What 'p value' should be used depends on how smooth the user would like the result to be. If the result does not look satisfactory the procedure

can be restarted by clicking 'Reload profile' button at the bottom which will activate the original imported data. In case the slider bar for the p-value may be too coarse, the user can just input the number in the edit box (e.g. the default p-value of 0.9999 is often enough to smooth a noisy curve to find a peak).

### *Baseline fitting*

This is provided for baseline removal (e.g. to make the peak symmetric before searching for peak position). The baseline fitting has three choices to fit for a baseline. The Spline choice is general and usually the good one to be used. The other two choices usually require some specific behavior of the data.

Two options are provided to specify the points to fit for the baseline.

'Pick baseline points' will open an interface graph and ask you to click on the graph for the points which will be used to fit for the baseline. Number of points can be as many as necessary, then hit Enter when done. To get reasonable fit, you will need to click a few points between peaks on the expected baseline. The specified points will be displayed in the 'Baseline points' box. These points can be manually altered if required. The numbers in the box will be read in to be used for the baseline fitting.

'Specify peak bases' will open an interface graph and ask you to click on the graph at the bases of the peaks you want (i.e. two points for each peak). The module will then use all the points outside the specified peaks for baseline fitting using cubic spline. However, before it fits for the baseline the module will perform smoothing of the points (using cubic spline with p-value=0.8). This is to avoid large oscillation in the peak area in case the points between peaks are noisy or contain small peaks near the wanted peaks. The specified points for peak bases will be displayed in the 'Baseline points' box. These points can be manually altered if required. The numbers in the box will be read in to be used for the baseline fitting.

After clicking 'Remove baseline' the baseline will then be fitted, and subtracted from the data. If the result does not look satisfactory the procedure can be restarted by clicking 'Reload profile' button at the bottom which will activate the original imported data.

### *Search peaks*

The peak search can be done for the whole spectrum in the 'Search peaks' area, or a single peak search in the 'Search single peak' area. In the 'Search peaks' area there are two choices for fitting. The Spline is usually the workable choice. The Polynomial (9 orders) function means fitting the data with polynomial of up to the 9<sup>th</sup> order (which are rarely useful). The peaks are then searched by differentiation of the fitted results. If the data is noisy the result might be troublesome, in which case some level of smoothing before doing peak search often helps. In this area the button 'Pick peaks by hand' is also provided. Clicking this button will open an interface graph window for you to manually click on the peak positions.

The single peak search uses Gaussian fitting of the data (the number of Gaussian can be chosen between 1-8). The peak can then be immediately obtained from the fitted Gaussian. If the data contain many peaks it might be worthwhile to search each single peak separately by using the 'Limit data range' button, then doing the single peak search.

The searched peaks will be displayed in the white edit box 'Peak positions'. These values can be altered if required. The module will read in the peak positions from this box for the line profile fitting.

### *Specify exact peak positions*

If the exact peak positions are known and you want to use them. You will first have to either have to perform the peak search or activate the button 'Pick peaks by hand' and then pick the peaks approximately on the profile graphs. The peaks positions will then be displayed in the white edit box. You can then correct the peak positions to the known values. When doing line profile fitting the program will read the peak positions from the displayed box. The reason you cannot just put the peak positions values in the box is that the program also need approximate intensity values at the peak positions for the fitting. Picking the peaks from the graph first will give that information.

### *Line profile fitting*

The line profile fitting fits the data using Gaussian, Lorentzian or combination of the two functions. The model functions are defined as follows:

The Gaussian function

$$G(x, x_0) = A \exp \left[ -\frac{(x - x_0)^2}{\sigma^2} \right]$$

where  $A$  is the amplitude and  $\sigma$  is the standard deviation.

The Lorentzian function

$$L(x, x_0) = A \frac{1}{1 + \left( \frac{x - x_0}{\gamma} \right)^2}$$

where  $\gamma$  is the FWHM.

The Pseudo Voigt function is given by the linear combination of Gaussian and Lorentzian components with the same FWHM,

$$F_{PV}(\Delta x) = A \left\{ (2\eta / \pi \Gamma) [1 + 4(\Delta x / \Gamma)^2]^{-1} + (1 - \eta)(2 / \Gamma)(\ln 2 / \pi)^{1/2} \exp(-4 \ln 2 (\Delta x / \Gamma)^2) \right\}$$

where  $\Delta x = x - x_0$ ,  $\Gamma$  is the FWHM and  $\eta$  is the Lorentzian fraction.

The Thompson-Cox-Hastings Pseudo Voigt function (TCH) [J. Appl. Cryst. 20(1987)79] is the Pseudo Voigt function where the FWHM is approximated by the series expansion

$$\Gamma = (\Gamma_G^5 + 2.69269\Gamma_G^4\Gamma_L + 2.42843\Gamma_G^3\Gamma_L^2 + 4.47163\Gamma_G^2\Gamma_L^3 + 0.07842\Gamma_G\Gamma_L^4 + \Gamma_L^5)^{1/5}$$

and the Lorentzian fraction is given by

$$\eta = 1.36603(\Gamma_L / \Gamma) - 0.47719(\Gamma_L / \Gamma)^2 + 0.11116(\Gamma_L / \Gamma)^3.$$

In the fittings, parameters are varied according to the chosen function and the best fit is obtained by least-square fit.

All outputs are provided in Excel files using the filename of the data appended with \_LPA.xls, saved in the working directory.

### *Reload profile*

This will reload the original profile in case you want to start again from the beginning without rereading the data.

Please note that the peak fitting here is for preliminary use. More effective peak fitting (e.g. to calculate crystallinity) should be done with the Peak function fitting module in the 'Fitting' menu.

## Merge data sets

**mmmergedata** SRugmai, March 2012

### Merge two data sets

**Working directory** E:\SAXSIT3p3

This module will read in 2 data sets from Excel files. The second data set will be scaled to match the first data set using straight line fitting for the chosen range of overlapped data. The two data sets (in the chosen ranges) will then be merged.

Note that each data set is required to be in two columns (e.g. q and Intensity).

☐ Use semilog scale  
☐ Use loglog scale

x\_min      x\_max  
0      0

Scaling factor for 2nd data set :  
1

x1\_min      x1\_max      x2\_min      x2\_max  
0      0      0      0

Output filename MergedData.xls

[Messages](#)

This auxiliary module is provided for automatic merging of two data sets. This is mainly intended for merging of data from the same sample taken with different sample-detector distances.

The module will use the overlapped data from the two data sets to fit straight lines for each data set and match the two straight lines to find the scaling factor for the second data set. The second data set will then be scaled and merged to the first data set. The range of data to be merged can be chosen via the interface window.

### *Clear all*

This button is provided to clear all the previous data in the memory. In the case that more than one group of data will be processed after opening the module this button should be used before the next group of data are read in.

### *Read data set from Excel*

This will open a file dialog box for the user to choose the file and the data. The data must be in two consecutive columns: the first column for variable (e.g. q values) and the second column for intensity. The first and second data sets will be read consecutively. The first read in data set will be taken as reference, meaning that the second data set will be scaled to the first data set.

### *Choose data range*

This will ask the user to choose the range of overlapped data that will be used for straight line fitting and calculation of scaling factor. The range of data is chosen via the interface graph window. Check boxes are provided for options to plot the graph in semi-log or log-log scale. If no box is checked the graph will be plotted in a linear scale. The chosen range of data will be displayed in the 'x\_min' and 'x\_max' boxes. These values can also be manually entered or modified.

### *Scale data*

This will read in the range of data from the 'x\_min' and 'x\_max' boxes and use the data in that range to fit straight lines. The straight line will then be scaled to find the scaling factor which will be displayed in the 'Scaling factor for 2<sup>nd</sup> data set' box. This value can also be manually entered or modified.

### *Choose data range to be merged*

This will open the graph window with the plots of the scaled data. The user is asked to two points on the graph to specify the range of the first data set. The same graph window will be open again for the user to choose two points to specify the range of the second data set. The chosen ranges of the two data sets will be displayed in the 'x1\_min', 'x1\_max', 'x2\_min' and 'x2\_max' boxes. These values can also be manually entered or modified.

### *Merge data set*

This will read in the chosen range of data from the displayed boxes. The two data sets within the chosen ranges will then be merged. The result will be saved to an Excel file with the specified 'Output filename'.

## Normalize data by area under curve

This auxiliary module is intended to be used for automatic normalization of intensity measured from the same sample in different condition, e.g. sample measured at different temperatures. The normalization is based on the assumption that the total scattering intensity of the sample within a given range is an invariant.

The module can read in multiple sets of data then calculate the area under curve of each data by integration. The first read in data set will be taken as reference, meaning the remaining data sets will be scaled to match the areas under curves to that of the first data set.

### *Clear all*

This button is provided to clear all the previous data in the memory. In the case that more than one group of data will be processed after opening the module this button should be used before the next group of data are read in.

### *Choose data format*

This allows you 3 options to choose how the data is arranged in the file;

[x y] means each data set is in each separate file with the data stored in two columns, x in the first column and y in the second column.

[x1 y1 x2 y2 ...] means all data sets are in one file with each data set stored in consecutive x and y column pairs.

[x y1 y2 y3 ...] means all data sets are in one file with the data stored in columns. All data sets use the same x values. The first column is x, the second and next columns are y of each data set in consecutive order.

### *Number of data sets*

You are required to specify the number of data sets that will be read in and normalized.

### *Read data set from Excel*

This will open a file dialog box for the user to choose the file and the data. The data must be in two consecutive columns: the first column for variable (e.g.  $q$  values) and the second column for intensity. The user will be asked to choose the file and the data consecutively until all the data sets (specified by the value in 'Number of data set' box) are read in.

### *Choose data range*

This will ask the user to choose the range of data that will be used for normalization. The range of data is chosen via the interface graph window. Check boxes are provided for options to plot the graph in semi-log or log-log scale. If no box is checked the graph will be plotted in a linear scale. The chosen range of data will be displayed in the 'x\_min' and 'x\_max' boxes. These values can also be manually entered or modified.

### *Calculate areas under curves*

This will read in the chosen range of data from the 'x\_min' and 'x\_max' boxes and integrate each data set. The scaling factors (i.e. the ratio between the area under curve of each data set to that of the first data set) will be calculated and displayed in the 'Scaling factor for each data set' box. The scaling factor will be displayed consecutively for each data set, including the first data set which is always 1). These values can also be manually entered or modified.

### *Normalize data and save*

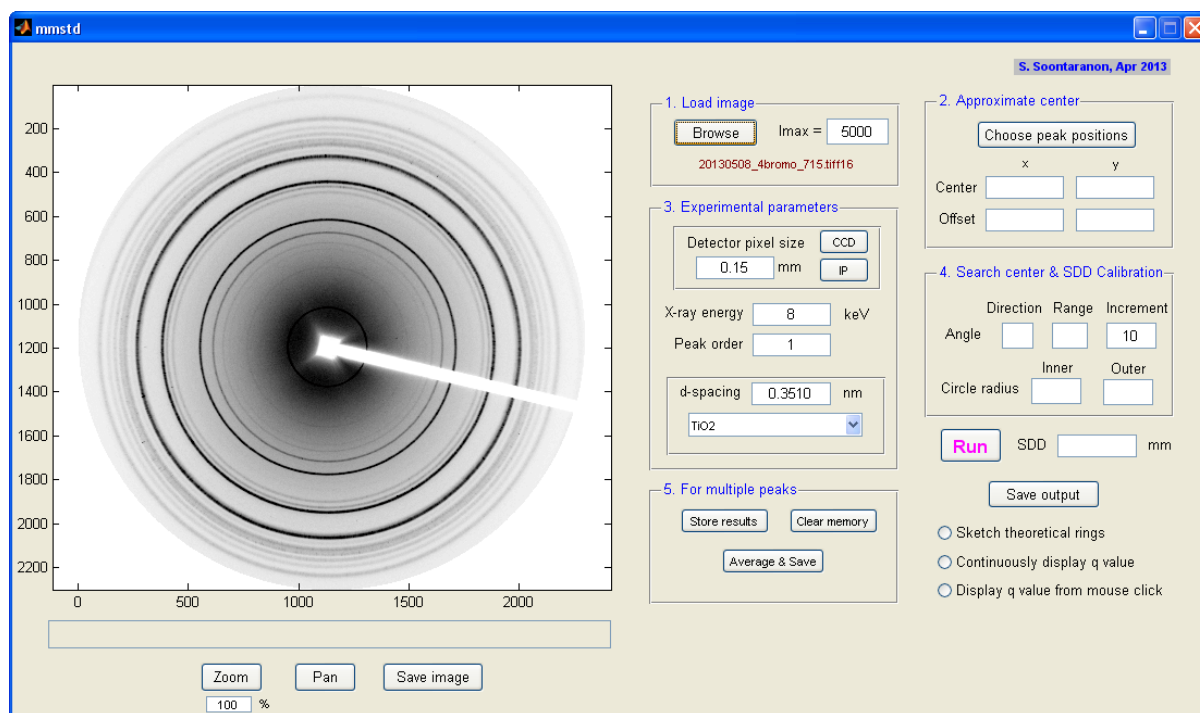
This will read in the scaling factor from the 'Scaling factor for each data set' box. The data sets will then be scaled and saved in an Excel file with the specified 'Output filename'. Each normalized data set will be written in a separate worksheet of the Excel file, together with its scaling factor and area under curve before scaling.

## Example 1: Calibration of sample-detector distance and primary beam position using standard sample

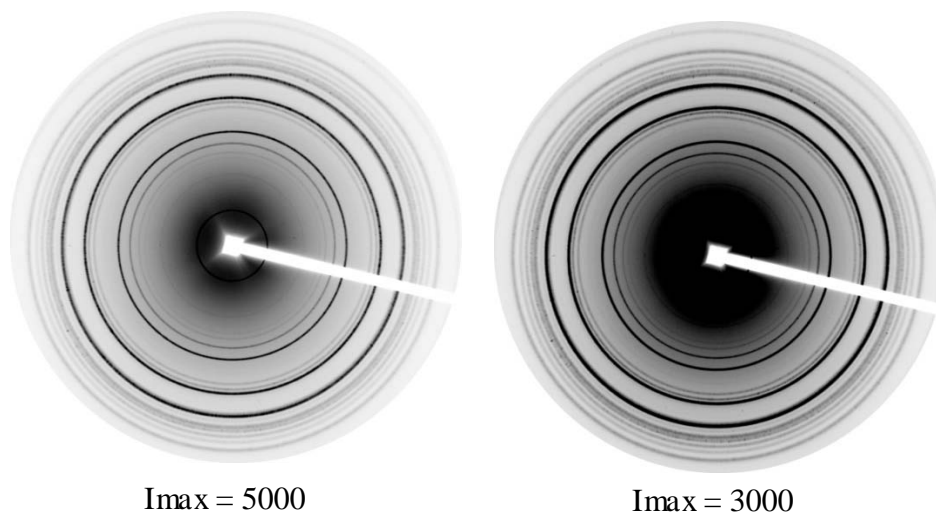
1. Put the standard pattern file into a new folder.
2. Open SAXSIT software and put the folder name into “Set working directory” box.



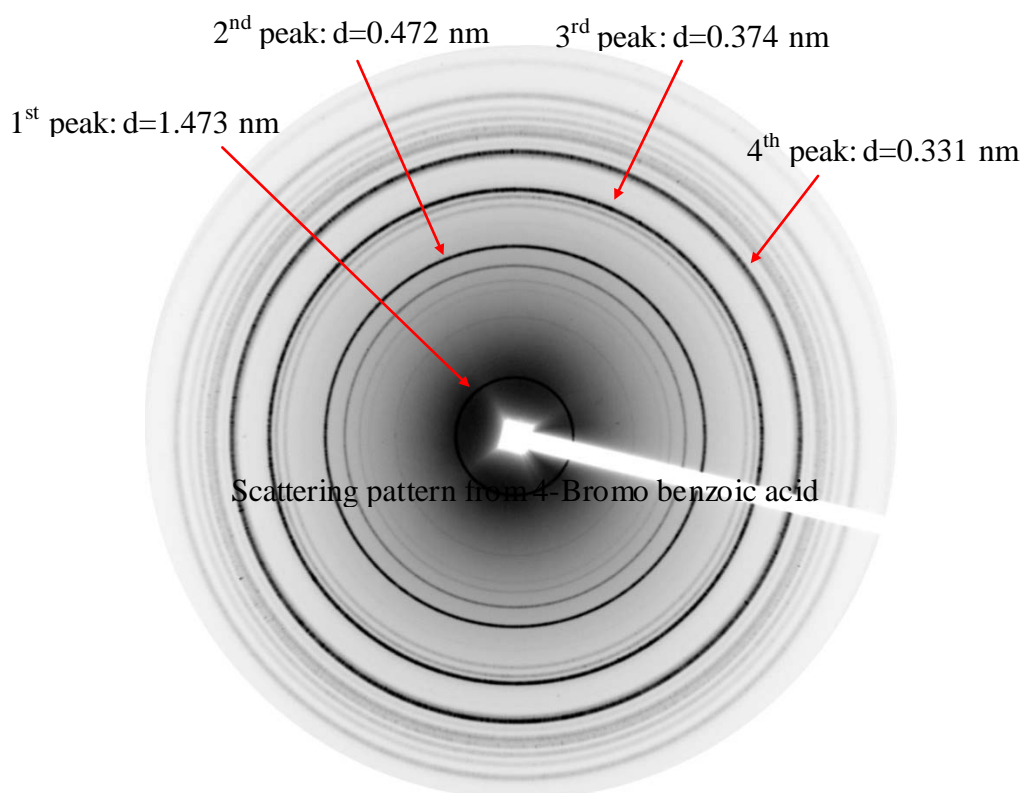
3. Click “Analyze standard” button, a new window “mmstd” will be opened.
4. In the “Load image” area, click “Browse” and select the standard file. The scattering image of standard is displayed on the left.



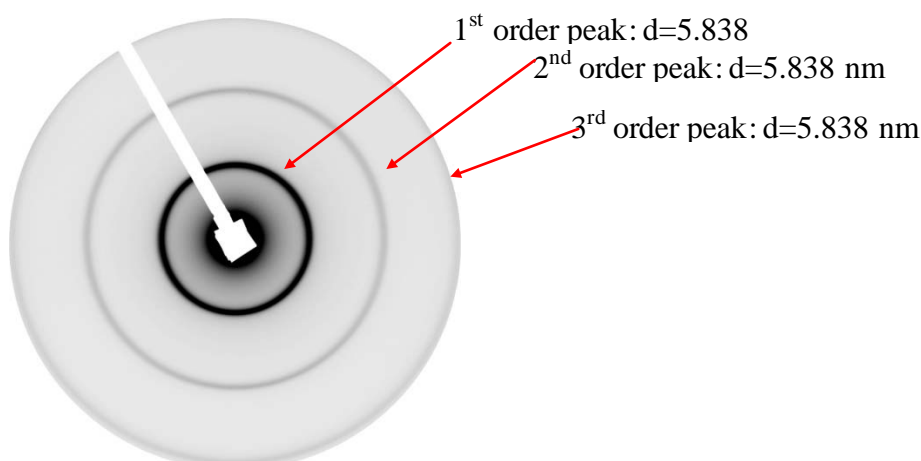
5. The number “Imax” in the “Load image” area can be changed for visualization purpose. The larger the number, the brighter the image.



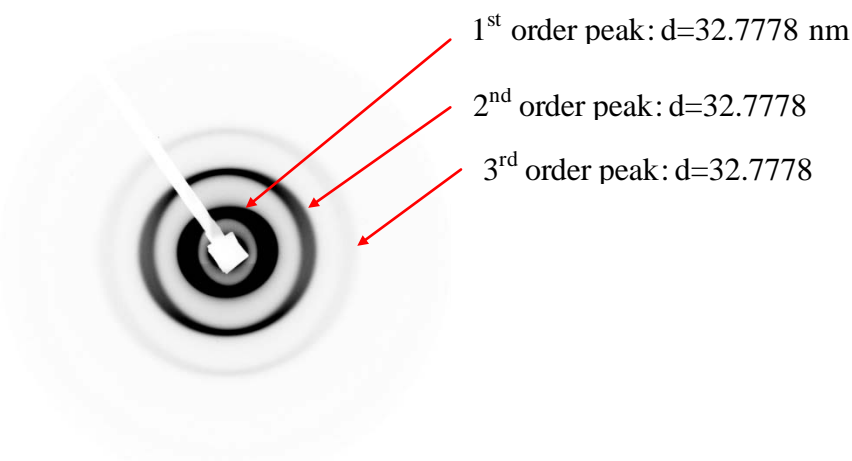
6. Make a decision on which peak to be analyzed. Some standard such as 4-Bromo benzoic acid give a lot of peaks. They come from different d-spacing and are not the order of each other. In this case, it is recommended to use the first fourth most intense peak as illustrate in the figure below.



Other standards such as silver behenate or SEBS may give several peaks. However they are order peak arising from the same d-spacing structure.



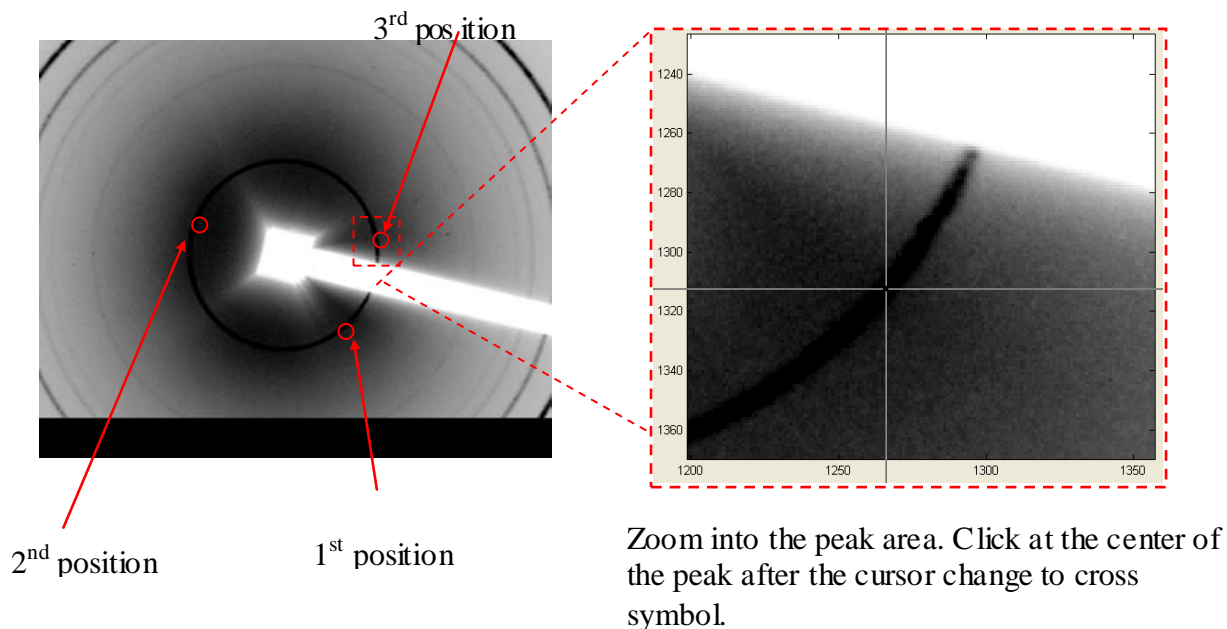
Scattering pattern from Silver Behenate



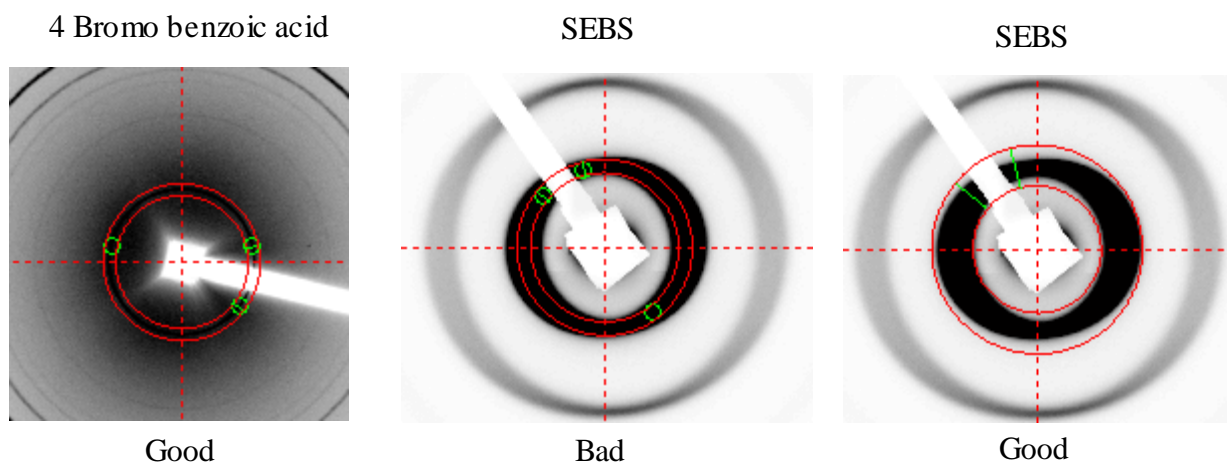
Scattering pattern from SEBS

If more than one peaks are available, it is recommended to analyze every peak and average the results.

7. In this example, we will consider the scattering pattern from 4-Bromo benzoic acid. First, the 1<sup>st</sup> peak with 1.473 nm d-spacing will be analyzed. Choose three positions on perimeter of this peak by clicking at "Choose peak positions" button located in the "Approximate center" area. Immediately after clicking the mouse pointer will change to a magnification glass symbol for 5 second, enough time for zooming. Click(s) or drag on the desired position of the peak. Then, the pointer change to cross symbol, click on the center position of the peak (see the picture in the next page). Repeat this process for three positions in clockwise direction start from next to the beamstop holder, at the opposite of the beamstop holder, and end at the other side of the holder.

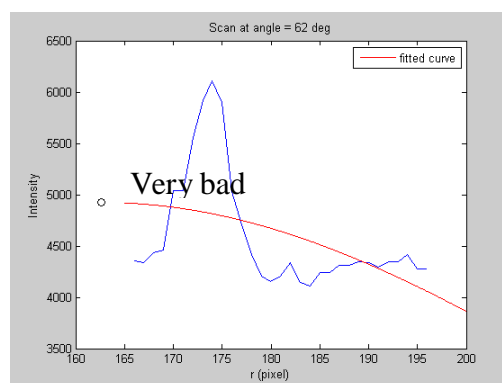
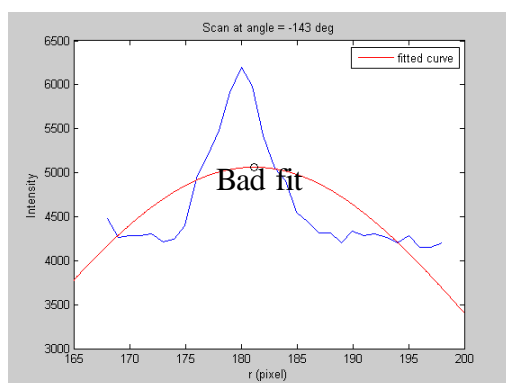
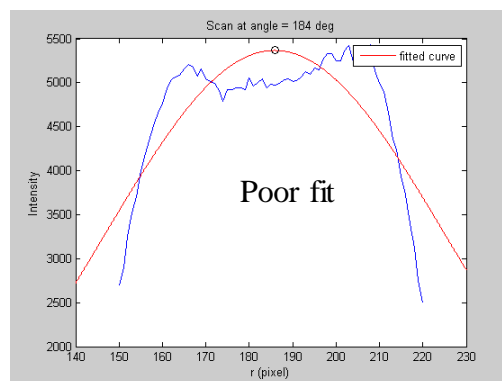
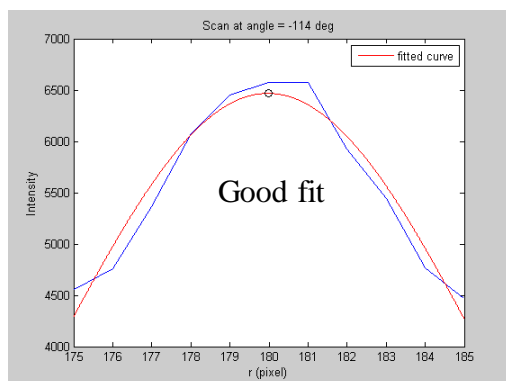


After finish choosing three positions, the software draw two red circles enclose the peak while green circular symbols indicate the clicked positions. Make sure that the peak in black color resides between the two red circles. Some standard give broad peak, this cause the peak to extend over the area between two red circles. This can be corrected by decreasing the size of inner circle and increasing the size of outer circle in the “Search center & SDD Calibration” area.



8. Change the experimental parameters which consist of detector pixel size, x-ray energy, peak order, and d-spacing value. The d-spacing values can be changed manually or by selecting the type of standard sample from the drop down list menu. For 4-Bromo benzoic acid, always set the peak order equal to 1.

9. Click “RUN” to begin the calculation. A new window will pop up and plot the peak profile together with its Gaussian-fitted result. Give attention to each plot, the fitted result should give a reasonable peak position. If not, try to change the size of the two red circles and click “RUN” again.



10. When finish, the sample-detector distance and the center position of the peak will be provided. The software also calculates the different of the results with that obtained in the previous calculation. Adjust the size of the two red circles such that a good fit is obtained and repeat the process for a few times (normally 3-4 times). A small different (less than 0.01% ) indicates that the calculation can be stopped.

Center position

Sample-detector distance

Percent different with previous calculation

If only one peak is to be analyzed, press the “Save output” to save the result into text file. If there are more than one peak, press the “Store results” button located in the “For multiple peaks” area. Then, repeat from step 7 for the next peak. When all peaks are analyzed, press “Average & Save” button to save the averaged results into text file. Each row in the output text file corresponds to the analysis from each peak. The last row is the averaged result that can be used in the data reduction process.

## Example 2: Processing SAXS patterns for nano particles dispersed in water

This example demonstrates the procedure how to use SAXSIT to process SAXS patterns from measurements of nano particles dispersed in liquid. The measurement used Silver Behenate as a standard for pattern alignment and sample-detector distance calibration.

The files needed for this example is contained in the folder 'Examples\_nanoparticles'.

### *Initial preparation*

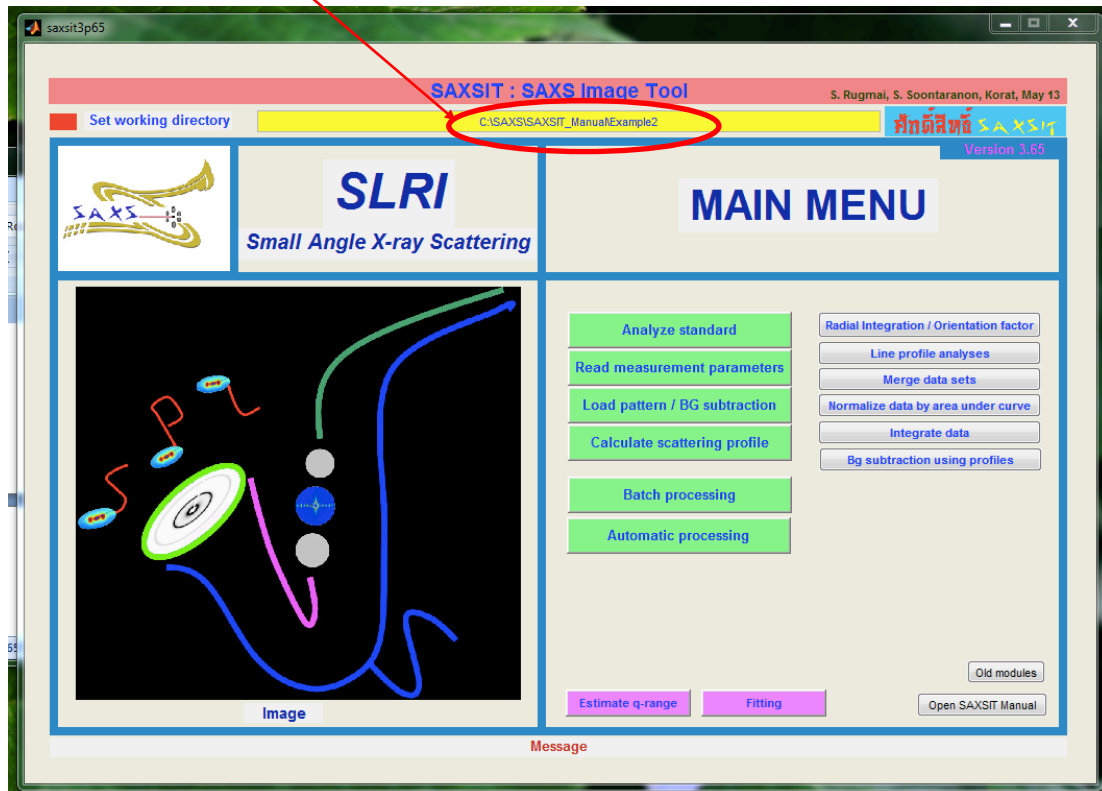
1. Create a folder (here the folder is C:\SAXS\SAXSIT\_Manual\Example2) consisting of the following files
  - 1.1 The file MeaPara.xls
  - 1.2 SAXS pattern of silver behenate (file 20130402\_AgBH.0001.mccd)
  - 1.3 SAXS pattern of sample (file 20130402\_Au20nm.0015.mccd)
  - 1.4 SAXS pattern of buffer (i.e. water in this case) (20130402\_Au20nm.0013.mccd)
  - 1.5 The text files containing values of reading from ionization chamber and photodiode for the reference (empty cell, file 20130402\_Au20nm.0011.txt), buffer measurement (cell with water, file 20130402\_Au20nm.0013.txt) and sample measurement (gold nano particle dispersed in water, file 20130402\_Au20nm.0015.txt).
2. Open the file MeaPara.xls and enter the measurement parameters in column B. Note that the calculation parameters in the section 'Parameters obtained from standard sample' will be set later after 'Analyze standard' is done. In the 'Profile calculation parameters', the 'Normalize beam current' and 'Normalize transmission' are set to 'Y'. These are usually needed for background subtraction of solution samples. Note that by 'Normalize beam current' uses integrated beam intensity values for normalization. Therefore the exposure time will automatically be normalized as well. The sample concentration value is usually set to 0, meaning the concentration is low enough to be negligible.

B23		20130402_AgBH.0001.mccd				
	A	B	C	D	E	F
1	<b>Experimental condition</b>					
2	Title	Au20nm_Ga_10mM				
3	X-ray energy (keV)	8				
4						
5	<b>Dark current</b>					
6	Dark current pattern filename					
7	Accumulated time (sec)					
8						
9	<b>Reference</b>					
10	Parameters for reference pattern (text file)	20130402_Au20nm.0011.txt				
11						
12	<b>Sample</b>					
13	Sample pattern filename	20130402_Au20nm.0015.mccd				
14	Parameter for sample pattern (text file)	20130402_Au20nm.0015.txt				
15	Median mask (Y/N)	Y				
16						
17	<b>Background</b>					
18	Background pattern filename	20130402_Au20nm.0013.mccd				
19	Parameter for background pattern (text file)	20130402_Au20nm.0013.txt				
20	Median mask (Y/N)	Y				
21						
22	<b>Parameters obtained from standard sample</b>					
23	Standard pattern filename	20130402_AgBH.0001.mccd				
24	Center offset X (pixels)	5.01		1155		
25	Center offset Y (pixels)	-13.88		1136.1		
26	Sample to detector distance (mm)	1002.92				
27						
28	<b>Detector parameters</b>					
29	Pixel size (mm)	0.07959	CCD = 0.07959, IP = 0.15			
30	Detector radius (mm)	82.5	CCD = 82.5, IP = 172.5			
31						
32	<b>Profile calculation parameters</b>					
33	Normalize beam current (Y/N)	Y				
34	Normalize exposure time (Y/N)	N				
35	Subtract dark current pattern (Y/N)	N				
36	Normalize transmission (Y/N)	Y				
37	Sample concentration	0	Put 0 if concentration is unknown			
38	Direction to calculate profile (deg)	270				
39	Averaging range (deg)	10				
40	Step size (deg)	0.25				

### 3. Run SAXSIT

#### 3.1 Set working directory to be the same as the folder created in step 1

Set working directory here



3.2 Click menu Analyze standard to analyze the standard pattern. Follow the procedure in Example 1.

3.3 Click 'Read measurement parameters'

3.4 In the 'Read measurement parameters' window, click 'Read from excel file', select the file Measpara.xls created in step 2. The parameters will be displayed.

3.5 Click 'Save parameters'

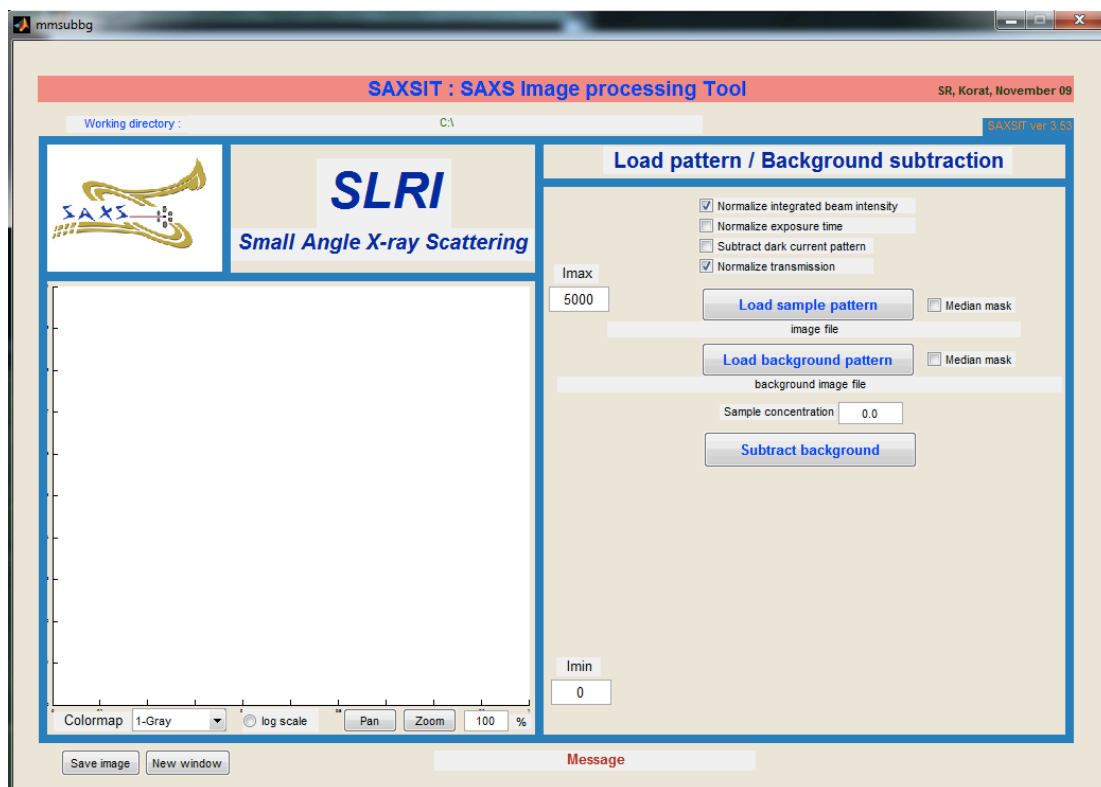
#### *Calculate scattering profile of the sample*

4. From the 'MAIN MENU' window click 'Load pattern / BG subtraction'. Note that all the parameters, including check boxes will already be set according to the inputs in the MeasPara.xls file. They can, however, be modified here, if desired.

4.1 Click 'Load sample pattern'.

4.2 Click 'Load background pattern'.

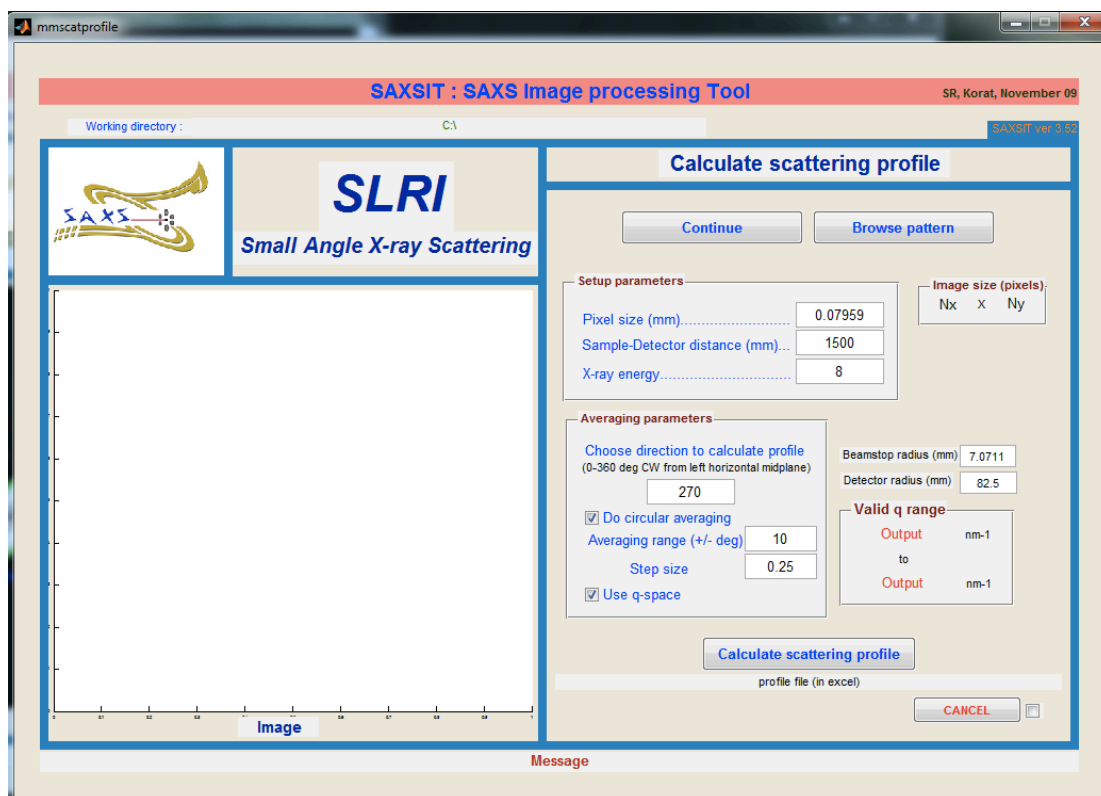
4.3 Click 'Subtract background'.



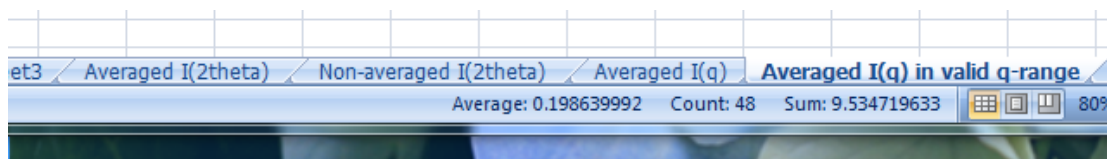
5. From the 'MAIN MENU' window click Calculate scattering profile. Note that all the parameters, including check boxes will already be set according to the inputs in the MeasPara.xls file. They can, however, be modified here, if desired.

- 5.1 Click 'Continue'.

- 5.2 Click 'Calculate scattering profile' and wait.



The output is plotted as a new figure. The numbers are saved as Excel files in the working directory, with the filename the same as that of the sample pattern suffixed by \_SP\_Xdeg.xls, where X is the 'direction to calculate profile'. The output file from 'Calculate scattering profile' contains 4 worksheets, 'Averaged I(2theta)' containing circularly averaged intensity as a function of  $2\theta$ , 'Non-averaged I(2theta)' containing scanned intensity, as a function of  $2\theta$ , at the 'direction to calculate profile' angle, 'Averaged I(q)' containing circularly averaged intensity as a function of the scattering vector  $q$ , 'Averaged I(q) in valid q-range' containing the same data as the previous worksheet but only in the range of the detector and outside the beamstop (the range is calculated from the 'Beamstop radius' and 'Detector radius' values).



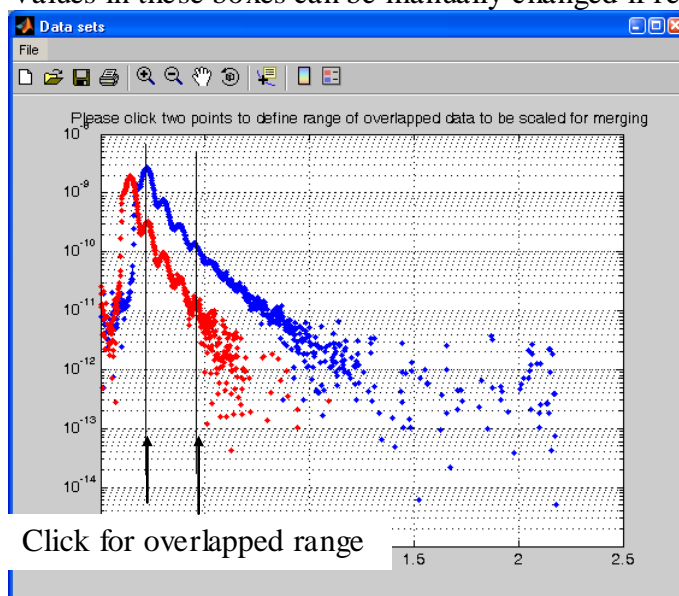
### Example 3: Merge data sets

Two output files, PMMA1430.xls and PMMS2430.xls, from measurements of the same PMMA nano particles samples at two different sample-detector distances are given in the example folders. The two data sets can be merged to give a single data set which covers the whole q-range from the original two data sets.

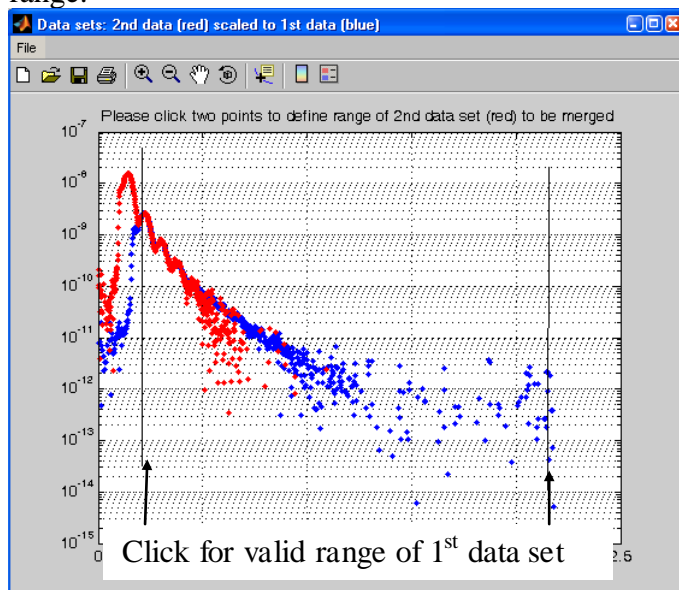
1. Open SAXSIT3.65 and set working directory to the folder which the files PMMA1430.xls and PMMS2430.xls are located.
2. In the MAIN MENU, click 'Merge data sets'

3. Click 'Read data from Excel', the file dialog box will open for you to choose the file.
4. Choose the file PMMA1430.xls, this will be the reference data. The Excel file PMMA1430.xls will then open and you are asked to choose the data.
5. Click the worksheet 'Averaged I(q)' and choose the values of q and Intensity, then click 'OK'.

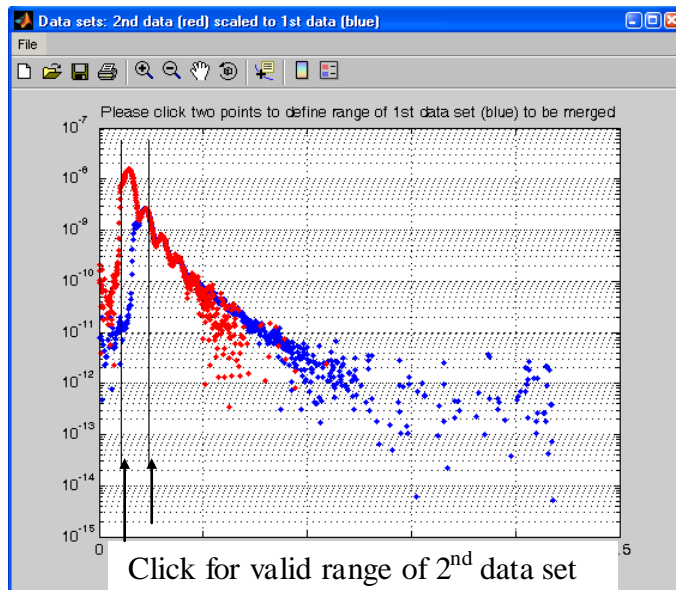
6. The file dialog box will open again. Choose the file PMMS2430.xls.
7. Click the worksheet 'Averaged I(q)' and choose the values of q and Intensity, then click 'OK'.
8. Check the box 'Use semilog scale'. This will help seeing data more clearly.
9. Click 'Choose data range'. The graph window will appear for you to choose the data range.
10. Click two points on the graph to cover the range where the two data sets overlap. The data range will be displayed in 'x\_min' and 'x\_max' boxes. Values in these boxes can be manually changed if required.



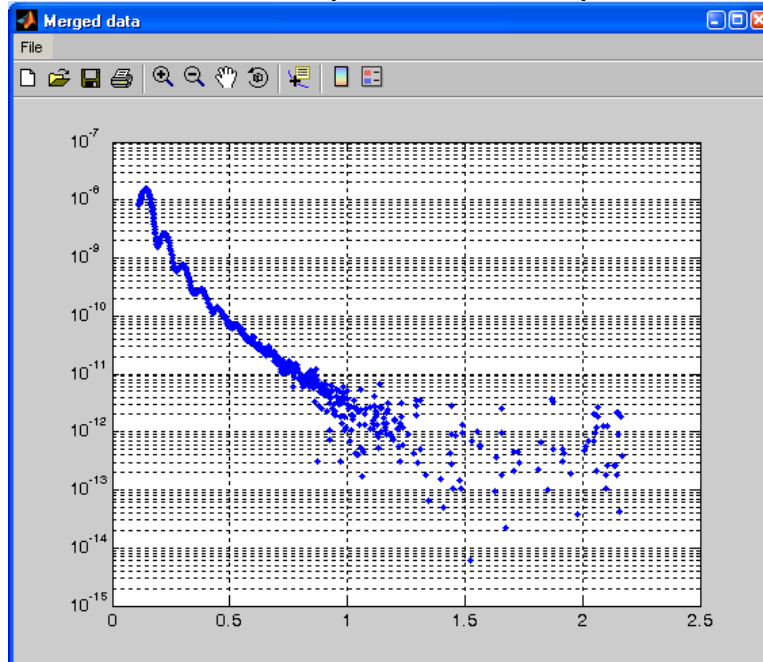
11. Click 'Scale data', the second data set will be scaled. The scaling factor is displayed in the 'Scaling factor for 2<sup>nd</sup> data set:' box. This value can be manually changed if required.
12. Click 'Choose data range to be merged'. A graph window will appear and you are asked to click two points on the graph to choose range of first data set to be used. Here we will choose the large range of the first data and will only choose the small range at low q of the second data with a small overlapped range.



13. A graph window will appear again and you are asked to click two points on the graph to choose range of second data set to be used.



14. Enter output filename in the 'Output filename' box.
15. Click 'Merge data'. The data within the chosen range of each set will be merged. Note that if you want to connect the two data sets without the overlap region you can just manually set the values in the 'x1\_max' and 'x2\_min' boxes to the same value. The merged data and the scaling factor will be written to the Excel file specified in the 'Output filename'.

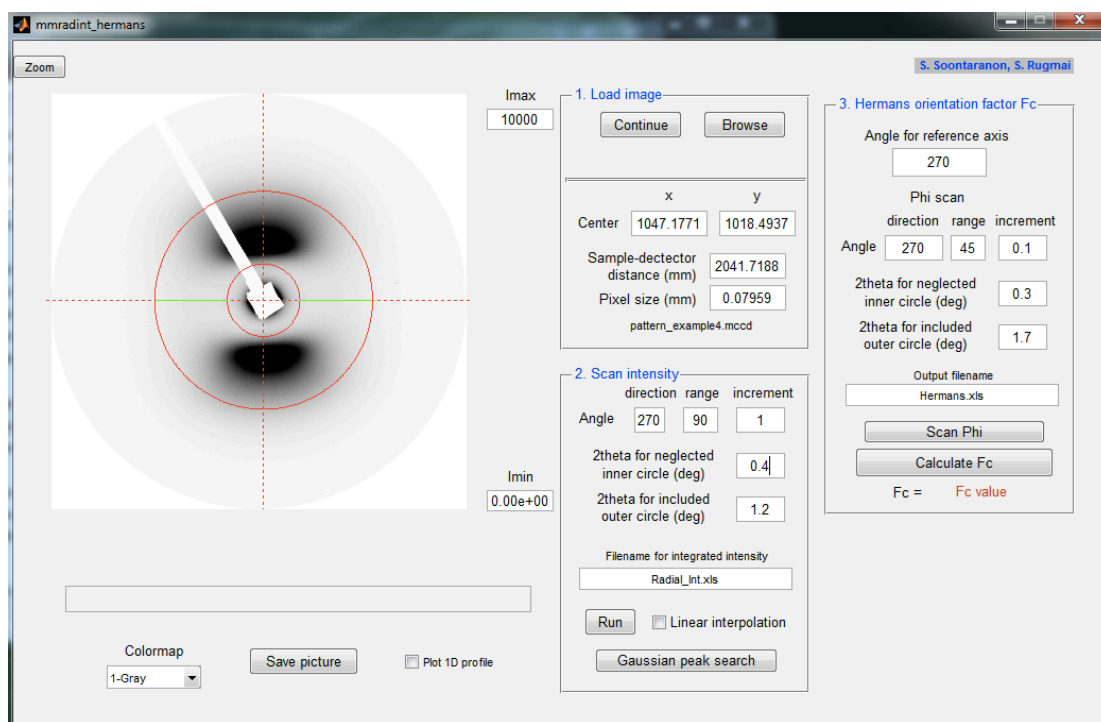


## Example 4: Calculation of Hermans orientation factor

The scattering pattern file of a drawn polymer sample, filename pattern\_example4.mccd is included in the Example4 folder.

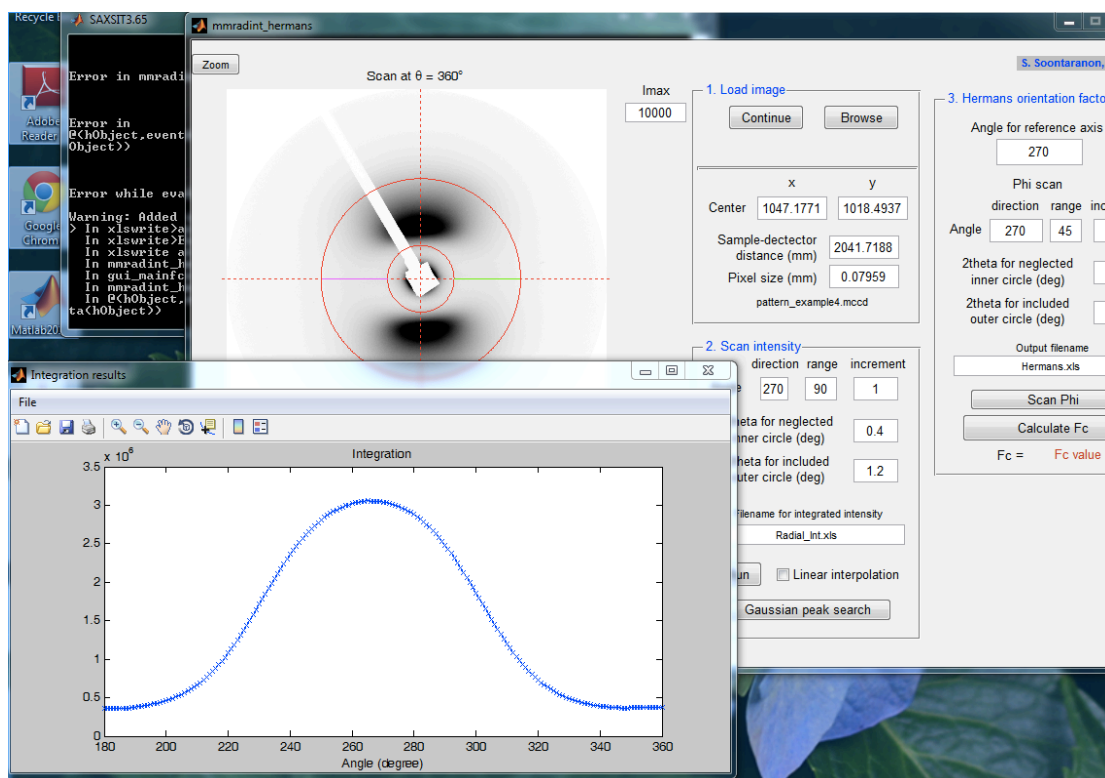
First, the values for the X and Y center position (in pixels) and the sample-detector distance (in mm) have to be known to align the sample pattern before calculations. These values can be obtained from 'Analyze standard' module (see Example 1). The standard pattern file AgBh.mccd is also included for this purpose. (Below, these alignment procedure is assumed to be done and the center values are therefore assumed to be known.)

1. Open SAXSIT3.65 and set working directory to the folder containing the file pattern\_example4.
2. Click the 'Radial integration / Orientation factor' menu on the Main Menu.
3. Click 'Browse' and open the pattern file.

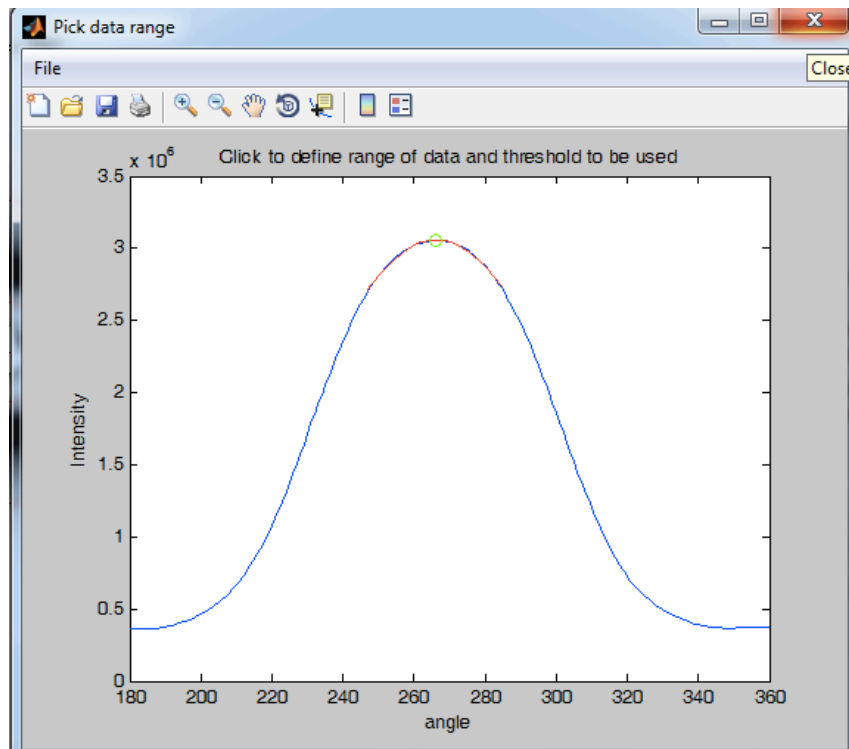


4. Input the values for X center (1047.1771), Y center (1018.4937), Sample-detector distance (2041.7188) (these values are obtained from the output file of 'Analyze standard' module), and the Pixel size (0.0769 mm for Mar165 CCD detector). You can also change the Imax value to see the pattern more clearly. The scattering pattern can be seen to have a double symmetric loops. This is a typical scattering pattern of a drawn polymer sample with the drawing direction being in the direction of the maximum intensity. The maximum intensity of the bottom loop appears at around the azimuthal angle of 270 degree. We will first therefore scan intensity to obtained radially integrated intensity as a function of azimuthal angle phi between phi = 180 to phi = 360 degrees.

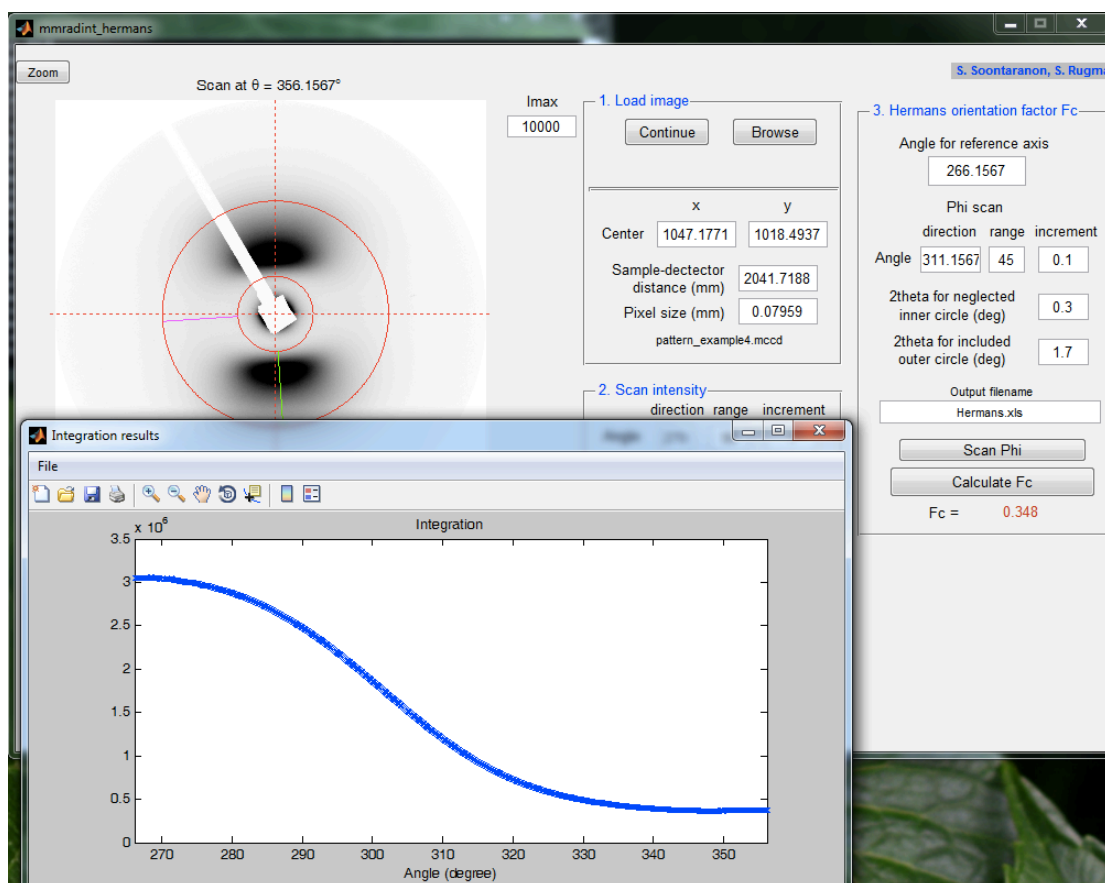
- Set the values in the 'Scan intensity' panel for 'direction'=270, 'range'=90, 'increment'=1 degrees. Choose integration range over 2theta to cover the peak position (here we choose 0.4 degree for inner circle and 1.2 degree for outer circle).
- Click 'Run'. The program will scan radially integrated intensity and plot the integrated intensity as a function of azimuthal angle. The results will also be written into the specified Excel file.



- Click 'Gaussian peak search'. The pointer will change to a cross bar to ask you to click on the plot to specify the minimum intensity to search for the peak. Click somewhere under the peak. The program will then search for the peak position and automatically put the peak value in the 'Angle for reference axis' box in the 'Hermans orientation factor' panel. The Phi scan angles will also be set automatically to scan for  $\Phi=0$  to  $\Phi=90$  degrees, where the  $\Phi=0$  is identified as the value in the 'Angle for reference axis'. The 'increment' is set at 0.1 degree as a default (this value should not be too large, since it is also the step size used for integration when calculating the orientation factor). These values may also be manually modified if required. The integration range over 2theta (inner circle and outer circle values) can also be modified.



8. Click 'Scan Phi' to scan for integrated intensity.
9. Click 'Calculate Fc' to calculate the Hermans orientation factor.

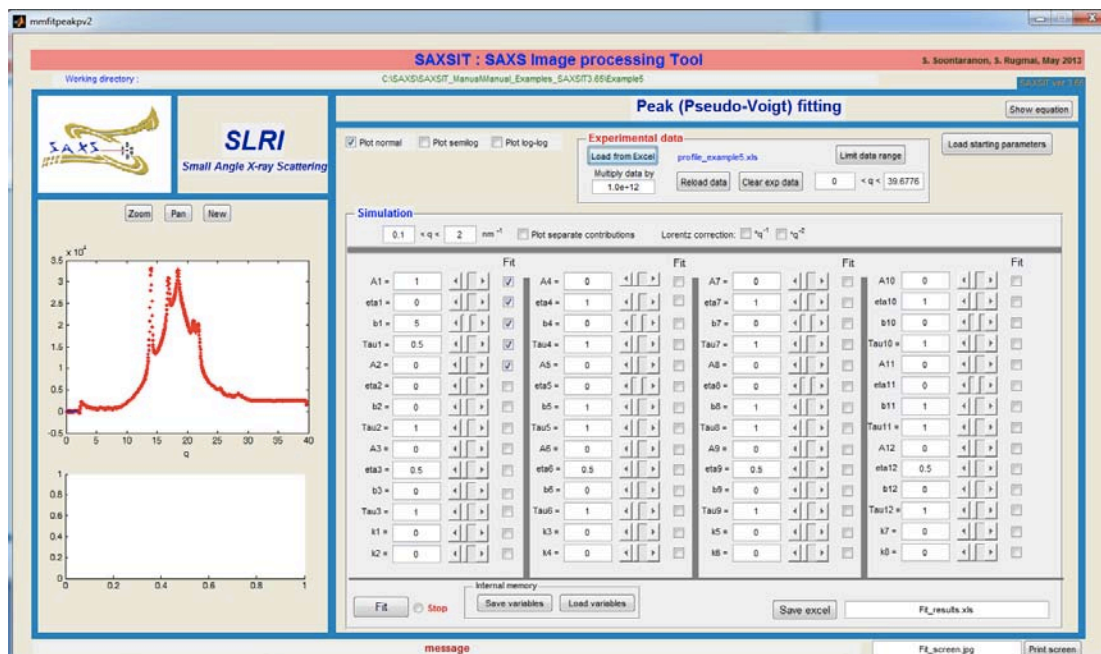


The calculated Hermans orientation factor is displayed in the window. The results will also be written to the specified Excel file. The calculated  $F_c$  value is between -0.5 to 1.0. The obtained positive value indicates that the scattered structure (i.e. the lamellae) is oriented parallel to the reference axis ( $F_c = 1$  implies perfect parallel orientation). If the orientation is perpendicular to the reference axis, the  $F_c$  is negative ( $F_c = -0.5$  implies perfect perpendicular orientation). Completely random orientation gives  $F_c = 0$ .

## Example 5: Peak fitting of WAXS data for calculations of crystallinity

This example will show how to use the peak fitting module to fit WAXS data using Pseudo Voigt functions. The Excel file `profile_example5.xls` is included in the Example5 folder. The file contains measured WAXS scattering profiles of a polymer sample, obtained from 'Calculate scattering profile' menu.

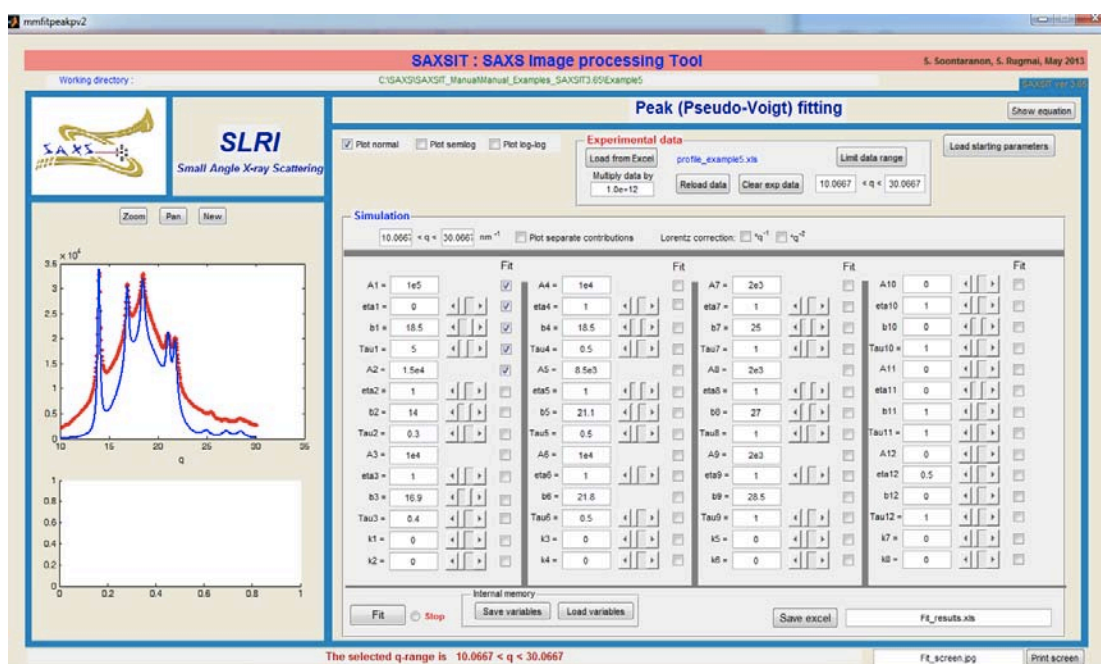
1. Open SAXSIT3.65 and set working directory to the folder containing the file `pattern_example4`.
2. Click the 'Fitting' menu on the Main Menu.
3. Click 'Peak Pseudo Voigt' menu on the 'Fitting Menu'
4. In the 'Experimental data' panel, click 'Load from Excel'. The file dialog box will be opened. Choose the file `profile_example5.xls` and open it. The Excel file will open together with the small 'Data selection dialog' box, choose the data in the worksheet 'Averaged I(2theta)' and click OK in the 'Data selection dialog' box. Note that, choosing the data can usually be done by clicking on the worksheet name in the bottom of the Excel file (the number data in the worksheet will be heightened). Otherwise, you can just open the required worksheet and make selection of the data (all the numbers in the 2 columns, without headers). The data will then be plotted in red in the window. Note that the data is multiplied by the scaling in the 'Multiply data by' box. This is just to scale the data for some data set which is too small and inconvenience for fitting. Since most of the cases, the scattering intensity is in arbitrary unit anyway, the scaling will therefore not affect the fit results.



5. Click 'Limit data range'. The pointer change to a cross-bar in the data plot window. Click 2 points on the plot to select range of  $2\theta$  for the data to be fitted. The selected range should include all the peaks that will be fitted. For example, in this case we choose the range between 10 and 30 degrees

(click at the positions  $q=10$  and  $q=30$  on the data plot which appears to contain 8 peaks). Note that the x-axis in the plot is always 'q'. This does not mean the data is actually plotted as a function of q, but it is plotted as a function of whatever read in from the first column of the Excel file (in this case it is 2theta). Next, before start fitting, we have to try to generate the peaks as close to the data as possible in order to give the program the starting parameters to carry out the fitting effectively.

6. Guess the parameters of the peaks in the data. Each peak have 4 parameters, the amplitude 'A', the Lorentzian factor 'eta', the peak position 'b' and the full width half maximum (FWHM) of the peak 'Tau'. For WAXS data, the first peak should represent scattering from amorphous. This first peak should therefore be something like a broad Gaussian peak, centered at around the center of the data. The 8 peaks appear in the data are from scattering of crystalline components, and should therefore be represented by narrow peaks. We have to add the peak one-by-one and see what the calculation looks like in the blue line plot. In this case the guesses are shown in the figure below.

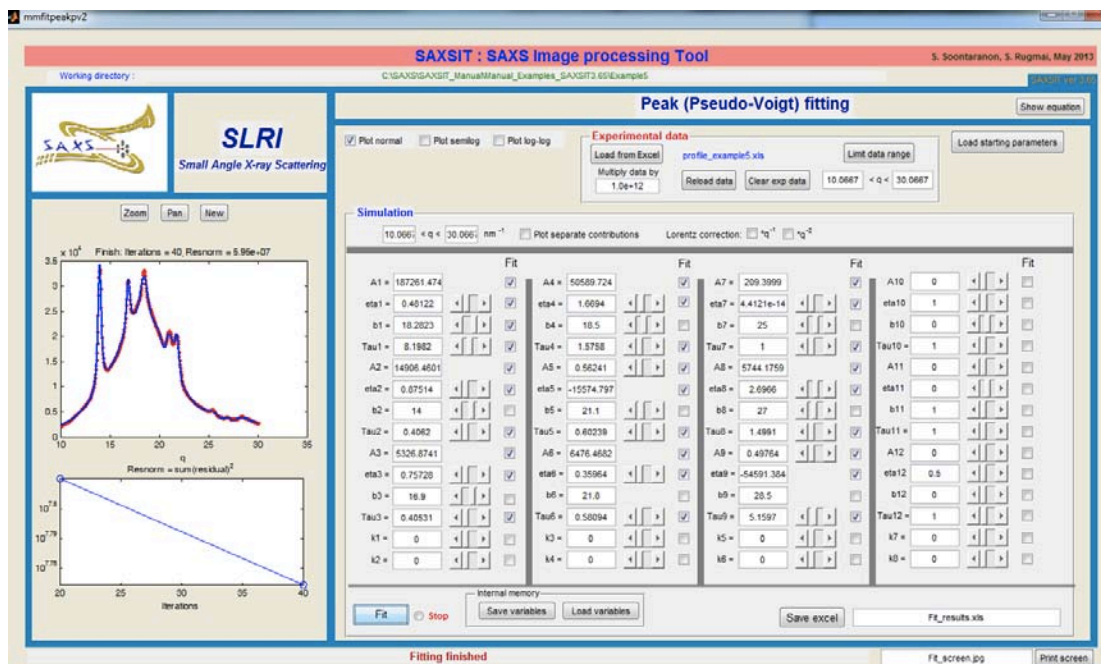


Fit			Fit			Fit			Fit		
A1 = 1e5	<input checked="" type="checkbox"/>		A4 = 1e4	<input type="checkbox"/>		A7 = 2e3	<input type="checkbox"/>		A10 = 0	<input type="checkbox"/>	
eta1 = 0	<input checked="" type="checkbox"/>		eta4 = 1	<input type="checkbox"/>		eta7 = 1	<input type="checkbox"/>		eta10 = 1	<input type="checkbox"/>	
b1 = 18.5	<input checked="" type="checkbox"/>		b4 = 18.5	<input type="checkbox"/>		b7 = 25	<input type="checkbox"/>		b10 = 0	<input type="checkbox"/>	
Tau1 = 5	<input checked="" type="checkbox"/>		Tau4 = 0.5	<input type="checkbox"/>		Tau7 = 1	<input type="checkbox"/>		Tau10 = 1	<input type="checkbox"/>	
A2 = 1.5e4	<input checked="" type="checkbox"/>		A5 = 8.5e3	<input type="checkbox"/>		A8 = 2e3	<input type="checkbox"/>		A11 = 0	<input type="checkbox"/>	
eta2 = 1	<input type="checkbox"/>		eta5 = 1	<input type="checkbox"/>		eta8 = 1	<input type="checkbox"/>		eta11 = 0	<input type="checkbox"/>	
b2 = 14	<input type="checkbox"/>		b5 = 21.1	<input type="checkbox"/>		b8 = 27	<input type="checkbox"/>		b11 = 1	<input type="checkbox"/>	
Tau2 = 0.3	<input type="checkbox"/>		Tau5 = 0.5	<input type="checkbox"/>		Tau8 = 1	<input type="checkbox"/>		Tau11 = 1	<input type="checkbox"/>	
A3 = 1e4	<input type="checkbox"/>		A6 = 1e4	<input type="checkbox"/>		A9 = 2e3	<input type="checkbox"/>		A12 = 0	<input type="checkbox"/>	
eta3 = 1	<input type="checkbox"/>		eta6 = 1	<input type="checkbox"/>		eta9 = 1	<input type="checkbox"/>		eta12 = 0.5	<input type="checkbox"/>	
b3 = 16.9	<input type="checkbox"/>		b6 = 21.8	<input type="checkbox"/>		b9 = 28.5	<input type="checkbox"/>		b12 = 0	<input type="checkbox"/>	
Tau3 = 0.4	<input type="checkbox"/>		Tau6 = 0.5	<input type="checkbox"/>		Tau9 = 1	<input type="checkbox"/>		Tau12 = 1	<input type="checkbox"/>	
k1 = 0	<input type="checkbox"/>		k3 = 0	<input type="checkbox"/>		k5 = 0	<input type="checkbox"/>		k7 = 0	<input type="checkbox"/>	
k2 = 0	<input type="checkbox"/>		k4 = 0	<input type="checkbox"/>		k6 = 0	<input type="checkbox"/>		k8 = 0	<input type="checkbox"/>	

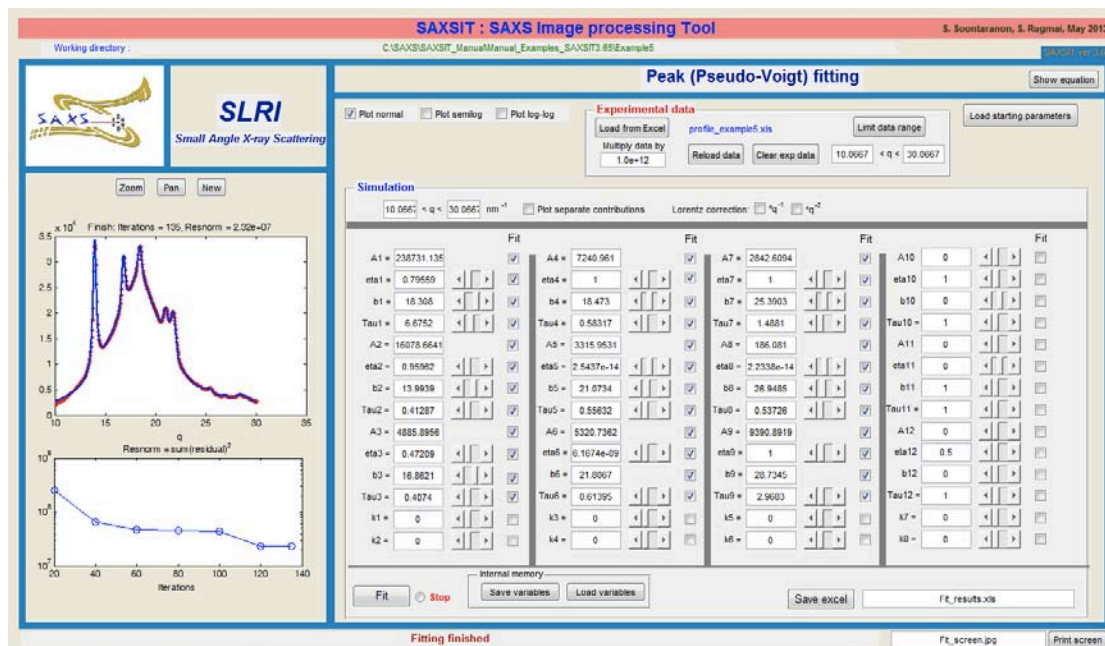
7. We will then start fitting by choosing which parameters for each peak to be fitted. Keep in mind that this is chi-square minimization, the solution can give either good or bad fits. Since we can make good guesses for the peak positions of the crystalline peaks, we then first try fitting all parameters, except the peak positions of the 8 crystalline peaks. Behind all the parameters, there are 'Fit' check boxes. If the box behind the parameter is checked, that parameter will be regarded by the program as a fitting parameter. For the first try, we therefore check the boxes for parameters of all the 9 peaks, except the parameters 'b' of the 8 crystalline peaks (as in the figure below).

Fit			Fit			Fit			Fit		
A1 = 187261.474	<input checked="" type="checkbox"/>		A4 = 50589.724	<input checked="" type="checkbox"/>		A7 = 209.3999	<input checked="" type="checkbox"/>		A10 = 0	<input type="checkbox"/>	
eta1 = 0.48122	<input checked="" type="checkbox"/>		eta4 = 1.6694	<input checked="" type="checkbox"/>		eta7 = 4.4121e-14	<input checked="" type="checkbox"/>		eta10 = 1	<input type="checkbox"/>	
b1 = 18.2823	<input checked="" type="checkbox"/>		b4 = 18.5	<input type="checkbox"/>		b7 = 25	<input type="checkbox"/>		b10 = 0	<input type="checkbox"/>	
Tau1 = 8.1982	<input checked="" type="checkbox"/>		Tau4 = 1.5758	<input checked="" type="checkbox"/>		Tau7 = 1	<input checked="" type="checkbox"/>		Tau10 = 1	<input type="checkbox"/>	
A2 = 14906.4601	<input checked="" type="checkbox"/>		A5 = 0.56241	<input checked="" type="checkbox"/>		A8 = 5744.1759	<input checked="" type="checkbox"/>		A11 = 0	<input type="checkbox"/>	
eta2 = 0.87514	<input checked="" type="checkbox"/>		eta5 = -15574.797	<input checked="" type="checkbox"/>		eta8 = 2.6966	<input checked="" type="checkbox"/>		eta11 = 0	<input type="checkbox"/>	
b2 = 14	<input type="checkbox"/>		b5 = 21.1	<input type="checkbox"/>		b8 = 27	<input type="checkbox"/>		b11 = 1	<input type="checkbox"/>	
Tau2 = 0.4062	<input checked="" type="checkbox"/>		Tau5 = 0.60239	<input checked="" type="checkbox"/>		Tau8 = 1.4991	<input checked="" type="checkbox"/>		Tau11 = 1	<input type="checkbox"/>	
A3 = 5326.8741	<input checked="" type="checkbox"/>		A6 = 6476.4682	<input checked="" type="checkbox"/>		A9 = 0.49764	<input checked="" type="checkbox"/>		A12 = 0	<input type="checkbox"/>	
eta3 = 0.75728	<input checked="" type="checkbox"/>		eta6 = 0.35964	<input checked="" type="checkbox"/>		eta9 = -54591.384	<input checked="" type="checkbox"/>		eta12 = 0.5	<input type="checkbox"/>	
b3 = 16.9	<input type="checkbox"/>		b6 = 21.8	<input type="checkbox"/>		b9 = 28.5	<input type="checkbox"/>		b12 = 0	<input type="checkbox"/>	
Tau3 = 0.40531	<input checked="" type="checkbox"/>		Tau6 = 0.58094	<input checked="" type="checkbox"/>		Tau9 = 5.1597	<input checked="" type="checkbox"/>		Tau12 = 1	<input type="checkbox"/>	
k1 = 0	<input type="checkbox"/>		k3 = 0	<input type="checkbox"/>		k5 = 0	<input type="checkbox"/>		k7 = 0	<input type="checkbox"/>	
k2 = 0	<input type="checkbox"/>		k4 = 0	<input type="checkbox"/>		k6 = 0	<input type="checkbox"/>		k8 = 0	<input type="checkbox"/>	

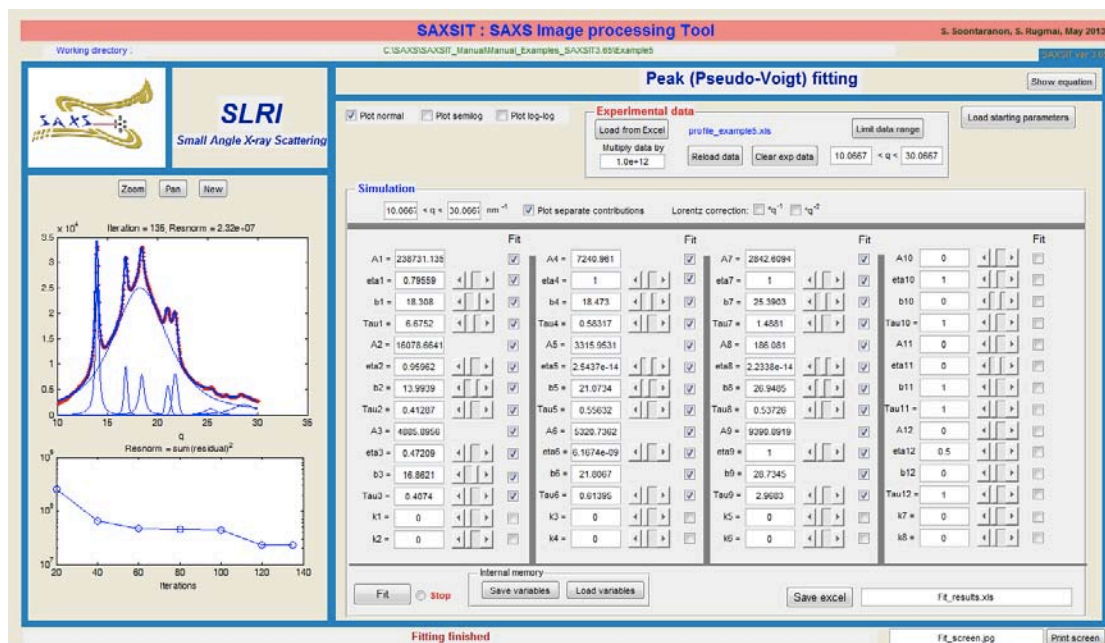
8. Click 'Fit'. After fitting is finished (see the status in the Message line at the bottom of the window) we have to examine the results. The fitting usually gives good looking fit, but may give nonphysical or unreasonable results for some parameters. For example in this case, the fit may give negative eta (must be between 0 and 1) and negative or too small amplitude for small peaks near the end.



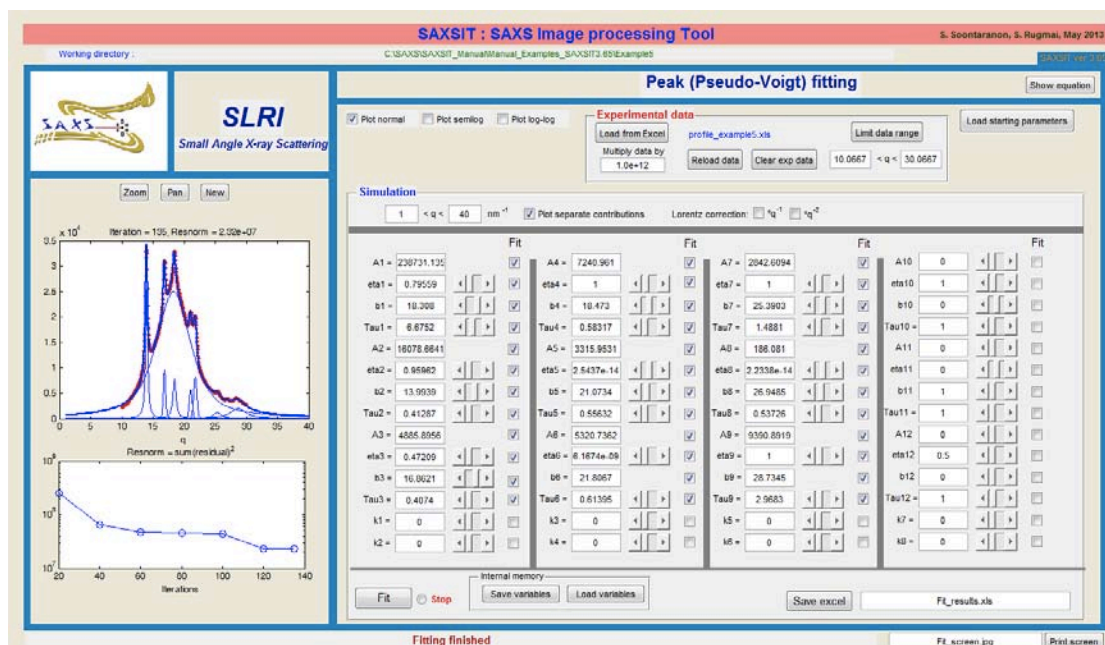
9. Manually correct the unreasonable parameters. For example, you may set negative eta to 1, set too small amplitude to the value close to that of the nearby peak, and set negative or unreasonably large Tau to the value close to that of the nearby peak. This choose more or less restore the shape of the peaks in the data.
10. Check the 'Fit' boxes for all the parameters, including the all the peak positions.
11. Click 'Fit' again. Examine all the obtained parameters. This time they should look fine (if not, repeat the step 9 and fit again).



12. Check the checkbox 'Plot separate contributions' to see each peak in the plot. You should see one broad peak from the amorphous scattering and the 8 narrow peaks from the crystalline scattering.



13. Before saving the results, we have to extend the range of  $2\theta$  of the calculation. This is because when saving, the program will also calculate the areas under each peak. The calculation range should therefore cover the whole peak in order to obtain correct areas. This can be done by just changing the values of calculation range in the boxes (in front and after  $\langle q \rangle$ ) in the 'Simulation' panel. Here we change to  $1 < q < 40$  (keep in mind that  $q$  is actually  $2\theta$ ).



14. Click 'Save excel'. The fit results will be written to the specified Excel file. The output file contains 3 worksheets. 'fit\_parameters' contains parameters from the last fit and the residual norm of the fit. 'fit\_results' contains the data, the calculations ('Sum\_all'), the calculation without

background (Sum\_PV) and the calculations for each peak and each background term. 'Peak\_areas' contains the areas under curve of the calculations (integrated values of the 'fit\_results').

15. The crystallinity can be calculated from the ratio of peak areas between the crystalline peaks and the total peak area (including the amorphous peak). In this case therefore the crystallinity can be calculated from the values in the 'Peak\_areas' worksheet by  $\text{crystallinity (\%)} = 100 \times (\text{Sum\_PV} - \text{PV1}) / \text{Sum\_PV}$

Note that the background terms may be included in the fit if required. The background is parameterized by k1 (constant), k2 (linear), k3 (quadratic), k4 and k5 (power). Clicking 'Show equation' button will display the equations for the fitting.