Reduction of 9IDC USAXS/SAXS data with Nika

Nika version: 1.78

Introduction

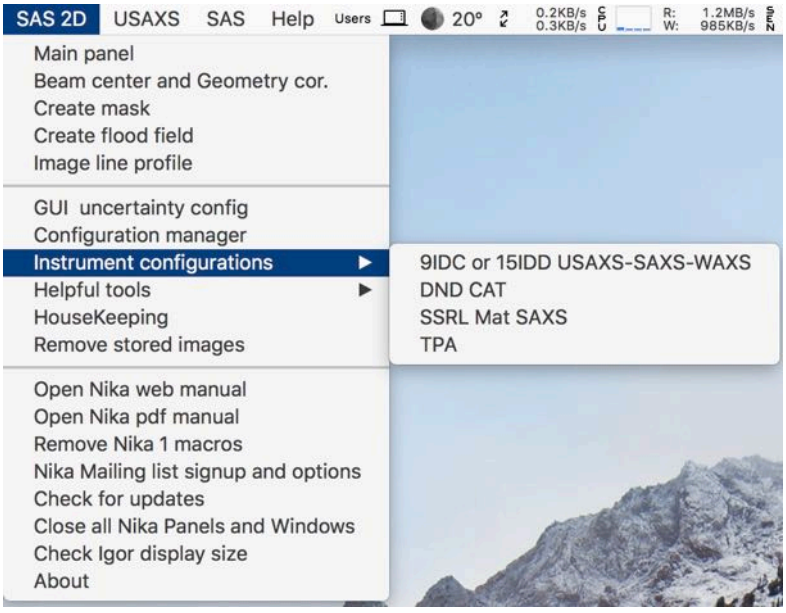
This is step by step procedure for data reduction of SAXS data from 9ID-C combined USAXS/SAXS instrument. This applies to data collected in 2017-03 APS cycle and later. This MAY NOT fully apply to prior cycles, especially before ~2015.

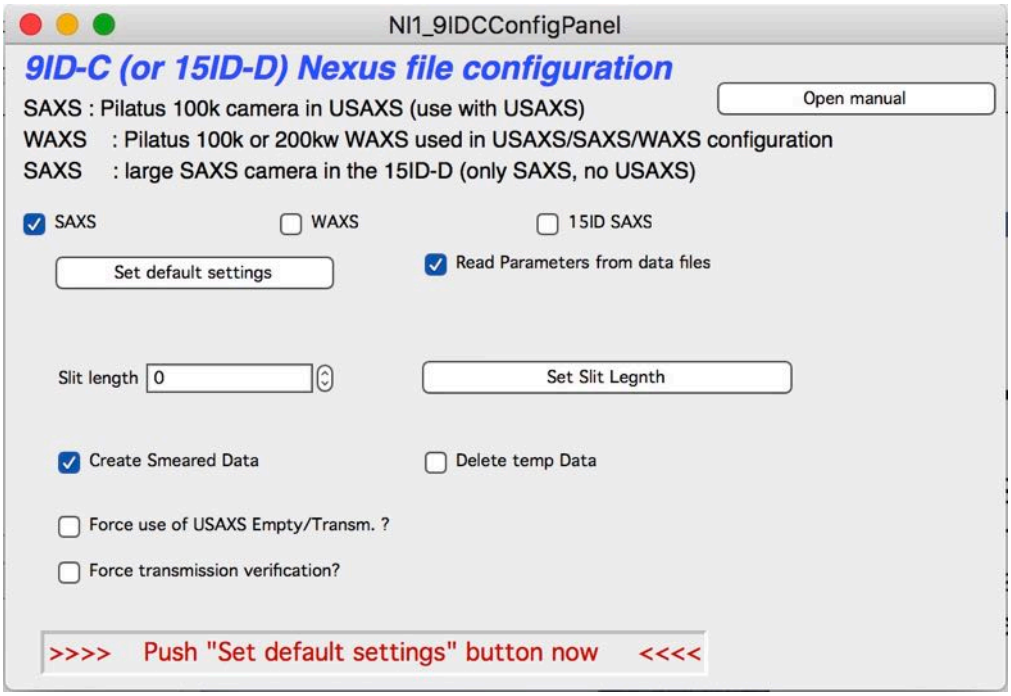
Suggested pre-requisite for slit smeared data

You should reduce USAXS data first, especially if you want to use slit smeared data. This will provide you with slit length for your setup – slit length depends on geometry and wavelength, not sample. Most users have only one slit length, unless their setup changed drastically – that is unless wavelength (X-ray energy) was changed. Usual minor distance changes have no measurable effect.

SAXS data reduction

Select from Nika "SAS 2D" > "Instrument configuration" > "9IDC or 15IDD USAXS-SAXS-WAXS"

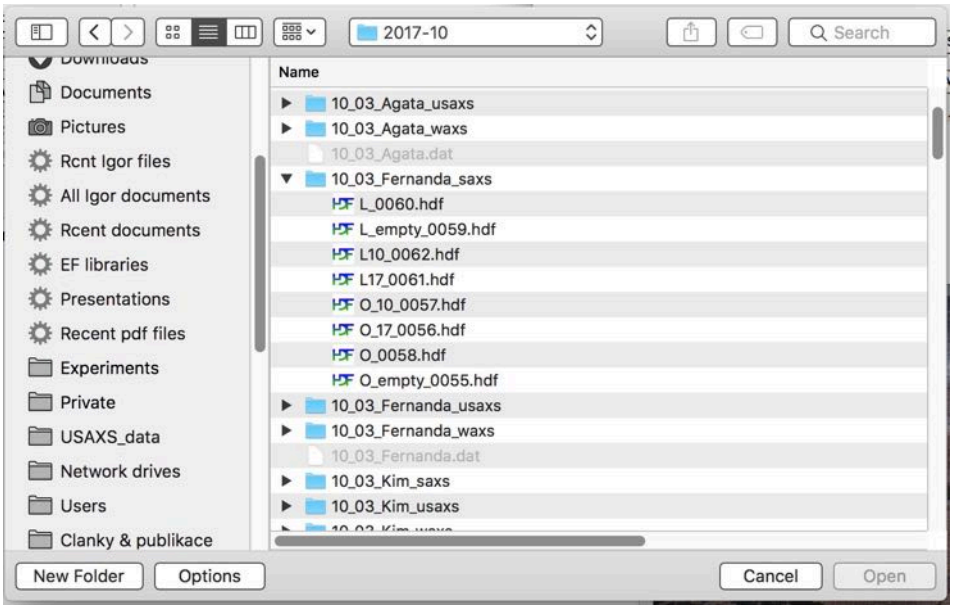




You should be able to open this manual when you push button "Open manual". If this is not available on your computer, reinstall the Nika package and let me know.

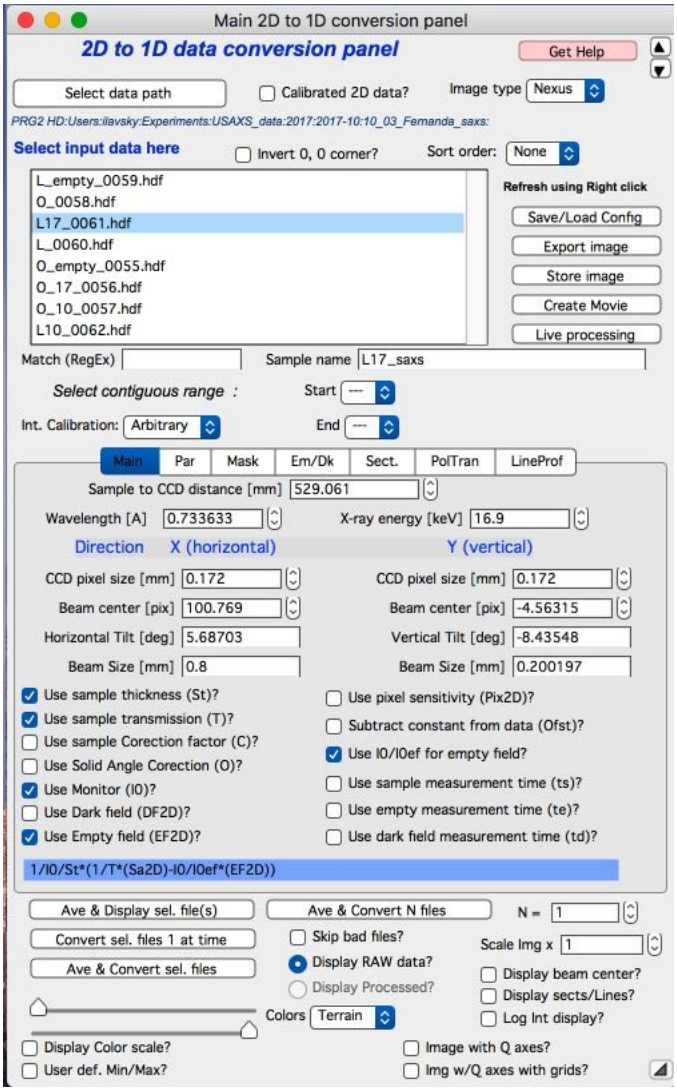
Basic Configuration

Push button "Set default settings" which will open dialog to locate a representative image from the folder of images you want to reduce. For SAXS data this is folder with _saxs at the end, containing number of SampleName.hdf files:



Pick one of images (NOT the Ag Behenate.hdf or LaB6.hdf or combination, that is standard and typically doesn't have the correct parameters in the file).

Selected image is opened and parameters from the file are loaded in Nika – if you have the checkbox "Read Parameters from data files" checked. At this moment the main Nika panel should have correct experiment geometry settings from SAXS (your exact numbers will be different):



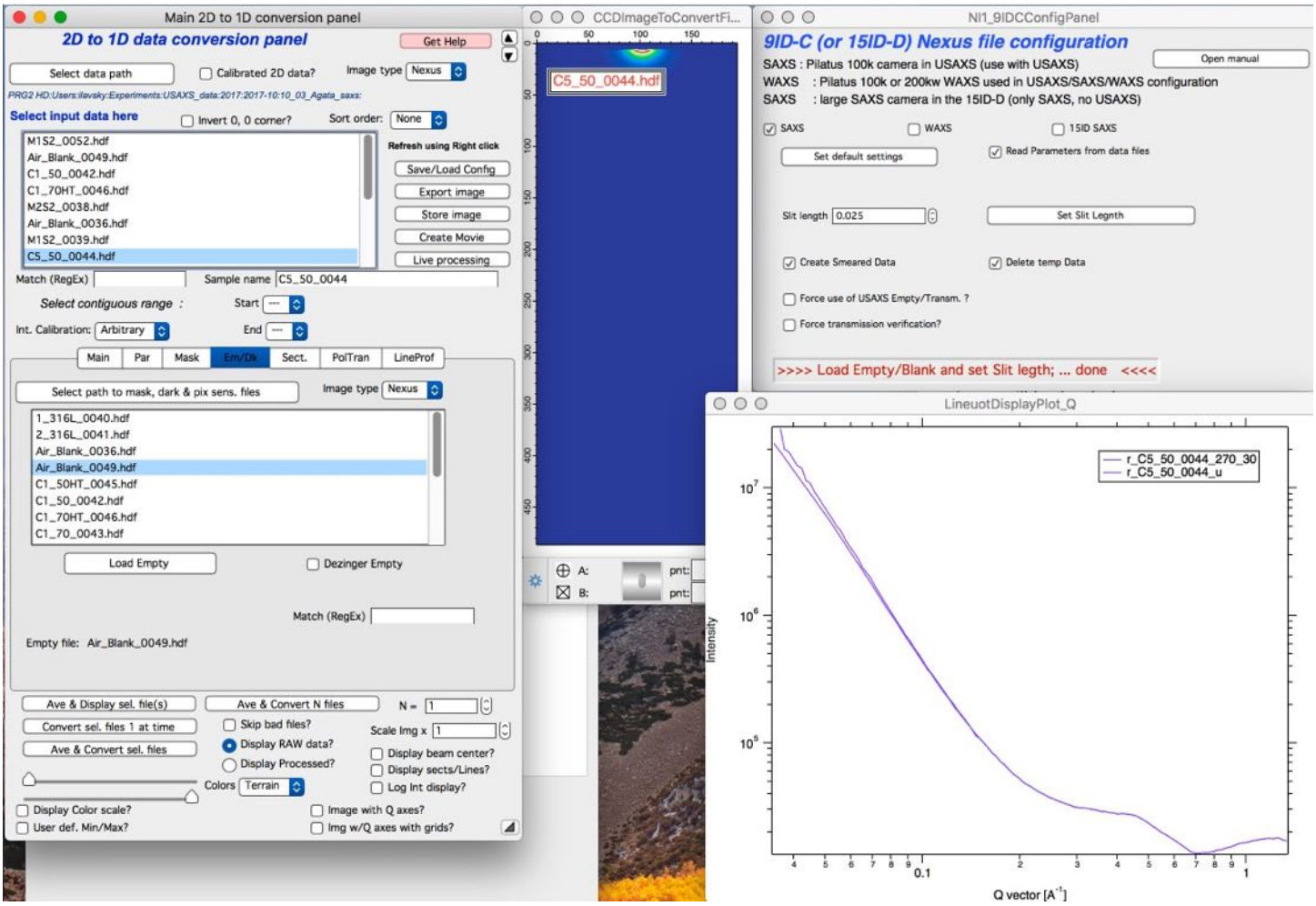
Parameters settings: Default behavior now is to re-read geometry parameters (distance, wavelength,..) for every image again – this is set by having checked "Read Parameters from data files" checkbox in the "NI1_9IDCConfigPanel". If this is not desirable behavior (wrong data recorded with the image), you can uncheck this checkbox and modify the parameters manually or using "Beam center and geometry calibration" tool. With the "Read Parameters from data files" checkbox unchecked, parameters are not changed when new files are loaded, you need to change them manually if necessary.

Note on mask: Images need to be masked. Default mask has been created in the above step – this mask masks off the top few lines on detector which are blocked by tungsten beam stop. If you need to mask more points, you need to do it NOW. Use Mask tool and create mask you want at this point. Keep in mind, that anytime you push the button "Set default settings" the code creates default mask again. You will need to reload your modified mask, if you run through this code again. Usually it is not needed to run through this configuration more than once, except if you want to change to different folder with SAXS data. In that case a new mask is likely necessary anyway?

Slit smeared data: If you are using Slit smeared data in USAXS (you did not desmear the data either during USAXS data reduction or after processing in Desmearing tool) keep checked the checkbox "Create Smeared Data" and either manually insert slit length or (if you already have USAXS data reduced) push button "Set slit length" and locate suitable USAXS data set (typically it does not matter which one, unless geometry changed drastically during your experiment).

Empty/Dark: You need to load Empty/Dark image field now. Select proper file and double click it. Or hit "Load Empty" button. Code has selected the right tab for you to select proper Blank/Empty. Keep in mind you may need to change these background data, depending on how you collected the data.

Now select one or more image files (.hdf) in the Listbox of Nika's main window and use button "Convert sel. files 1 at time" on main panel to process it. Resulting graph is below for reference. Note, that the data can be easiest accessed in Irena by checking "QRS" name system and they are located in folders in root:SAXS:



Typical names are SampleName_Something for folders. "_Something" is either "_u" or "_270_30" for SAXS data on our instrument.

If you have "Create Smeared data" checkbox checked, two data sets will be created:

SampleName_u	this is fake slit smeared data, to be merged with slit smeared USAXS data
Samplename_270_30	this is pinhole collimated data, to be merged with desmeared USAXS data

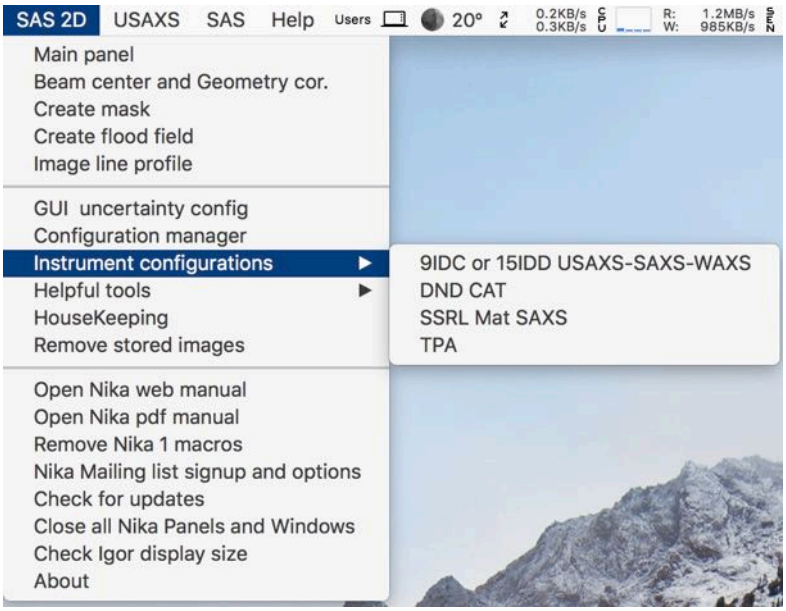
If you uncheck the "Create Smeared data" checkbox, only the _270_30 data are created. This is OK if you have desmeared the USAXS data already.

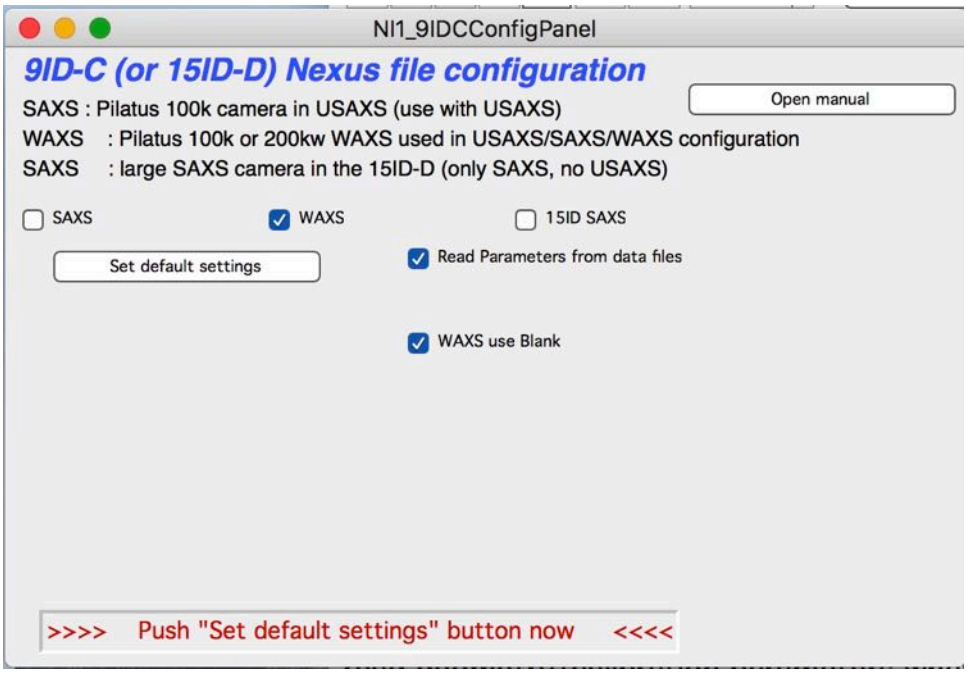
There is no need to generate desmeared data from USAXS (and SAXS) **IF YOU WILL BE USING IRENA** for data analysis.

For any other tool you MUST desmear the data and only after that export them (merged with SAXS preferably). No other common tool I know can *reliably* use our Slit smeared data correctly at this time.

WAXS data reduction

Select from Nika “SAS 2D” > “Instrument configuration” > “9IDC or 15IDD USAXS-SAXS-WAXS”

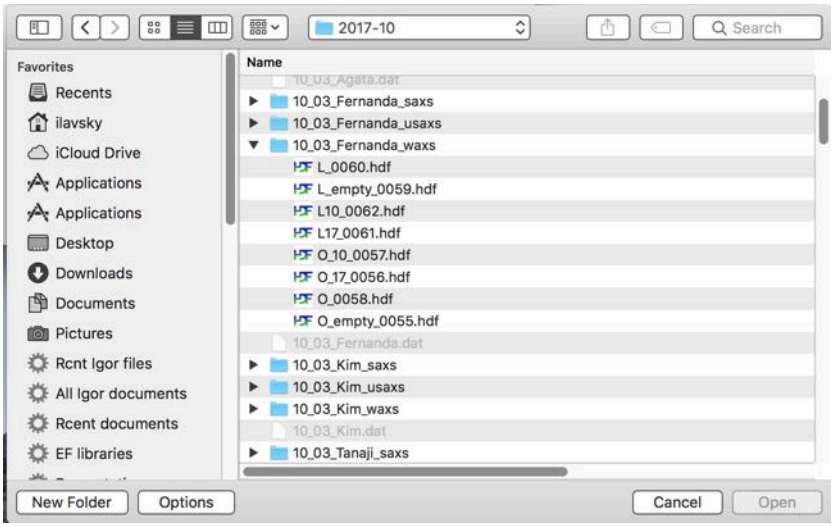




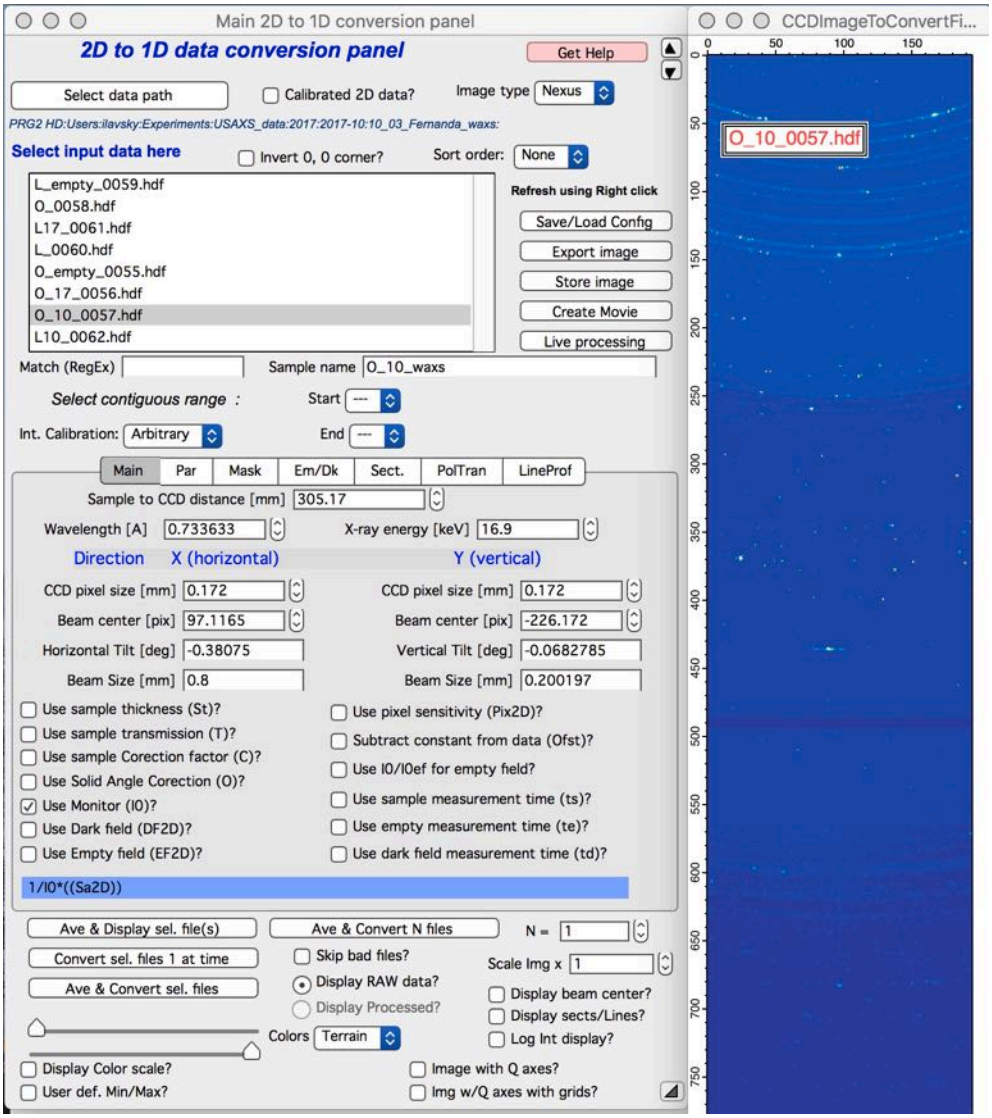
You should be able to open this manual when you push button “Open manual”. If this is not available on your computer, reinstall the Nika package and let me know.

Select WAXS checkbox and keep checked “Read Parameters from data files” checkbox.

Push button “Set default settings” and locate a representative data image (hdf file) in folder which ends with _waxs:



Select a representative image (not standard LaB6 or other). Image is loaded and parameters are read in Nika (your values will vary):



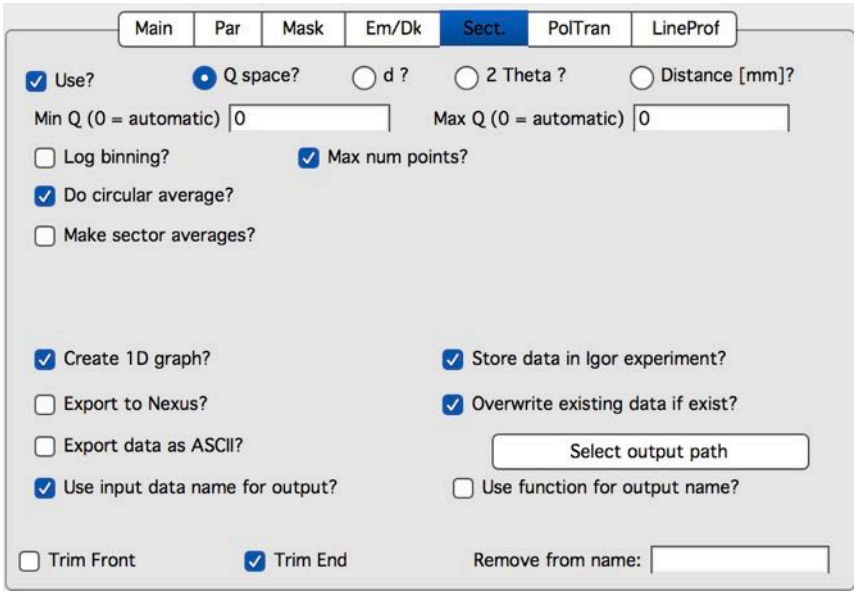
Parameters settings: Default behavior is to re-read geometry parameters (distance, Wavelength,..) for **every** image – this is set by having checked "Read Parameters from data files" checkbox in the "NI1_9IDCConfigPanel". If this is not desirable behavior (wrong data recorded in the image), you can uncheck this checkbox and modify the parameters manually or using "Beam center and geometry calibration" tool. With the "Read Parameters from data files" checkbox unchecked, parameters are not changed automatically, you need to change them if necessary.

Note on mask: Images need to be masked. Default mask has been created in the above step – this mask masks off the middle few lines on detector which are between the two chips in this detector. If you need to mask more points, you need to do it NOW. Use Mask tool and create mask you want at this point. Keep in mind, that anytime you push the button "Set default settings" the code creates default mask again. You will need to reload your modified mask, if you run through this code again. Usually it is not needed to run through this configuration more than once, except if you want to change to different folder with WAXS data. In that case a new mask is likely necessary anyway?

Empty/Dark: You need to decide if you want to subtract the air scattering background NOW. If you keep checkbox "WAXS use Blank" checked, you now need to load Empty/Dark image field. Select proper file and double click it. Or hit "Load Empty" button. Code has selected the right tab for you to select proper Blank/Empty. Keep in mind you may need to change these background data, depending on how you collected the data.

If you choose NOT to subtract the background, you are likely going to have higher background to your data. No problem for highly crystalline samples which scatter well, but if you have amorphous and poorly scattering samples, background subtraction is highly recommended.

Important: Select which x-axis you want to use – default is to produce data as function of q, but you may want d or even Two-theta. This is changed on "Sect" tab:



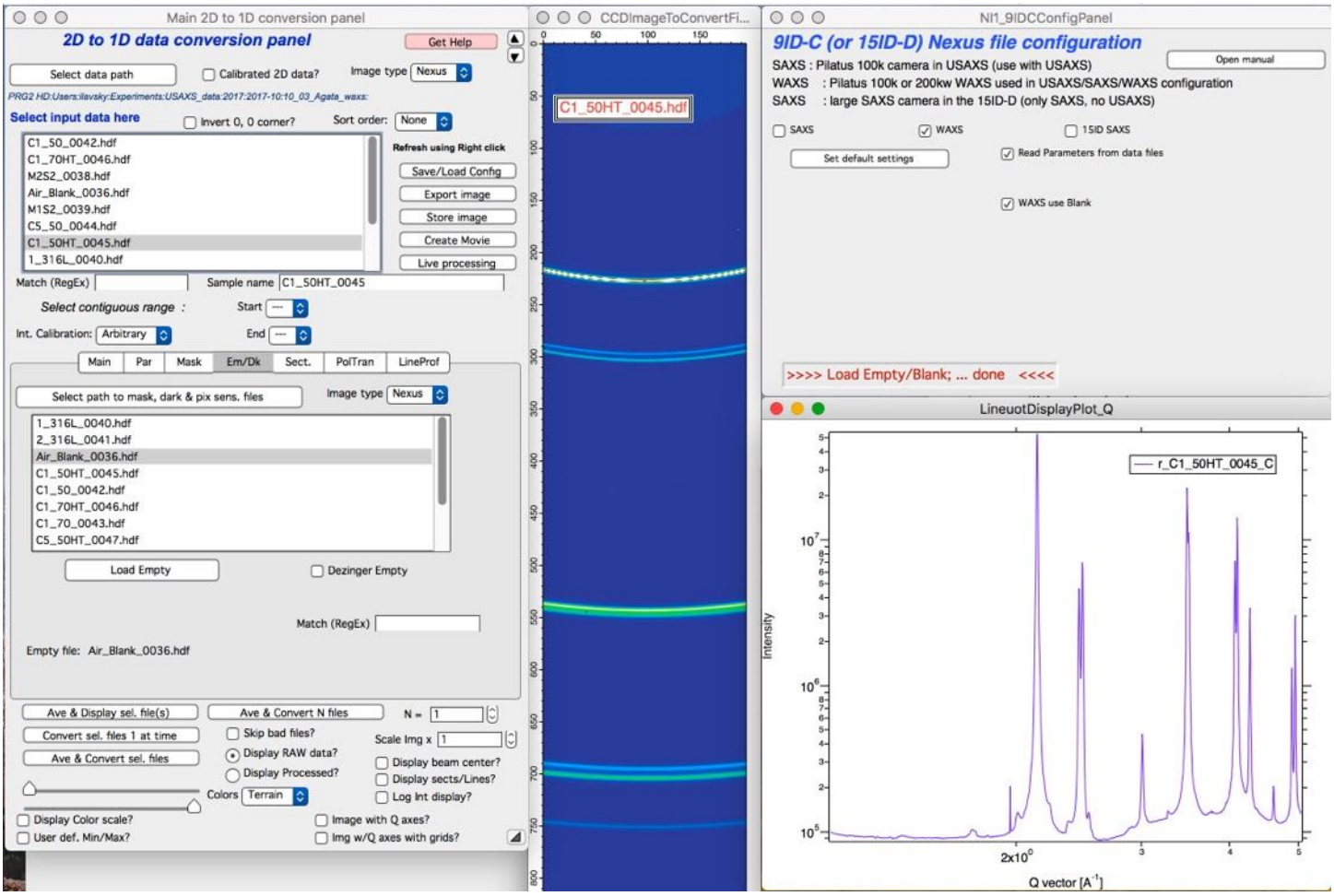
Note: Irena tools do not care – Irena WAXS tool will convert data from Q, d, or Two theta to Two Theta and fit data as Intensity vs Two-theta. This is done automatically using proper wavelength. For other tools, figure out ahead what you need…

Select image in the main window Listbox and push button "Convert sel. Files 1 at time" and data will be processed. Keep in mind that you may need to select correct Blank for each sample – sometimes that is

easy (same one for all), but if it changes, you need to keep track of it and change them as needed. Blanks/empty are changed on "Em/Dk" tab.

Resulting graph is below for reference. Note, that the data can be easiest accessed in Irena by checking "QRS" name system and they are located in folders in root:WAXS:

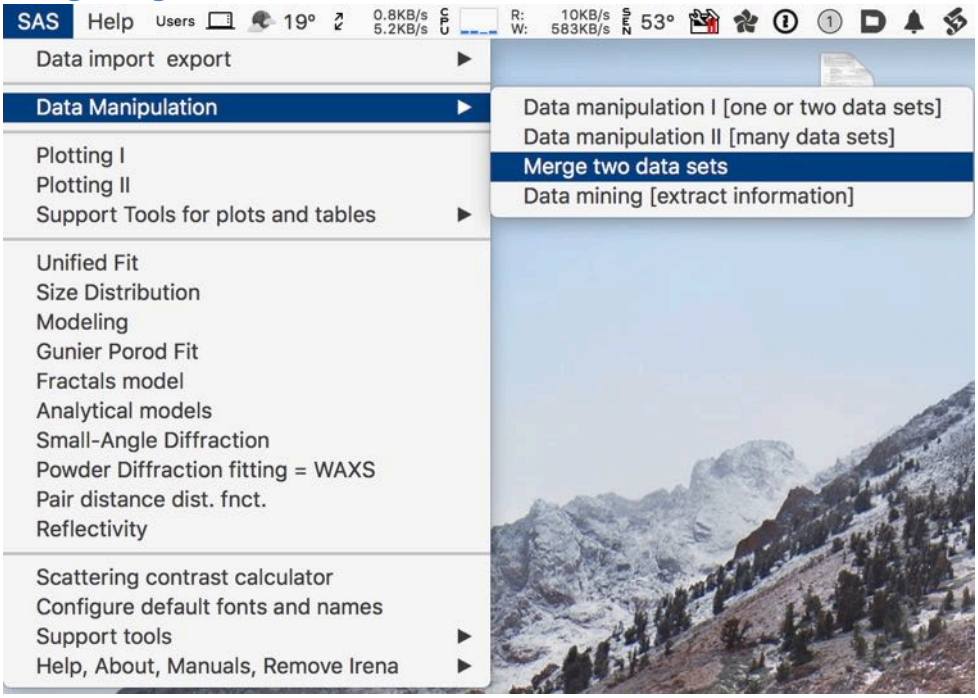
Typical names are SampleName_C for folders.



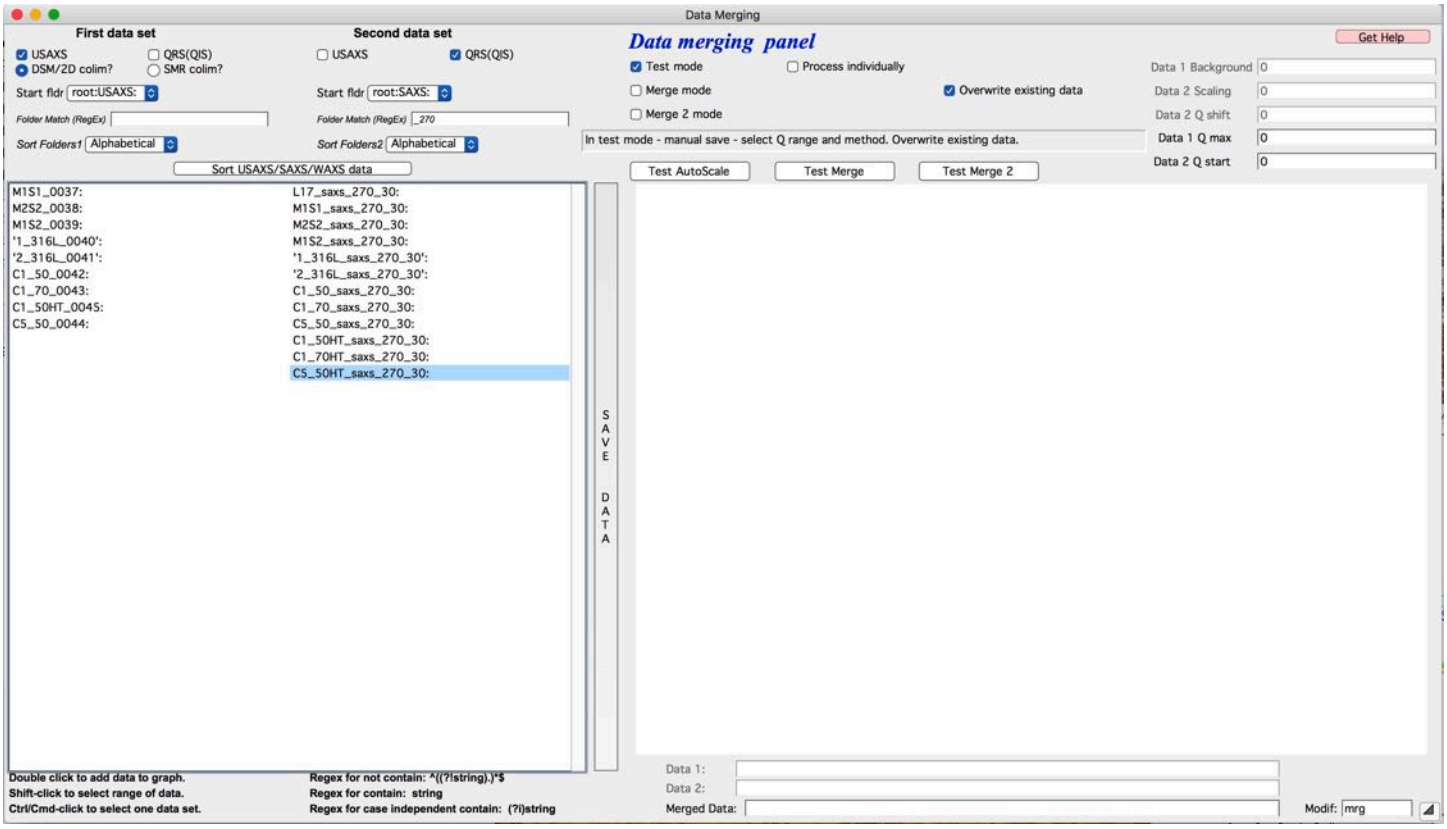
Merging the data together

To merge the data together we can use the _u data from SAXS and SMR data from USAXS, or desmeared data – _270_30 data for SAXS and DSM data for USAXS.

Using Merge two data sets tool

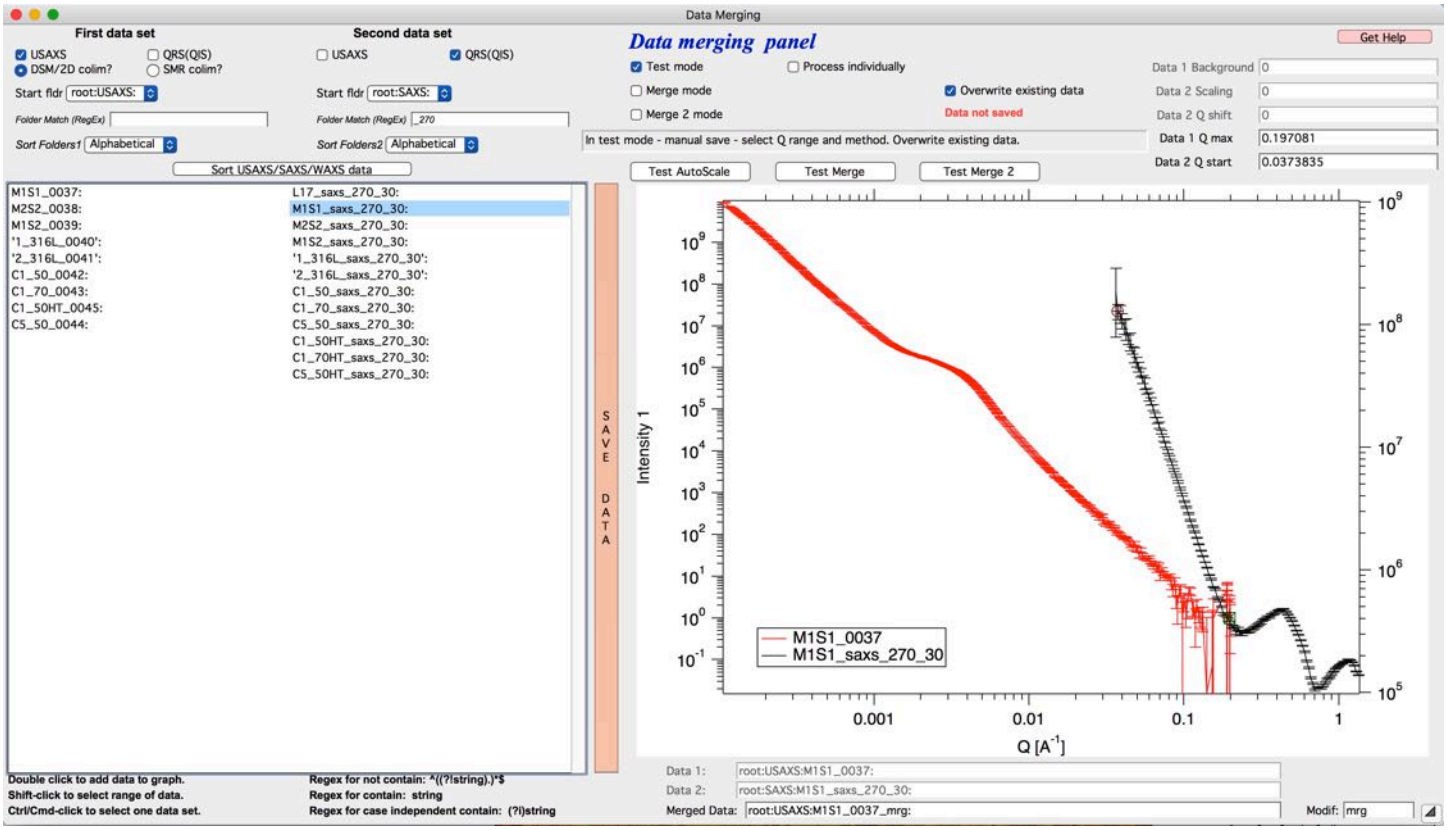


Here is the tool set for desmeared data:



Note the selections at the top left corner. USAXS data for first data set, DSM/2D Colim for desmeared data. If you change between SMR colim? And DSm.2D colim? radio buttons, rtoll will fill match string for Second data set to display only appropriate data files.

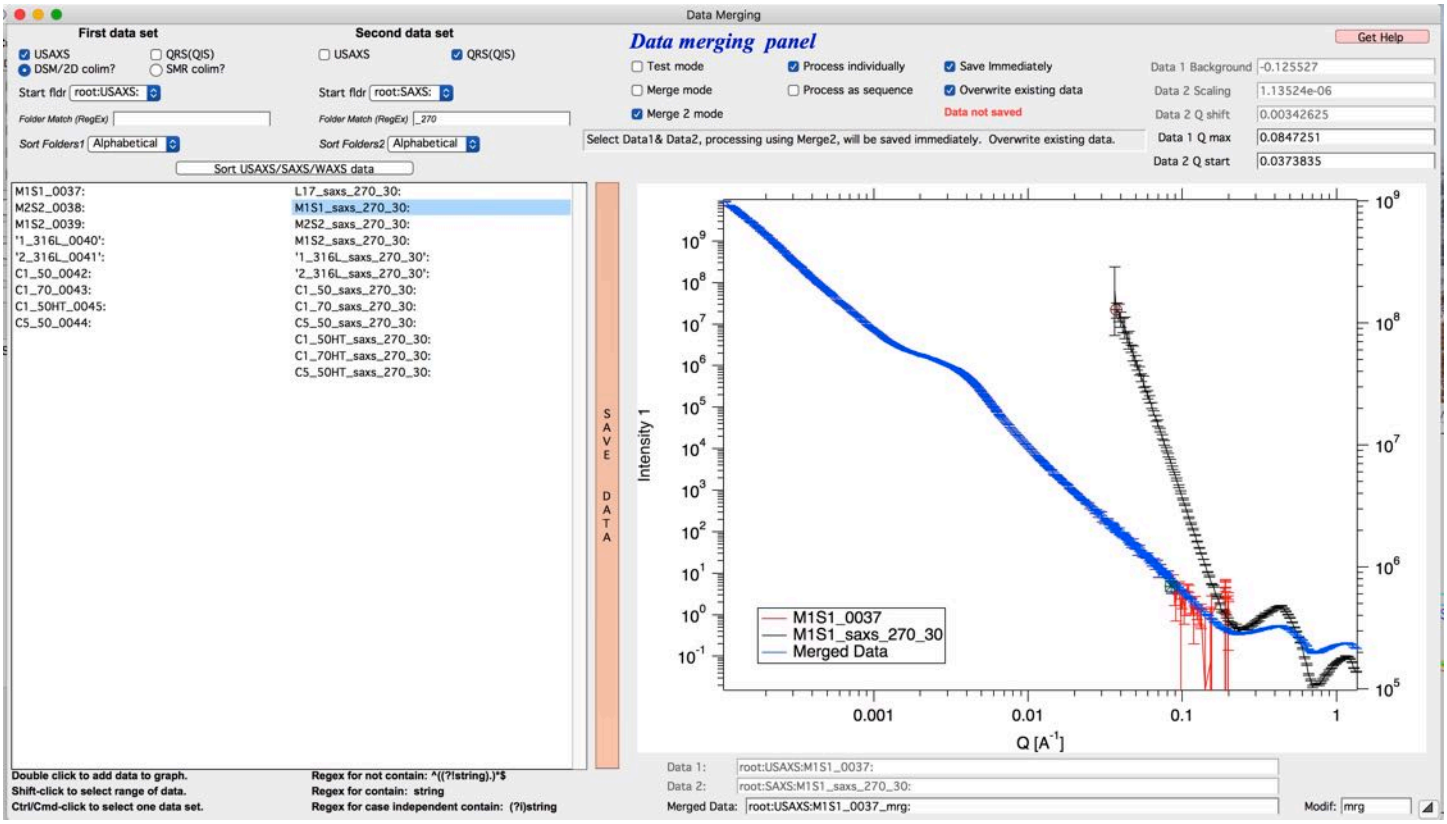
The tool comes in "test mode" (right hand side checkboxes). Double click on one data set in first column. Then on same name (well, similar name) in right column.



Now set cursors so the select range in which the data should overlap – typically the rounded (start of data set 2) is c orrect, unless there are some bad points at the beginning. But the square one ("B") needs to be moved to be usually higher above the flat background. Typically around 0.1 or slightly below… Then try "Test Merge" and "Test Merge 2" buttons. See which produces reasonable results. Test method optimizes two parameters (background for Data 1 and Scaling for Data 2) and is preferable. Test 2 optimizes three parameters – it adds also q shift for Data 2. Less optimal, but sometimes necessary…

I decided on test 2. Once you decide on cursor position and method, check the checkboxes above to proper method and how you want to process the data. You can process many data sets by selecting range of data in both columns and processing many data sets at once – but you have less control… Check the movie on YouTube for more explanation.

Here is usually suitable solution for limited number of files to merge:

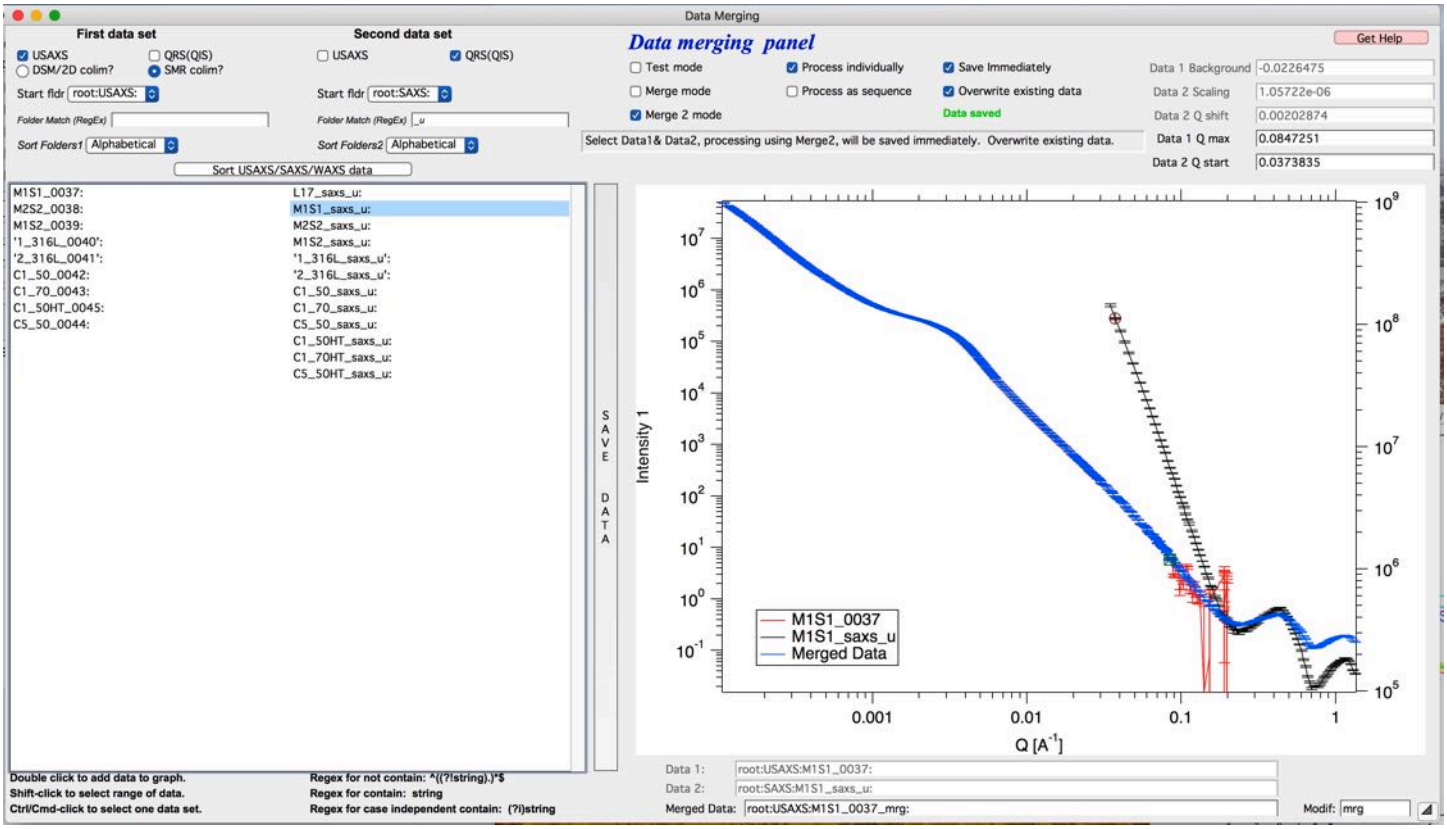


In this case you will dfouble click on left column, First data set will be added, then double click on matching second column, Second data set will be added. Then code will, on its own, optimize using Merge 2 method and save immediately new data set, which is combination of the two data sets.

These data sets are processed and new one created:

Note, the new data set is still USAXS data, new folder will be created and string in “Modif:” field added with “_” in front of it. Default is _mrg, but you can change it to anything reasonable and short…

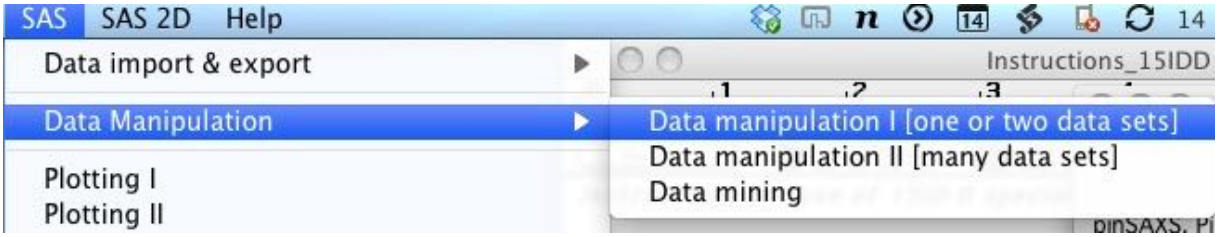
Herte is the tool set to process Slit smeared (SMR data):



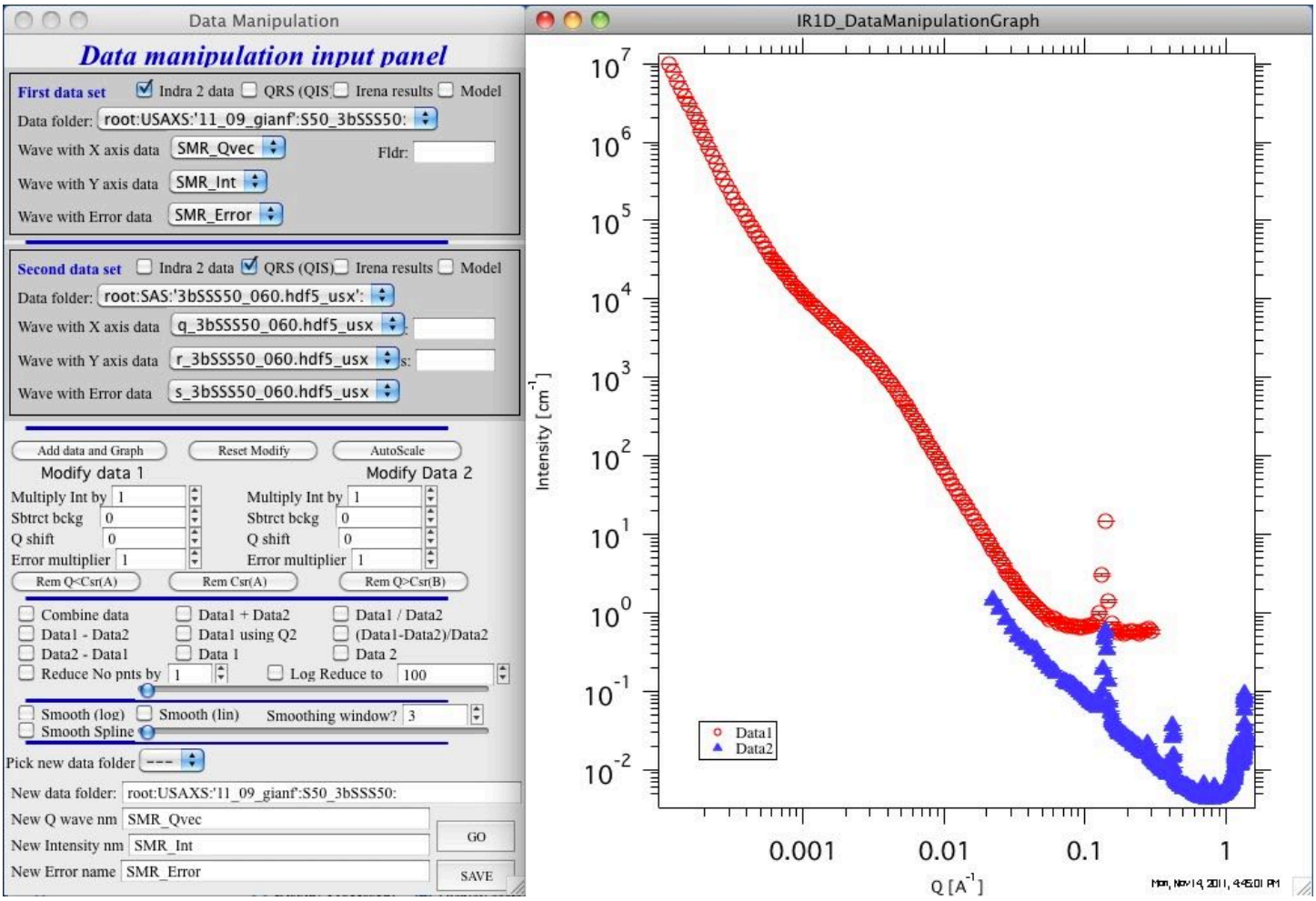
As you can see, there is really no difference in settings.

Using Data Manipulation I tool

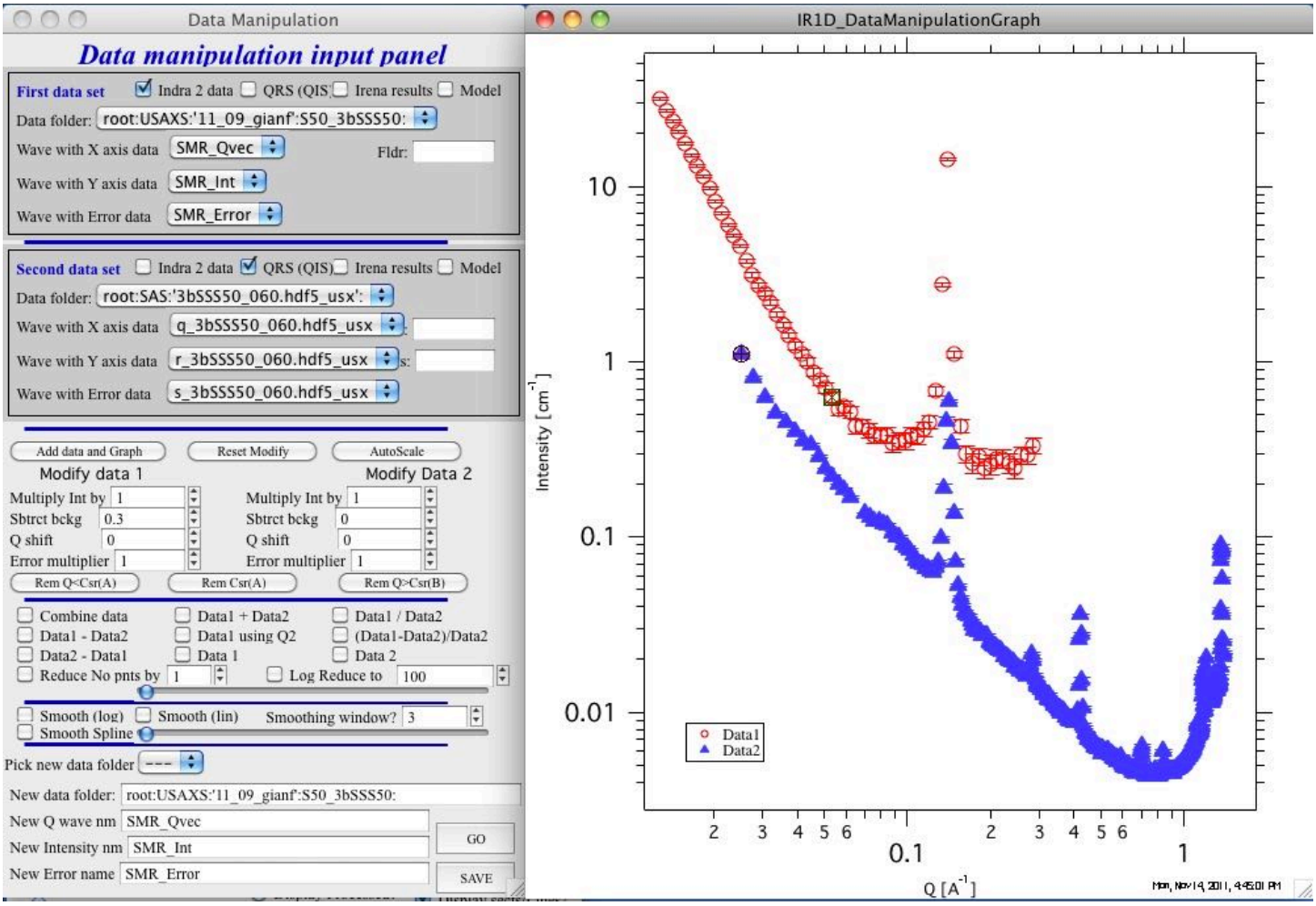
If automatic method in the Merge two data sets tool fails, we can use the "Data manipulation I" from Irena. It has lot more controls and one can merge data sets which fail to merge automatically…



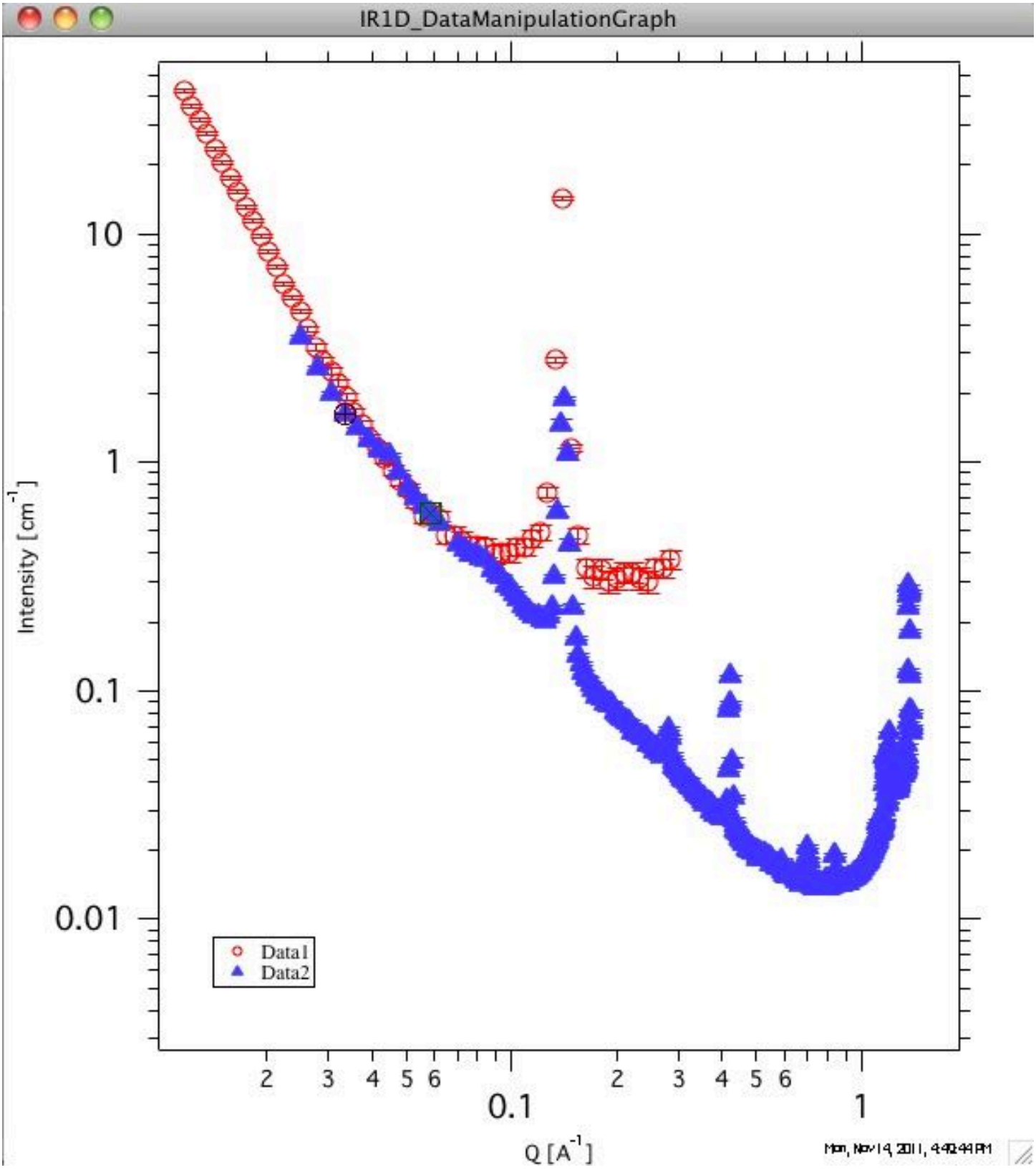
Select "Indra 2 data" for First data set and "QRS" for second data set, select the same sample measured and "Add data and Graph" button.



Zoom to range where data overlap and subtract background from data set 1 (Modify data 1 area) to match the slope of the curves in the overlap region:



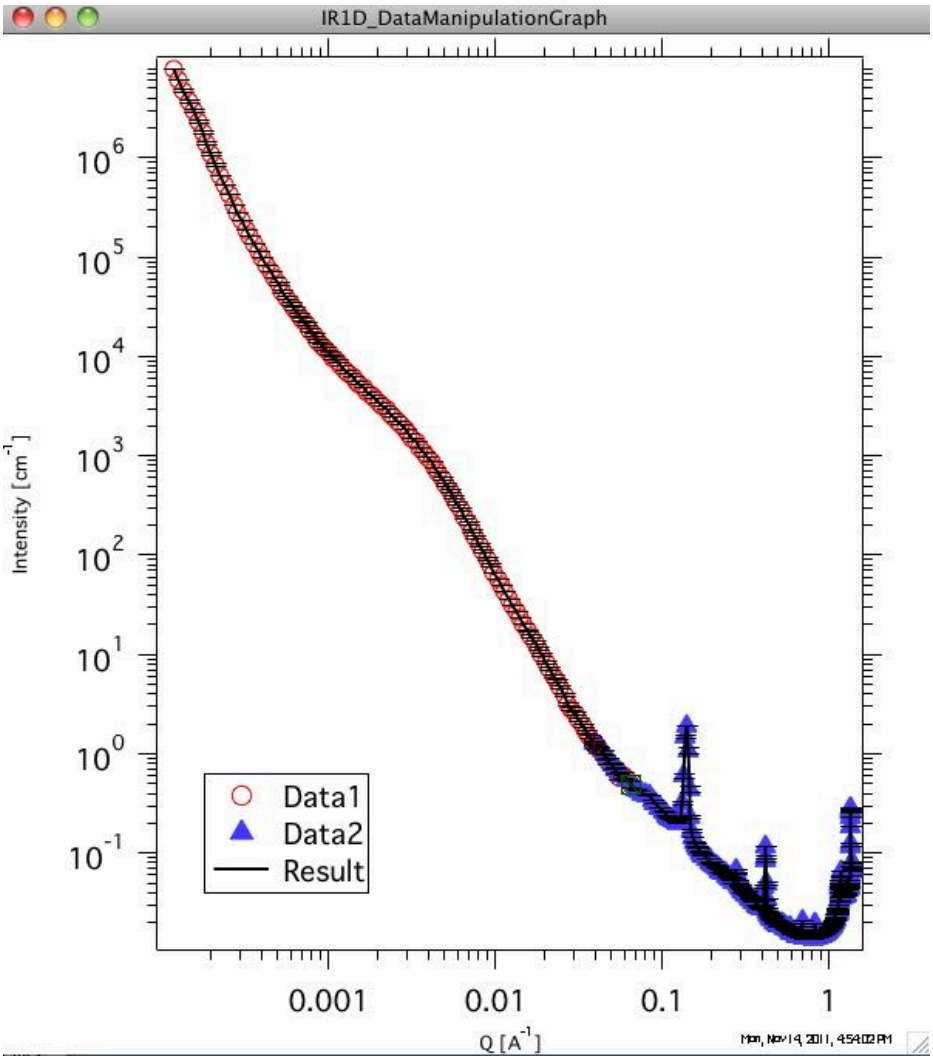
Do not over subtract or the slopes will not match. Select with cursors are where the data overlap well and use “AutoScale” button to scale the data together:



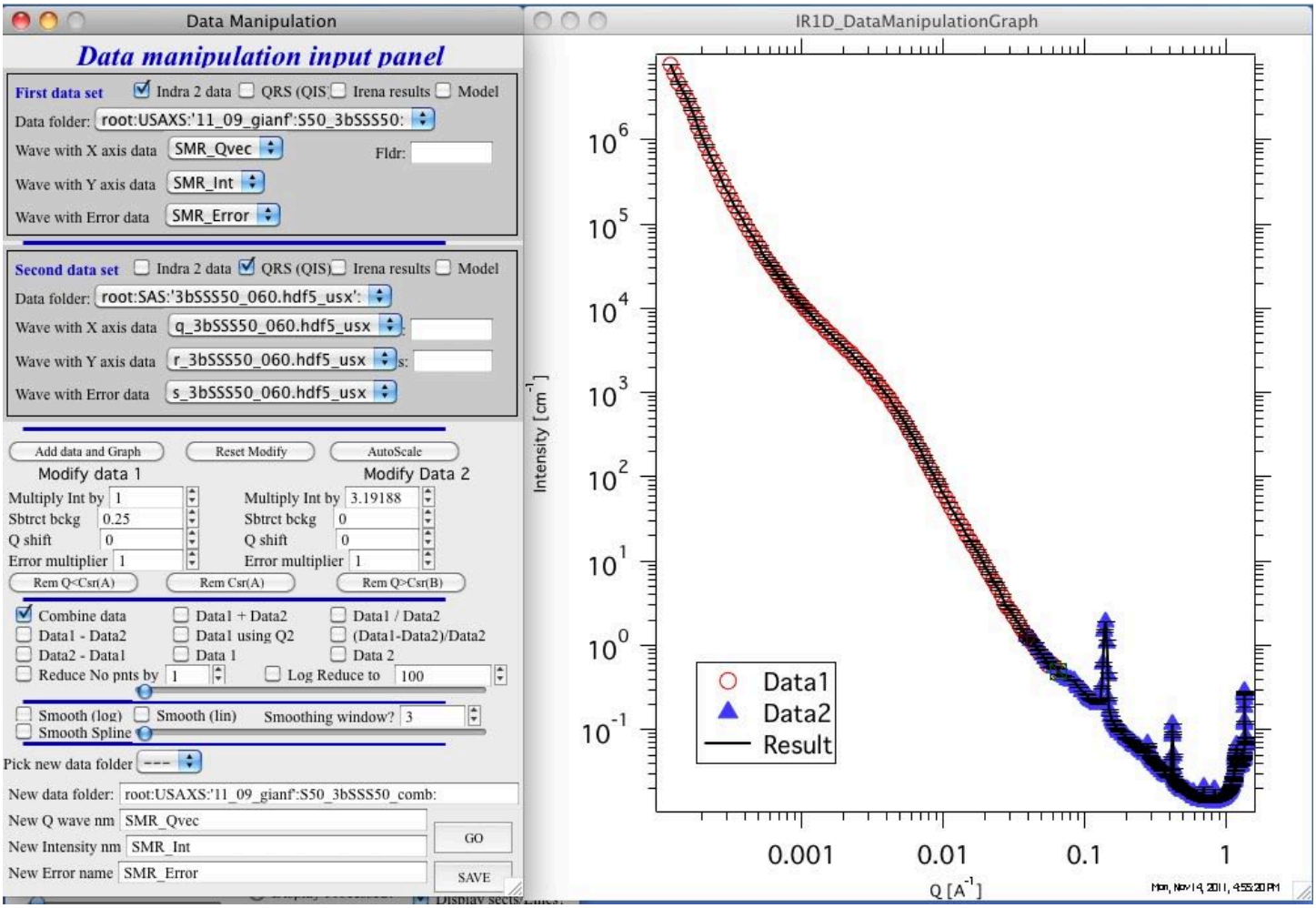
Now you should remove the pinSAXS data for first few points (3-5 points typically) where there are edge effects and other artifacts in pinSAXS data. Use rounded cursor and "Rem Q<CSR(A)" button. Remove

USAXS data at high Q where the noise or background are too large using rectangular cursor and "Rem Q>Csr(B)" button. Make sure the cursors are on the right data set.

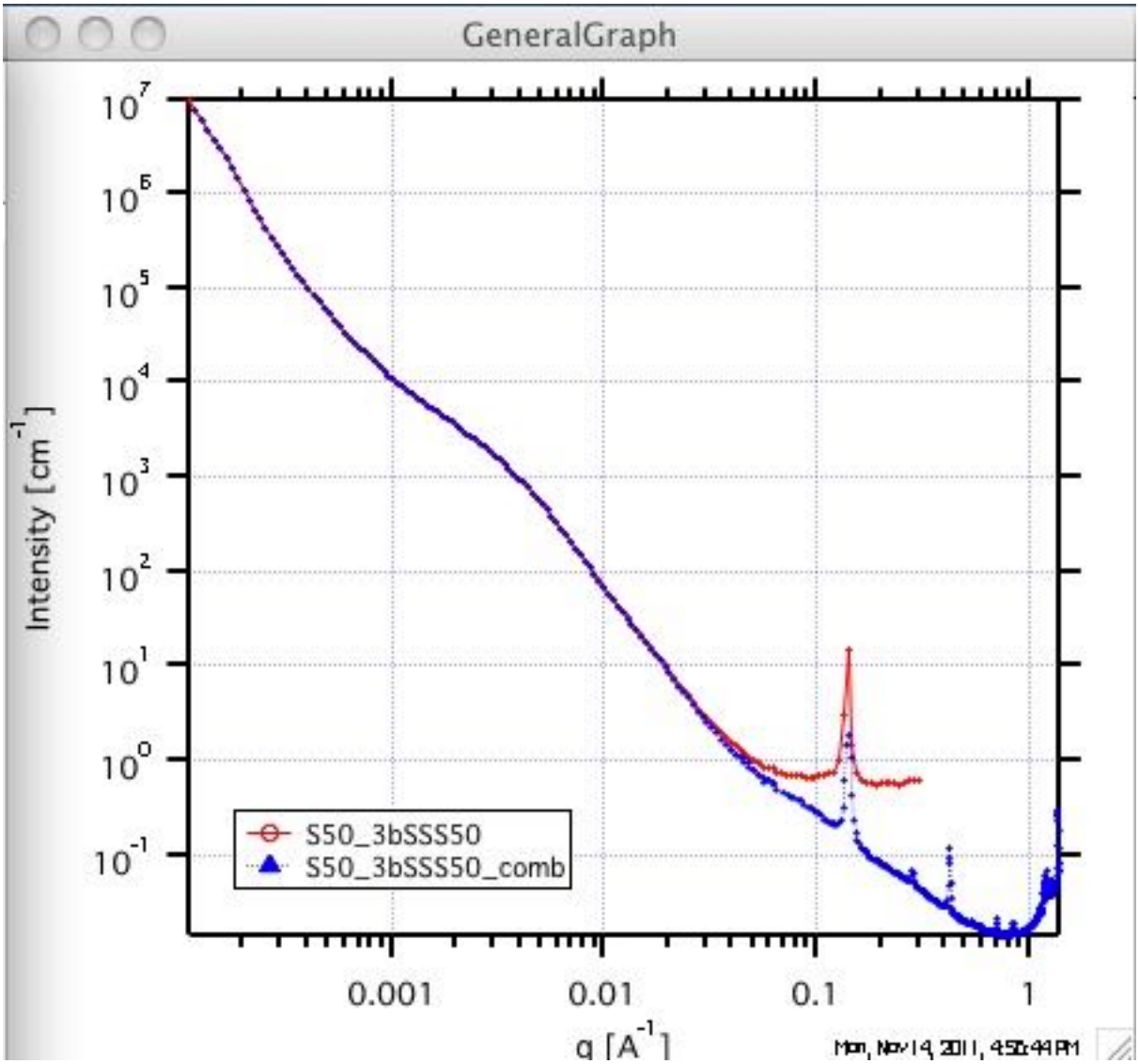
Then select "Combine data" as processing method and hit button "Go".



When happy with the result modify the name of the "New data folder" (may be add behind the name: "_comb") and save the data.



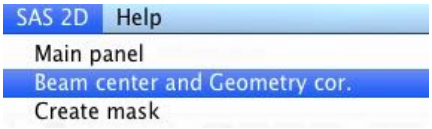
New data were created. These data have absolute calibration from USAXS instrument. They also are still slit-smeared with the original slit length and can be desmeared or modeled by Irena as slit smeared:


GeneralGraph
Intensity [cm^{-1}]
10^{7}
10^{6}
10^{5}
10^{4}
10^{3}
10^{2}
10^{1}
10^{0}
10^{-1}
S50_3bSSS50
S50_3bSSS50_comb
0.001
0.01
0.1
1
q [A^{-1}]
Mon, Nov 14, 2011, 4:52:44 PM

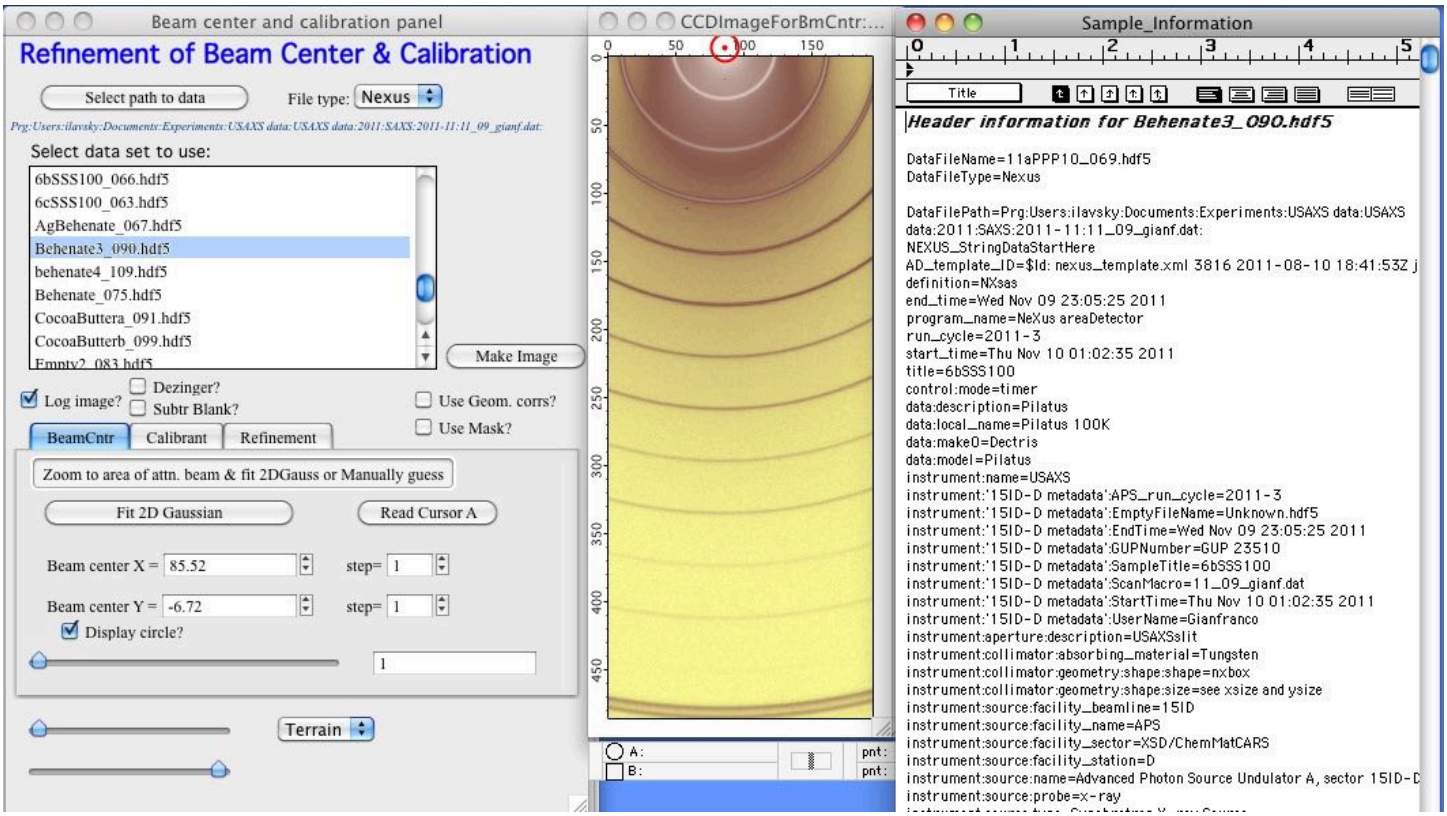
Beam center/distance fine tuning

Stored beam center and distance between sample and area detector are generally good enough, but may not be perfect. We need to refine them next using measurement of Ag Behenate. Note, that you may have more than one Ag Behenate measurement if you had multiple "batches" of samples collected. These are likely close together, so you may choose to use only one setting for all samples, but correct procedure is to optimize Beam center/distance for every batch independently.

Select "Beam center and Geometry cor." From "SAS 2D" menu.

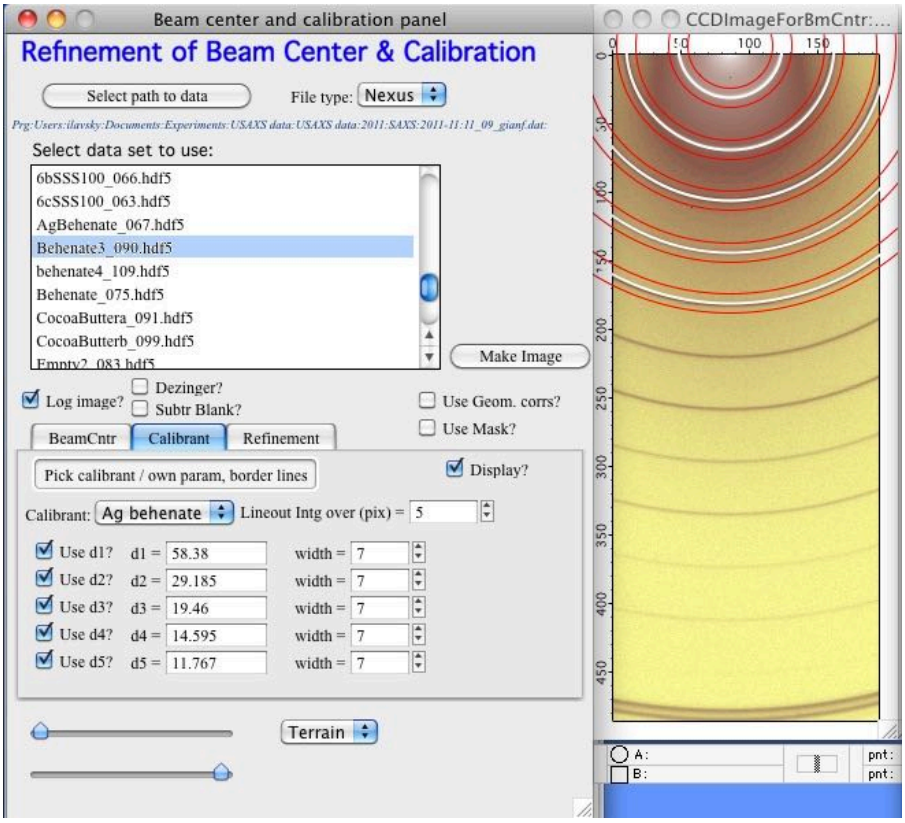


Find appropriate Ag Behenate data set and hit "Make Image", select "Log Image?" checkbox in the middle left of the panel:



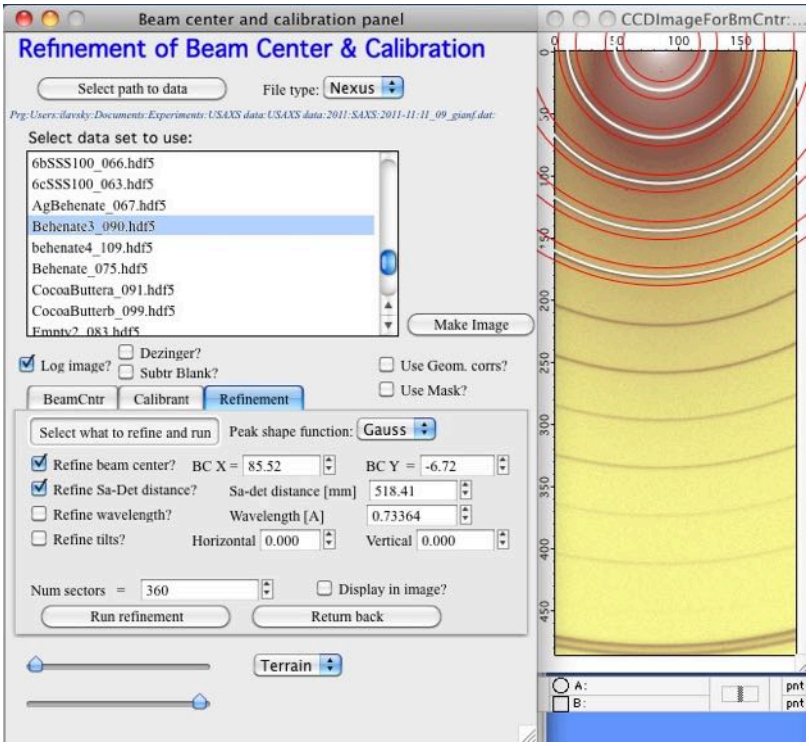
Note the red dot and circle at the top is position of the beam center stored in the metadata by beamline scientist.

Select tab "Calibrant" and you should see:

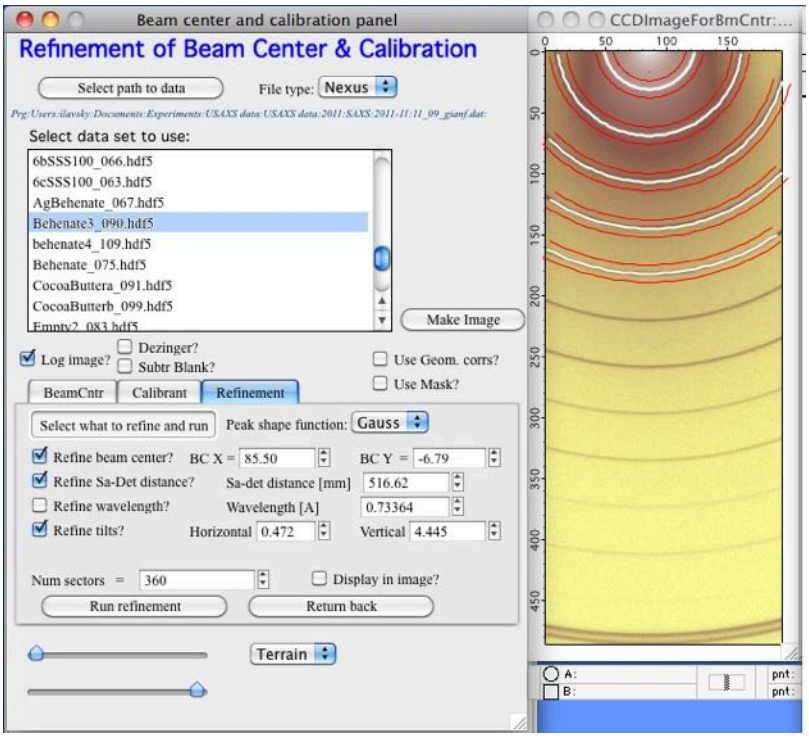


This shows that Ag Behenate is predefined as calibrant. The grey lines of Ag Behenate should be between the two red lines around the white line. The white line is estimate of position of the diffraction lines using the distance and beam center from the metadata, two red lines indicate how wide will Nika search for the peak positions. This should be more or less correct. If higher orders are too weak, you may choose to uncheck higher orders of the diffractions here (d5, d4 etc.).

Select tab “Refinement” and select “Refine beam center” and “Refine Sa-Det distance”. Run first the refinement (“Run refinement” button) with “Gauss” selected as “Peak shape function”. If the fitting to peak profiles starts failing, select “Gausswithslopedback”. Gauss should be more stable, but when background is significant, the “Gausswithslopedback” may be necessary.



After fitting, history are will contain message with achieved chi-squared. Now select checkbox “Refine tilts” and run again. The chi-squared should improve somehow, there are likely tilts (about 0.5 deg horizontal, 4.5 deg vertical in my case):



This is done… You now have the best geometry parameters you can have for the batch of data the AG Behenate file is associated with. Close the panel, Sample_Information notebook, and image.