2-D data reduction using Nika package, part I

Basic Nika setup

This handout describes how to reduce 2D SAXS (pinhole) data using Nika package. In this case we use data collected on 15ID beamline during February 2010. Note, that the data reduction varies from instrument to instrument, this is just one of methods how to reduce the data.

This handout has number of parts. This is part I – basic setup and calibration. Part II is data reduction itself.

Experiment description (aka: what you need to know):

We used MarCCD detector. This is detector, which produces, in 2x2 binning, 1k x 1k images (with circular active area, see below). The pixel size is (after 2x2 binning) 0.158mm x 0.158mm. Image file format is Tiff.

We used 12keV X-ray radiation (1.0332 A).

Our detector was approximately 570mm from the sample area.

We collected following frames to provide calibration and data corrections:

Dark image at necessary times

Empty beam shot at necessary times

Ag Behenate measurement (distance/beam center calibration)

Glassy Carbon measurement (intensity calibration). Note: transmission of Glassy Carbon is: 0.818 at this energy.

We measured transmissions for samples, provided below:

Sa1 was measured as 0.943

Sa2 was measured as 0.979

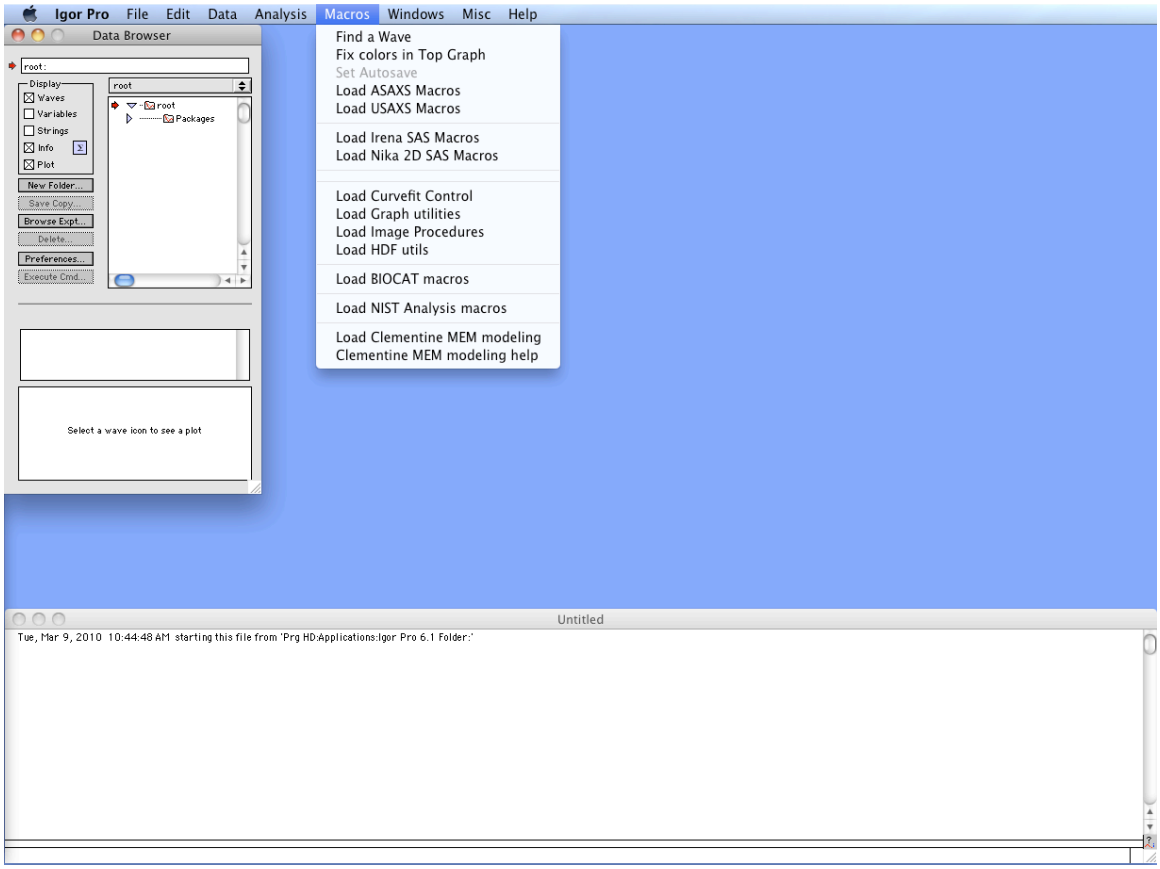
Sa3 was measured as 0.960

Sa4 was measured as 0.968

Basic Nika setup

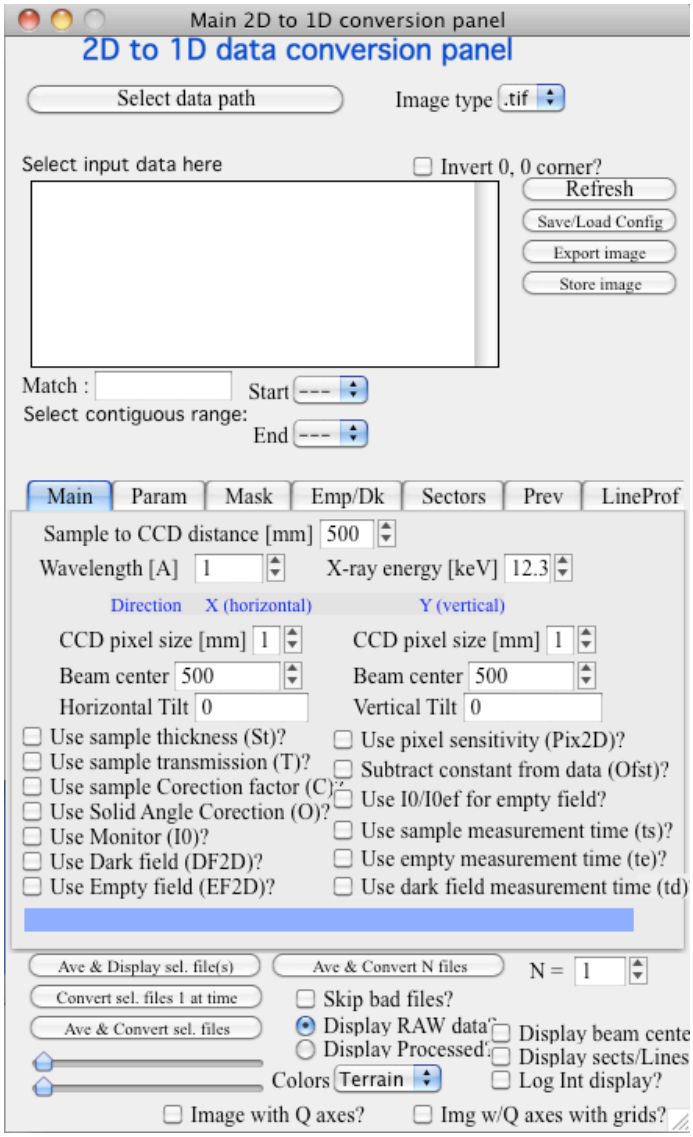
Main Panel

Start Igor Pro and use “Load Nika 2D SAS Macros” command in “Macros” to load Nika package.



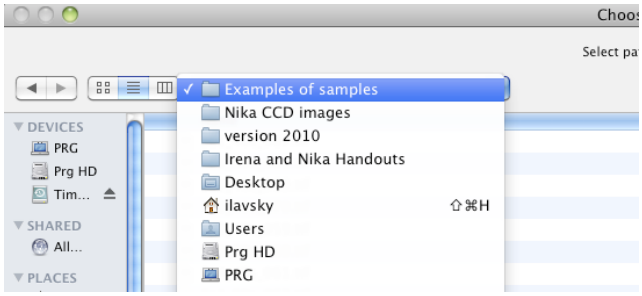
Save experiment with appropriate name (just in case).

Use command “Main Panel” from “SAS 2D menu” to load the main Nika panel.

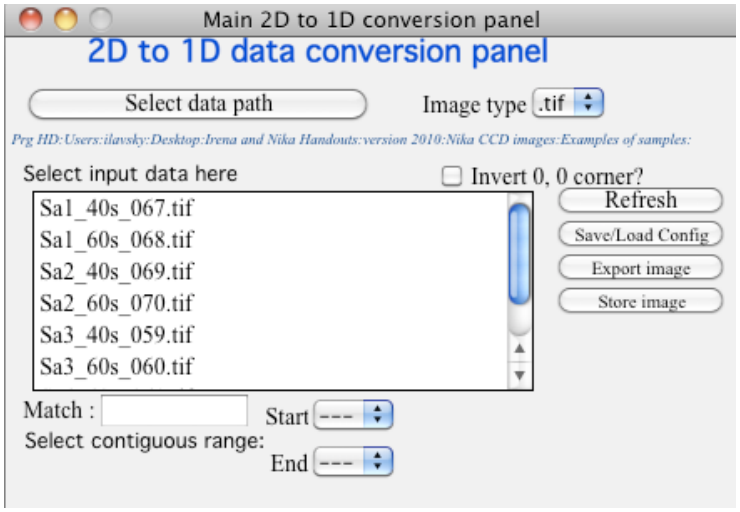


Let's populate the panel with known information… We will start at the top and go towards bottom.

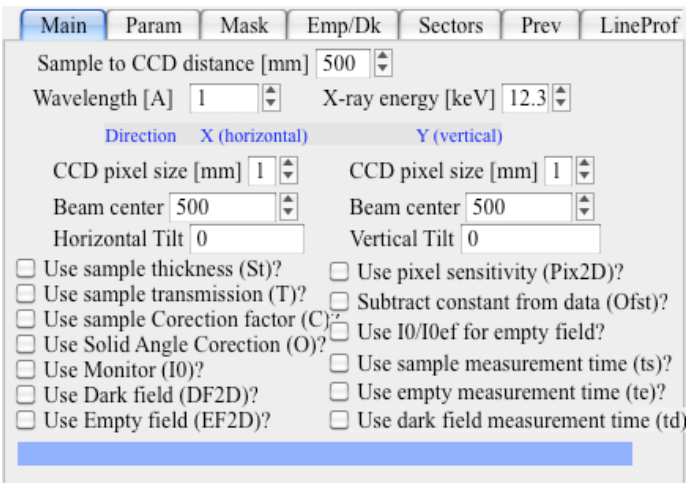
First, push button "Select Data Path". Navigate to where the example Images are.



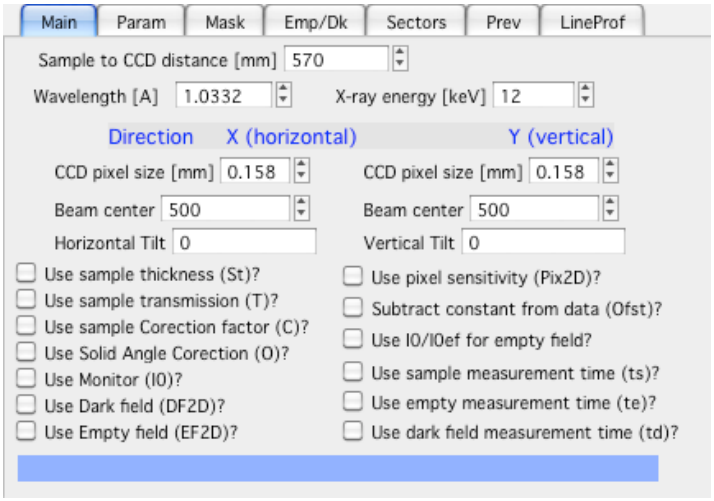
Now the "Select input data here" Listbox should be populated by example images included :



Next, we need to fill the first tab with parameters. This is tab called "Main" (and should be open by default. If not click on it):



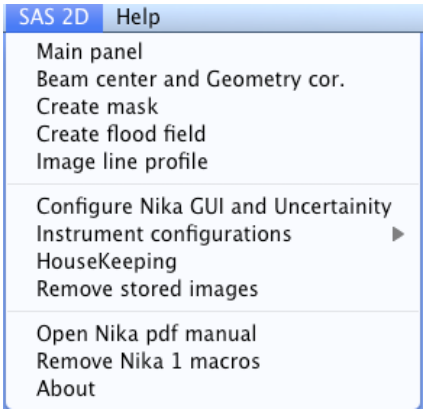
Input known parameters, we may optimize some later, but let's fill it in:



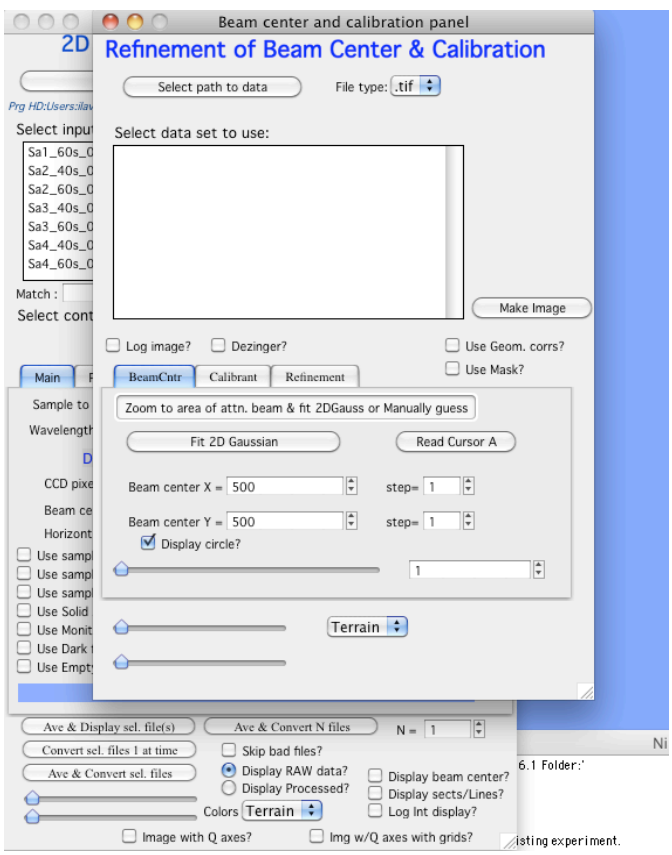
Beam center and distance calibration

Next, we need to deal with Beam center and optimize the distance calibration.

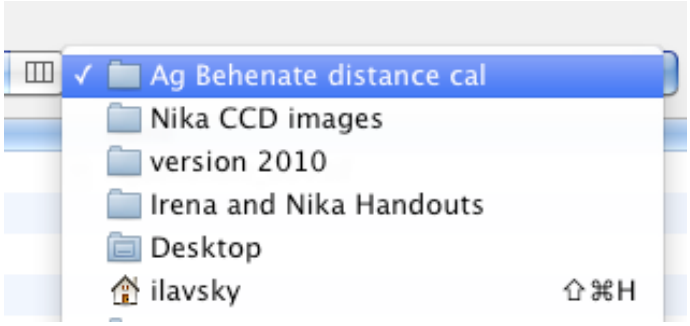
Use command “Beam center and Geometry cor.” In “SAS 2D” menu:

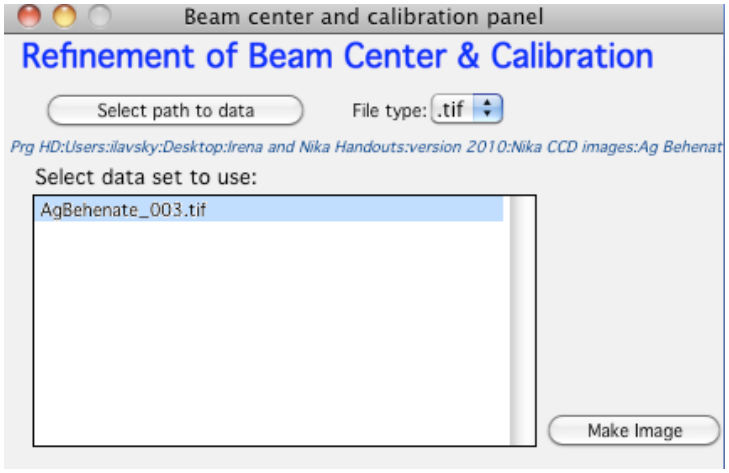


and you should get new panel:

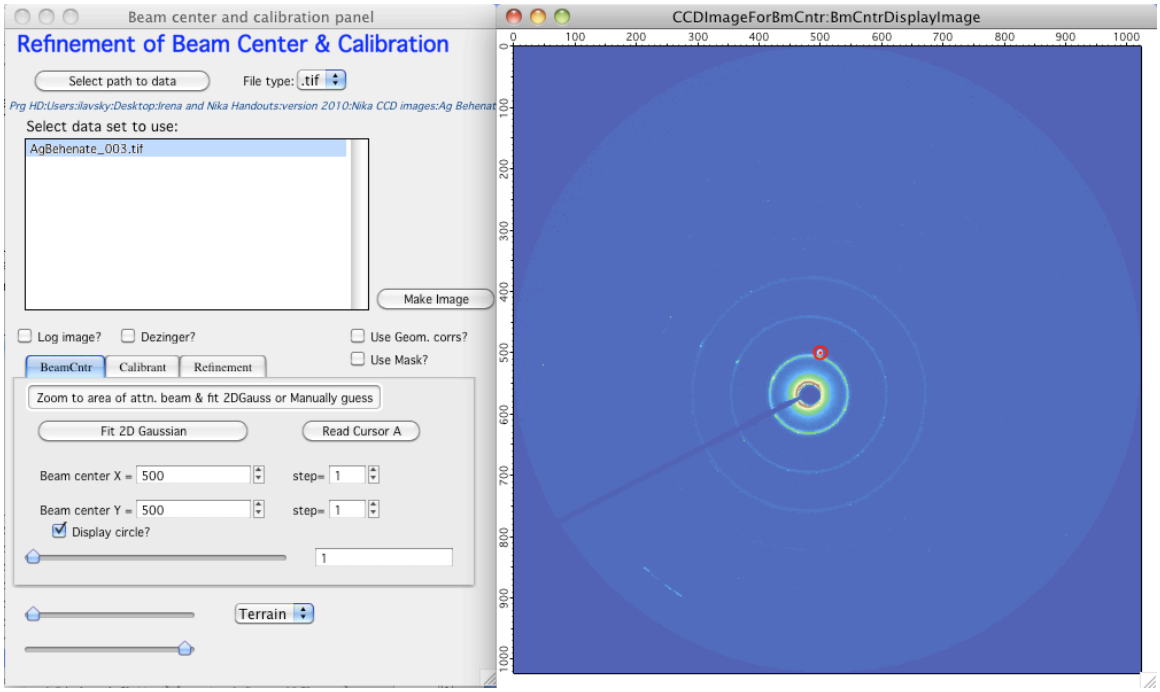


Use “Select path to data” to locate folder with Ag Behenate data provided:



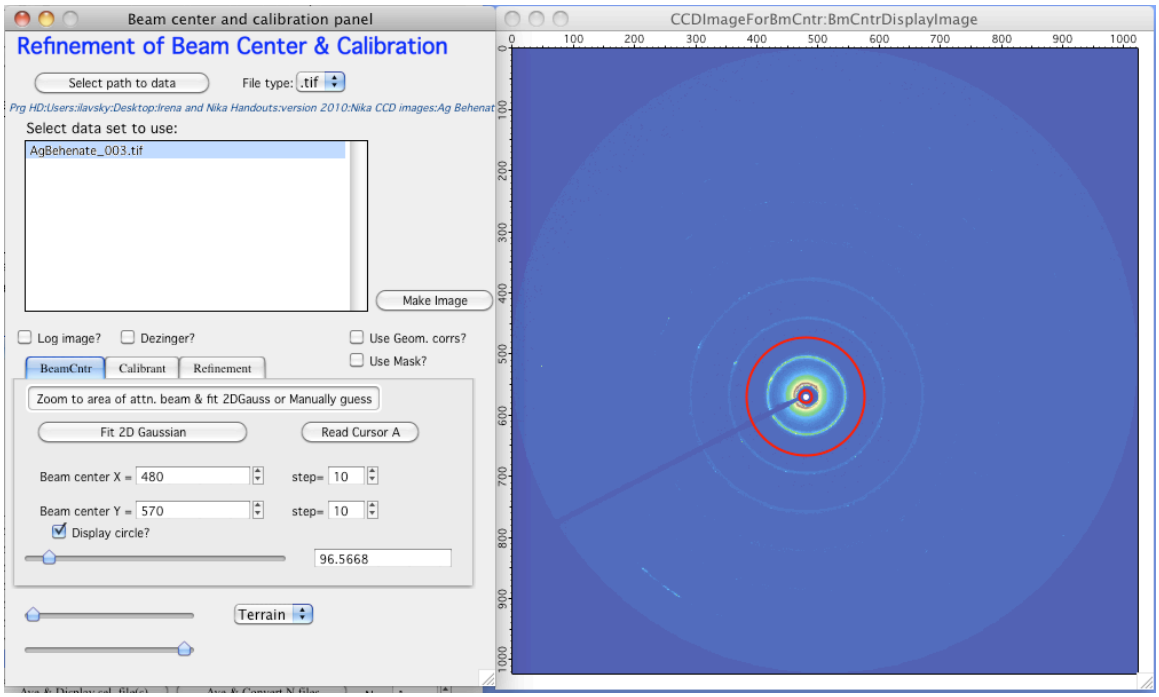


Select the file with measurement and push "Make image" to create 2D image on the screen:



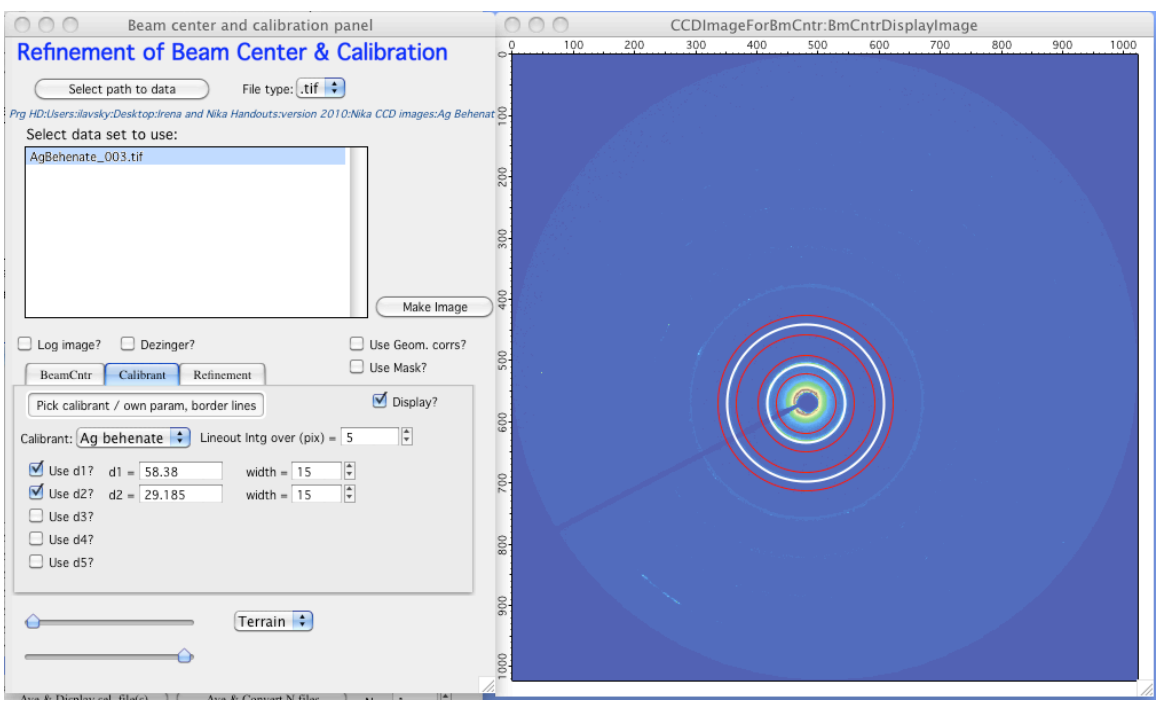
First we need to find reasonable estimate to beam center. Note, the red dot above the beamstop shadow, that is our default estimate for beam center. You can walk the

center there – set steps for both Beamcenter X and Beamcenter Y to something sensible and you can click the red dot to sensible estimate. Also, you can use slider below "Display circle" checkbox to create circle around the center to help with alignment:



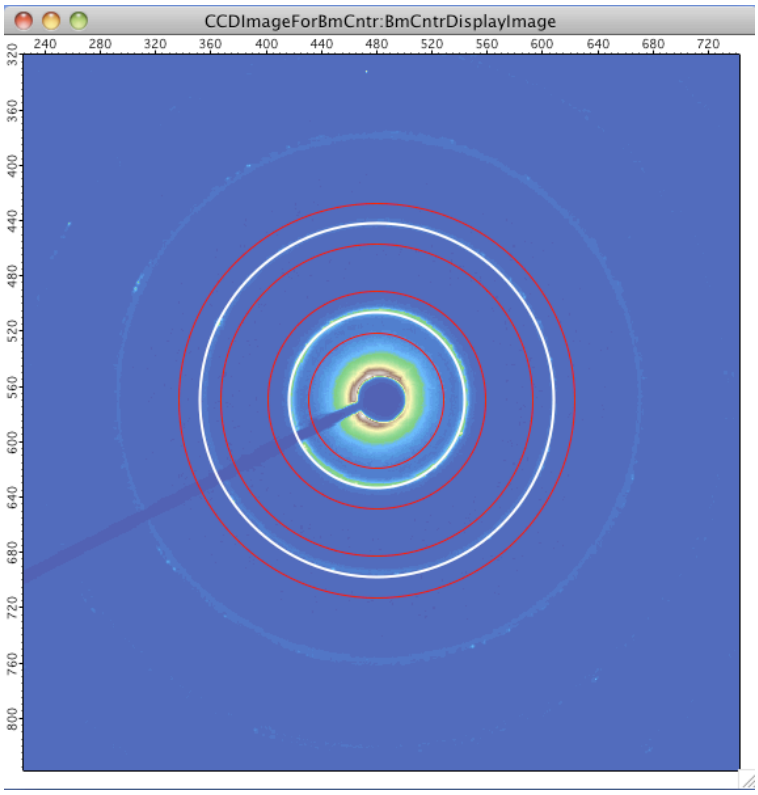
This is reasonable estimate for now…

Next we need to choose calibrant, pick tab "Calibrant" and select Ag Behenate:



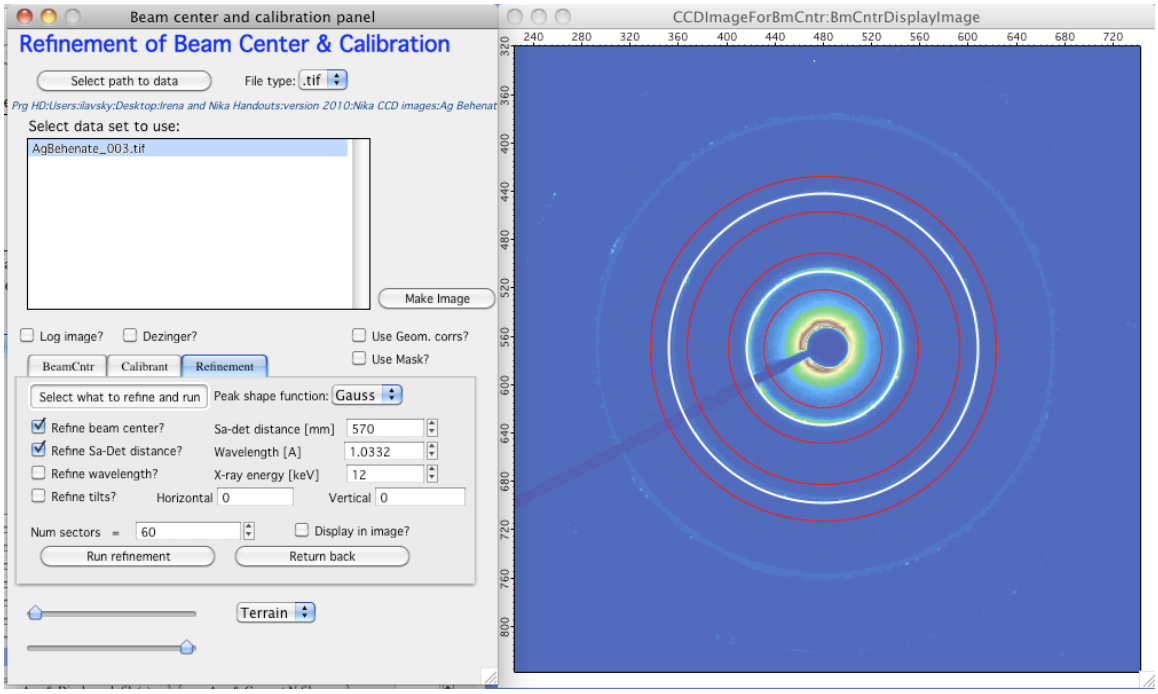
Note, that you can define your own in case you have different material here.

Next we need to understand, what is going to happen next. There are now many "rings" in the image. The white rings are calculated positions for diffractions rings from our beam center, wavelength, sample-to-detector distance etc. The red ones are limits set by the "width" fields. You can zoom in the image to see in more details:

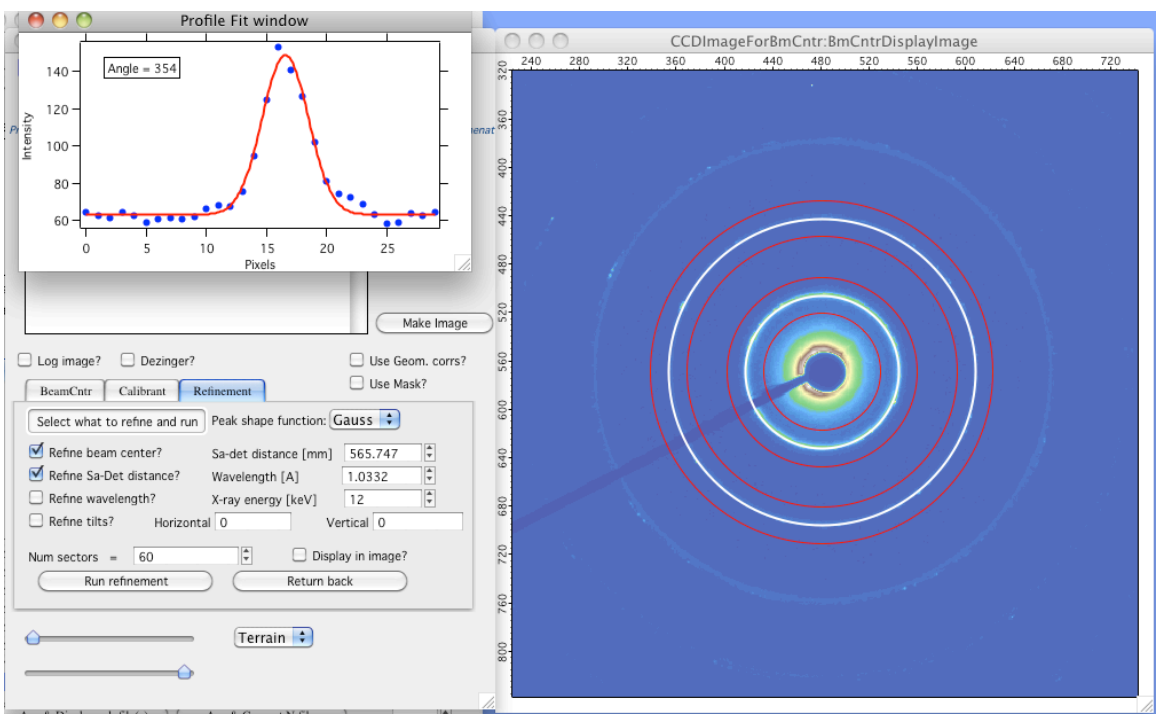


You can remove these lines by unchecking the "display" checkbox to see where the rings really are. In the next step Nika will evaluate positions around the rings between the two red circles – so we need to make sure the diffraction rings are really between them. You may need to make them wider sometimes – or narrower. In this case, we should be OK.

Now go to refinements and check following checkboxes: "Refine beam center" and "Refine Sa-Det distance". Wavelength is basically coupled to distance in small range of angles, so it cannot be refined in SAXS. Most of the times, SAXS data do nto need tilts refined, unless it is really bad…



Now run the refinement. You will see progress on how the code evaluates intensity in radial directions, fits Gaussian distribution to them and finally, uses least square fitting to optimize the parameters as instructed.

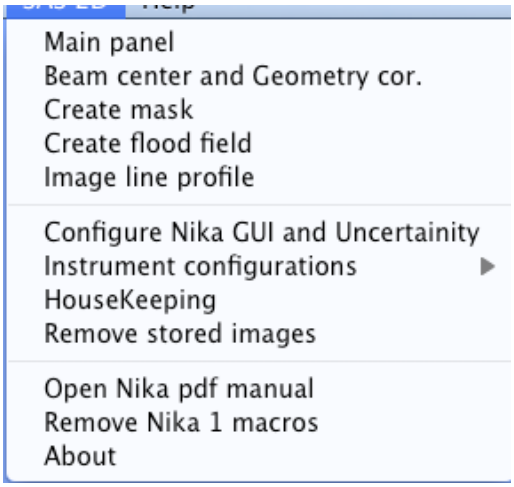


Notice, that the Sample-to-Detector distance changed, as well as the beam center positions.

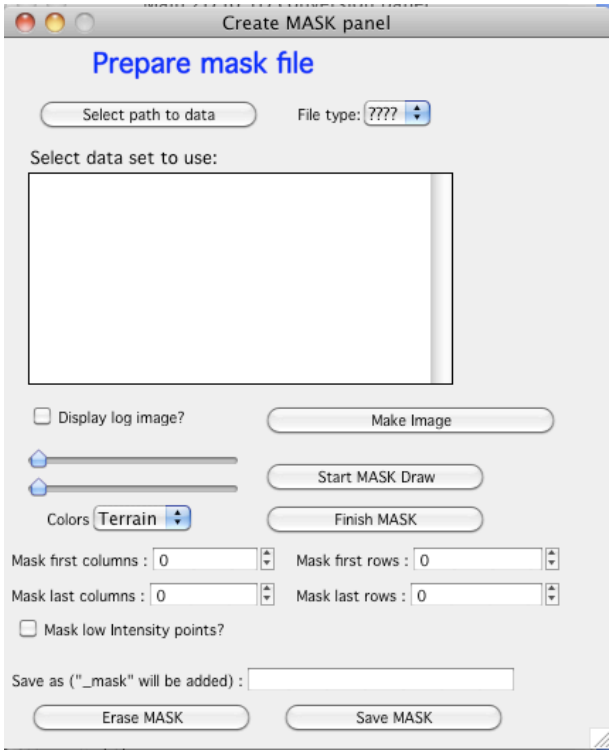
We can now close the three new widnows: “Profile Fit window”, “CCDImageForBmCntr”, and “Beam center and calibration panel”. The parameters were moved successfully to the Main Panel.

Mask design

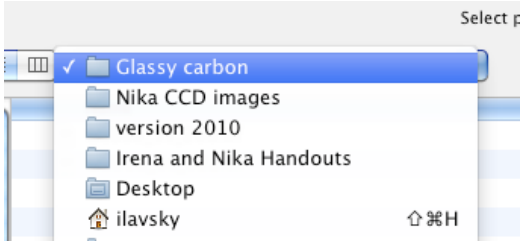
Now we need to design correct mask to be used. The mask design tool is in “SAS 2D” menu, called “Create mask”



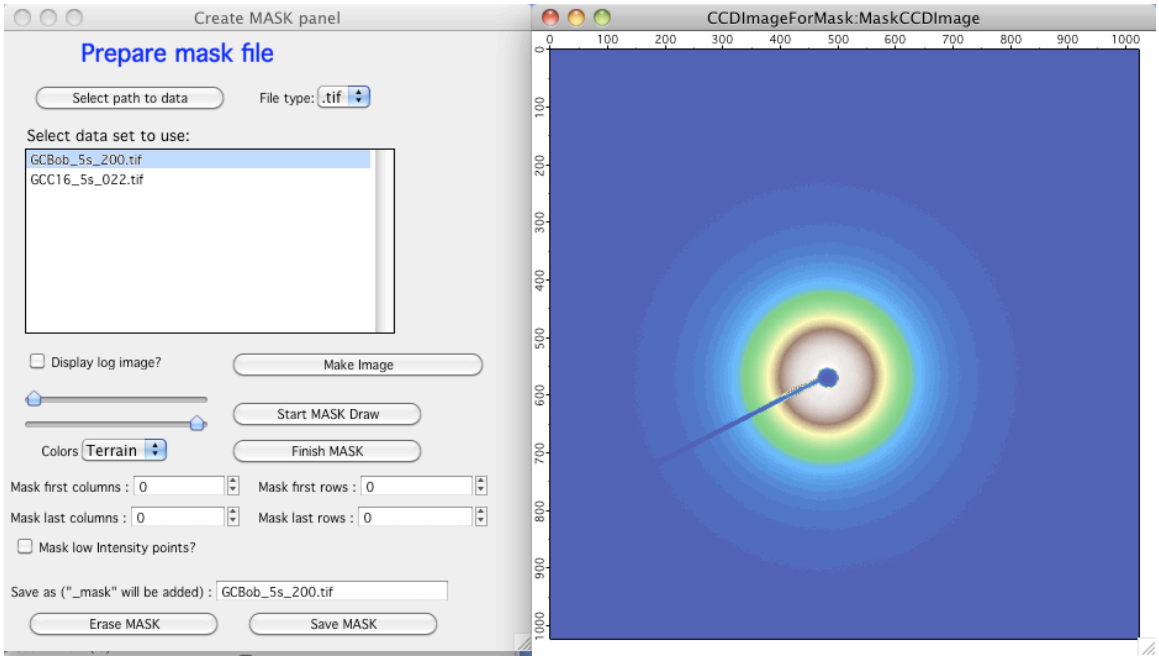
Select it and new panel is created:



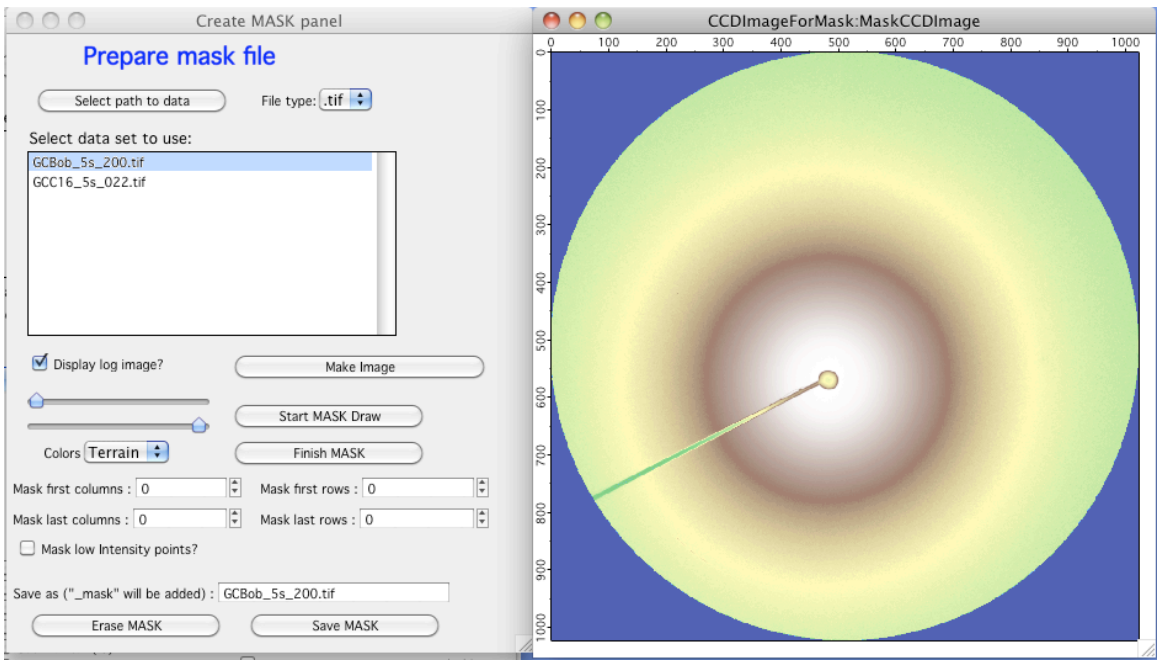
Select "File Type" as tif, "Select path to data" to point to included Glassy carbon data:



Select any of these images and push button "Make Image":

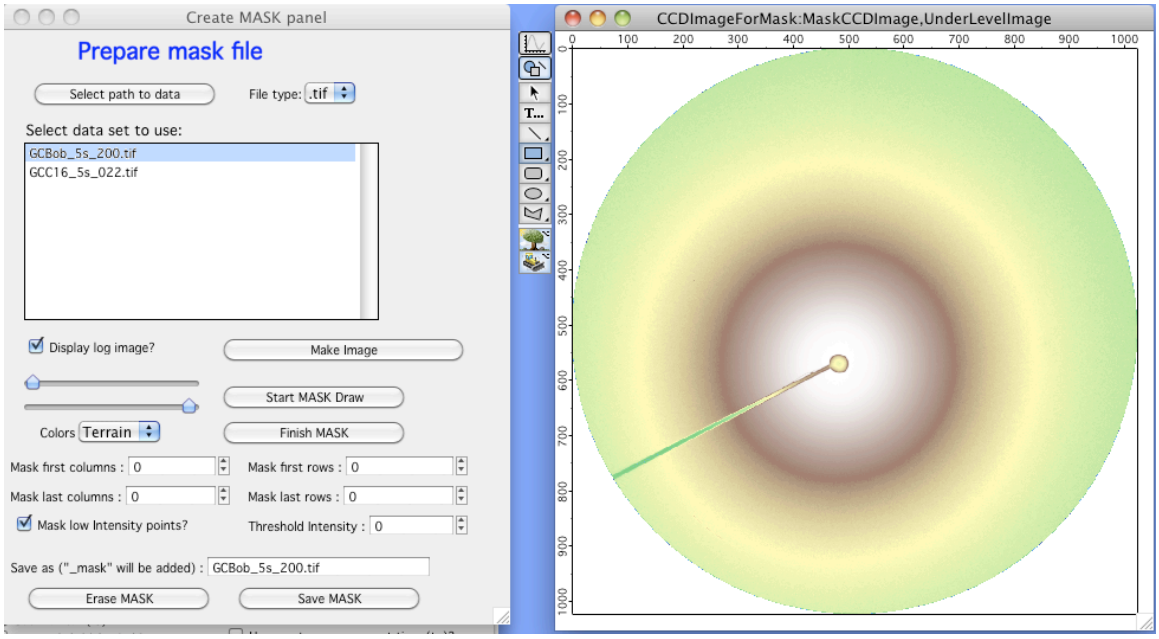


Now select checkbox "Display log image" – helps to recognize artifacts.



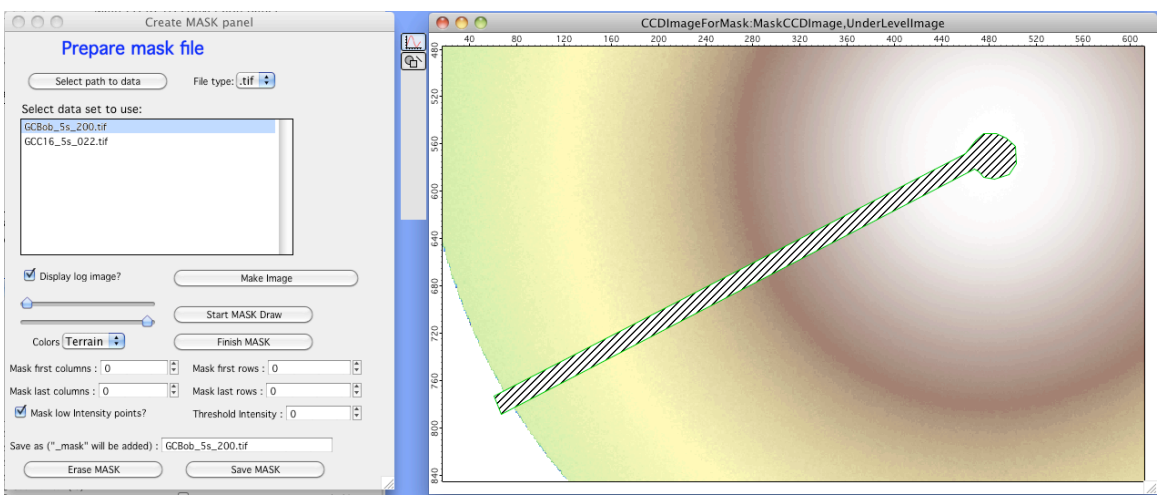
Notice the dark blue areas – that is inactive area of the detector. This can be masked by selecting "Mask low intensity points" (as these have 0 intensity always).

Next we need to mask off the beam stop. We will do it using combination of Igor drawing tools. First push "Start MASK Draw", this will add tools to the left top corner of the image. You may need to move image around:



Now you can zoom using mouse and unzoom (ctrl-A or cmd-A) as needed and use tools (rectangles, circles, etc.) to draw over what needs to be masked over. Cover area to remove. Note, that to zoom/unzoom, you need to select the very top button in the drawing tools, to draw, select the one below that and then the right tool…

This was drawn using the polygon tool:



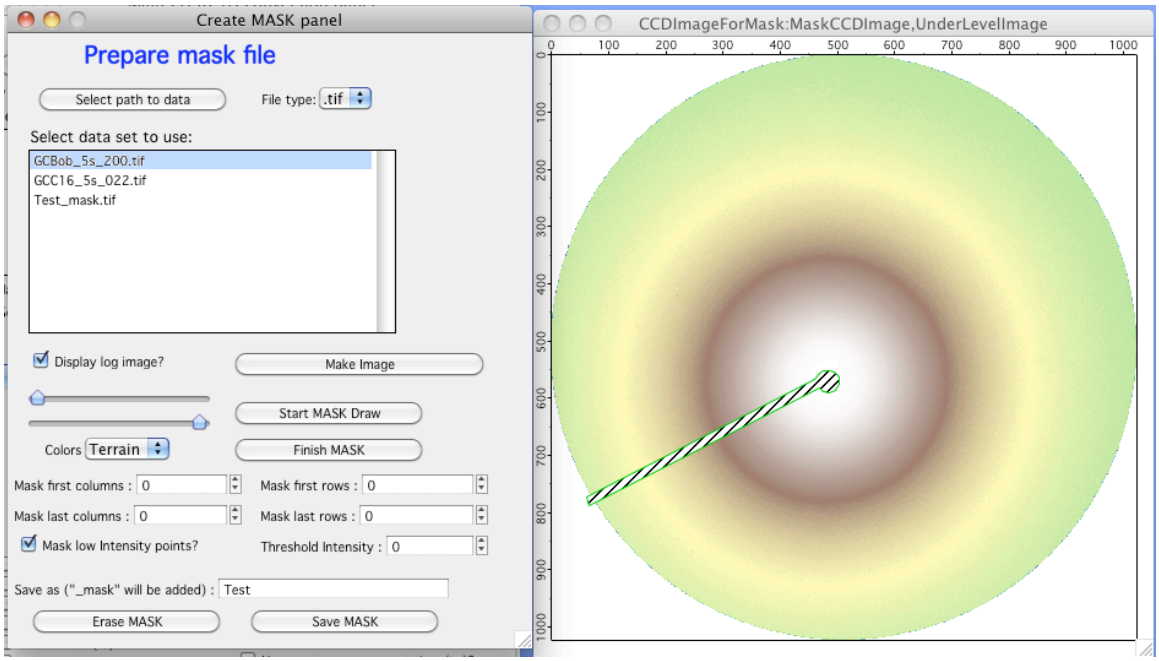
Do nto worry about aspect ratios here, everything is scalled correctly using zoom.

The above cover is OK for the beam stop.

You can also cover first few columns/rows… many detectors have issues with edge pixel sensitivity.

Push “Finish MASK”.

Then add names as necessary to the field “Save as…” and “Save mask”.

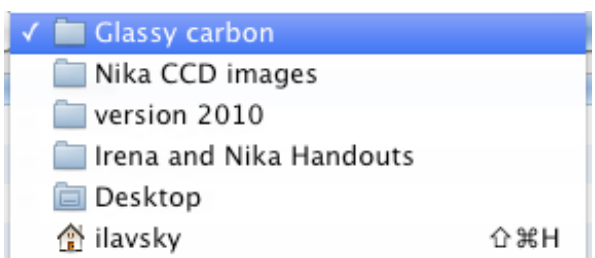


The mask has been created and also stored for use in current Igor experiment. In the future you can load this mask from the stored tiff file with _mask.tif as part of the name.

Close the “create MASK panel” and “CCDImageForMask” windows.

Absolute intensity calibration

We will use Glassy Carbon measurements (measured transmission 0.818). On the main panel, find the path to the Glassy Carbon measurements:



We will use GCBob_5s_200.tif measurement. Thi sis 5 second exposure measurement. The processing for this image will be as follows… Select in the checkboxes following ones:

Use Sample thickness (it is 1mm)

Use sample transmission (it is 0.818)

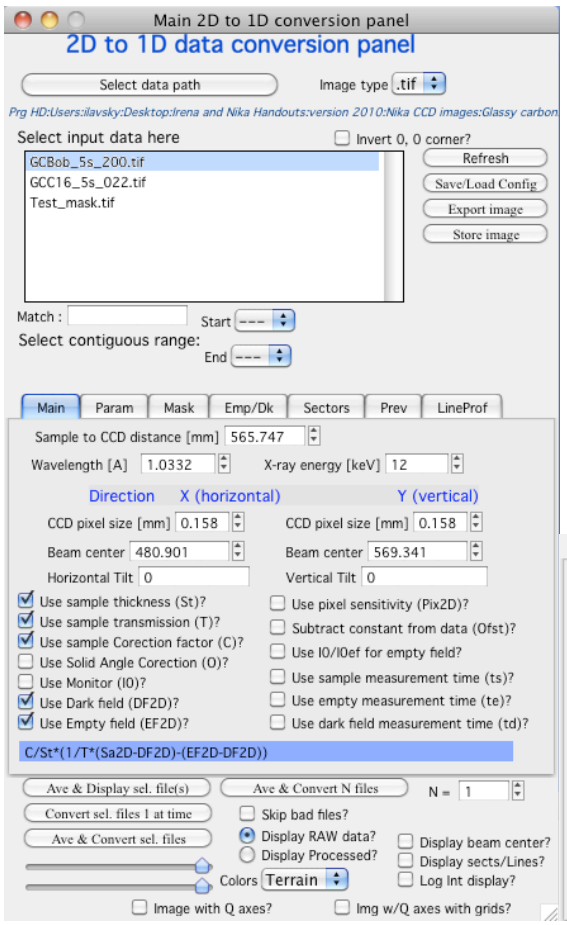
Use sample correction (and we'll set it to 1 for now).

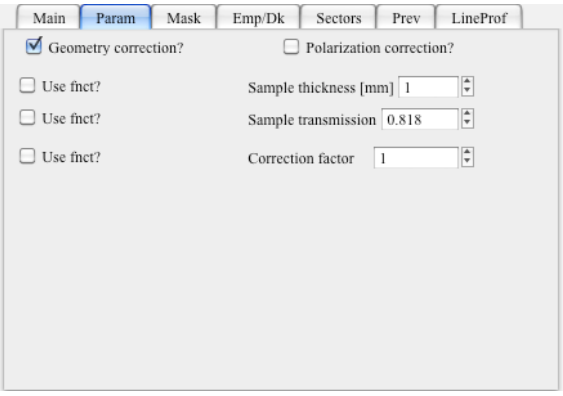
Use Dark filed

Use Empty field.

We will assume in this case, that the time is sufficiently good for normalization purposes. If available, we could use Monitor values for higher precision…

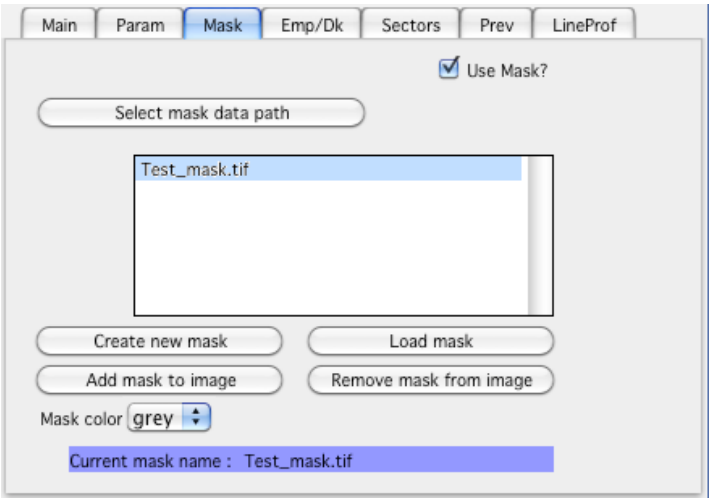
This is the setup (Main tab; Param tab):





Note, that we selected also "Geometry correction?" checkbox. Read more in manual, but in this short sample-to-detector distance this is better. Remember to input 0.818 for Sample transmission.

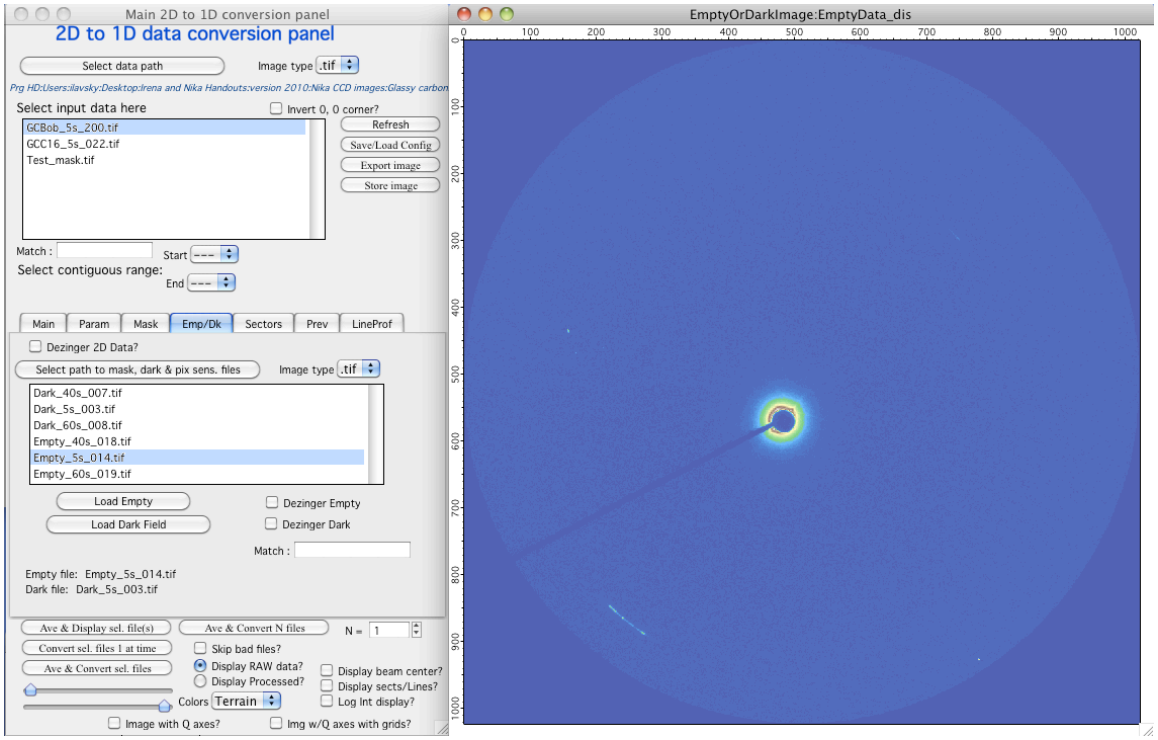
Further, in Mask Tab select "Use Mask".



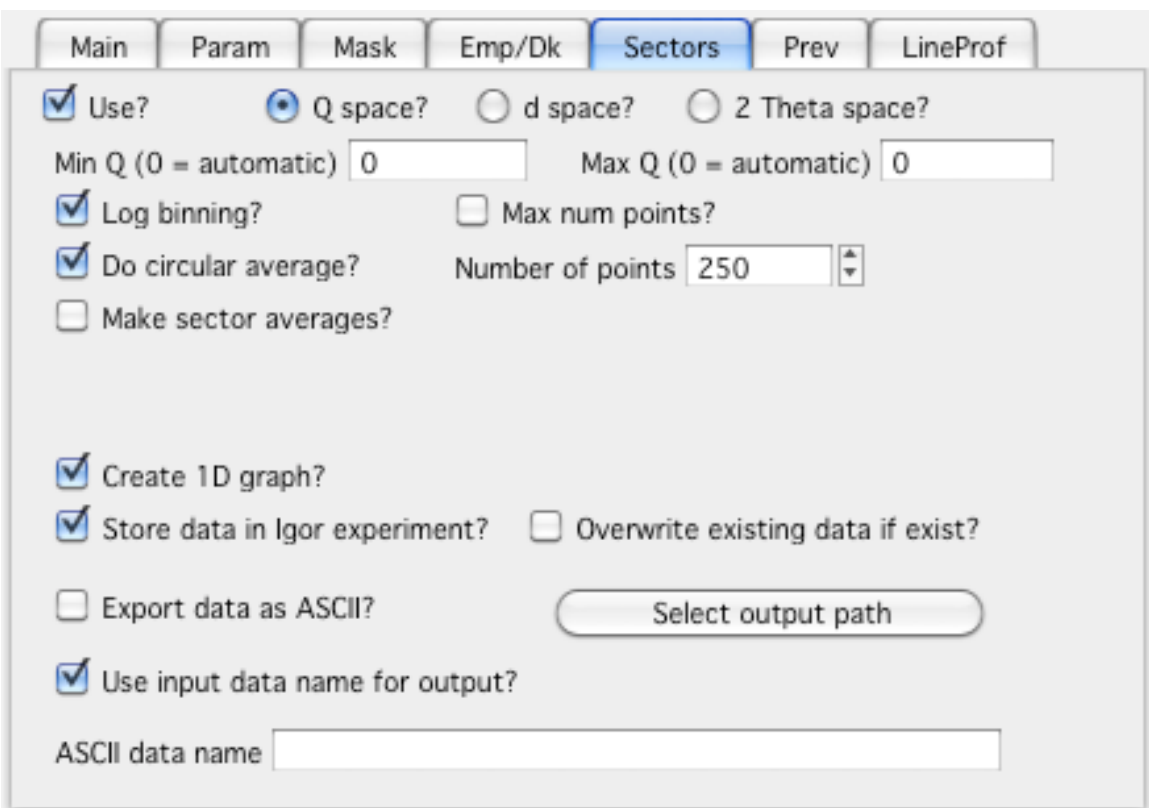
Since we just created the right mask, we can just use the one in Nika now. Else you would need to load mask you want from saved file (located with the data) or create new mask…

In Next tab, "Emp/Dk" use the "Select…" button to navigate to where empty and dark field measurements are:

choose Dark_5s_003.tif and push "Load Dark Field". Choose "Empty_5s_014.tif" and push button "Load Empty". The files are loaded and displayed…

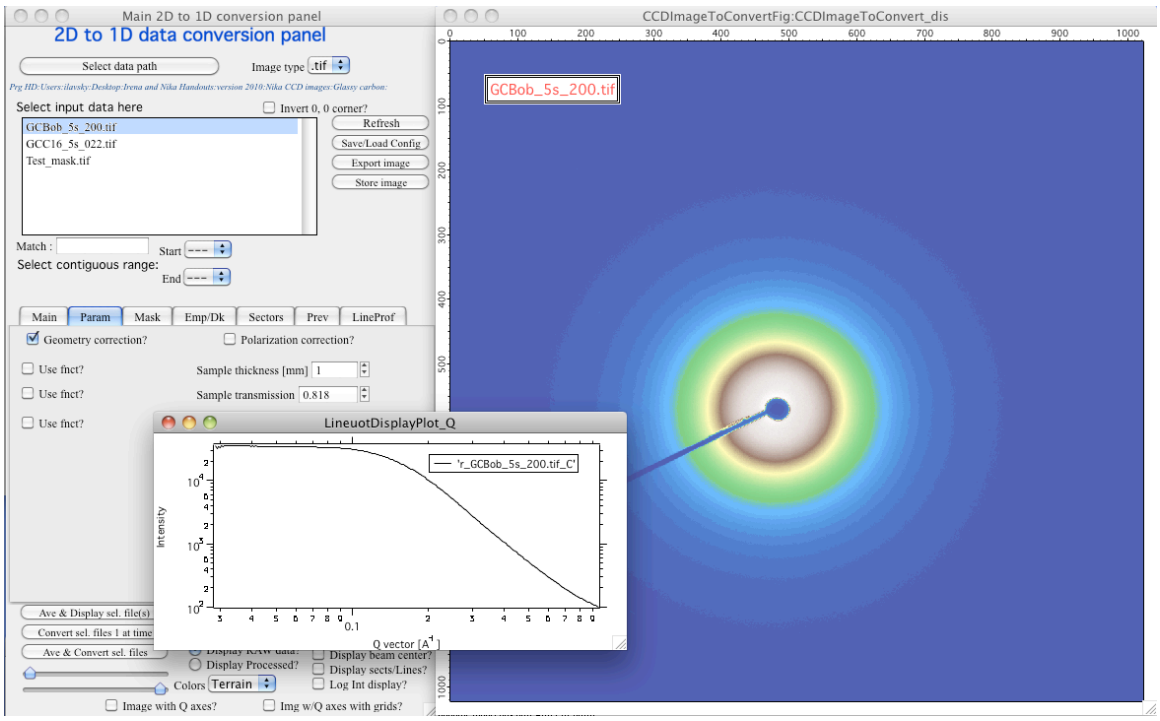


Next we will use "Sectors" tool to generate for our GCBob measurement the radially averaged data. Make selections as in the figure below:



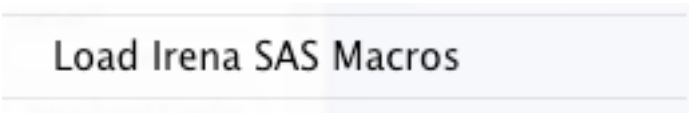
We will use log-Q binning (useful for SAXS), 250 points is more than enough, we will do Circular average (this is isotropic sample) and create 1D graph. We can use the image name for data name – makes everything easier…

Now, in the main panel in the top listobox "Select input data here" select the "GCBob_5s_200.tif" file and push button at the bottom of the Main panel "Convert sel. Files 1 at time". This will convert the file into 1D lineout:

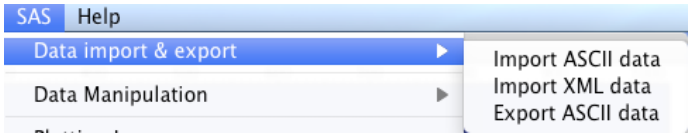


We have got now correct profile, but scaling of intensity is still in arbitrary units. We now need to calculate scaling constant which would put the data on absolute scale. For this we need Irena package and calibrated ASCII data for GC Bob included with the images.

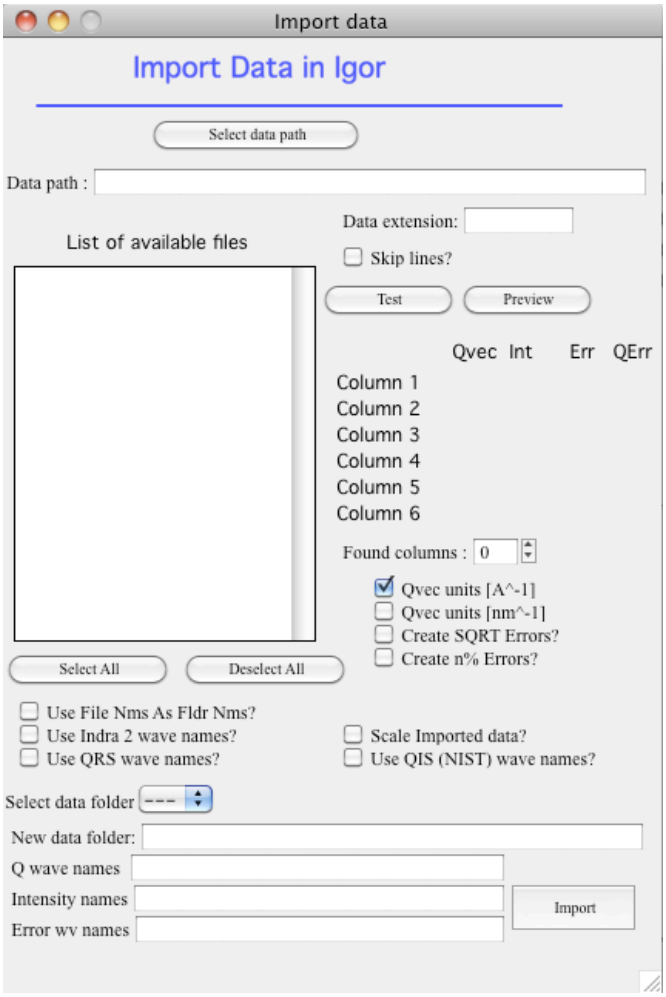
Load Irena package from "Macros menu" using command :



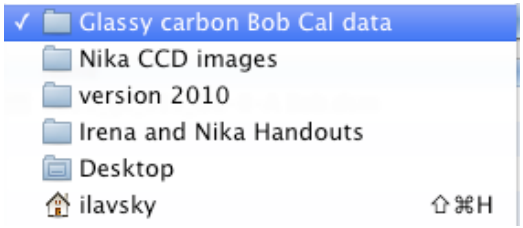
Then we will import data using SAS menu command:



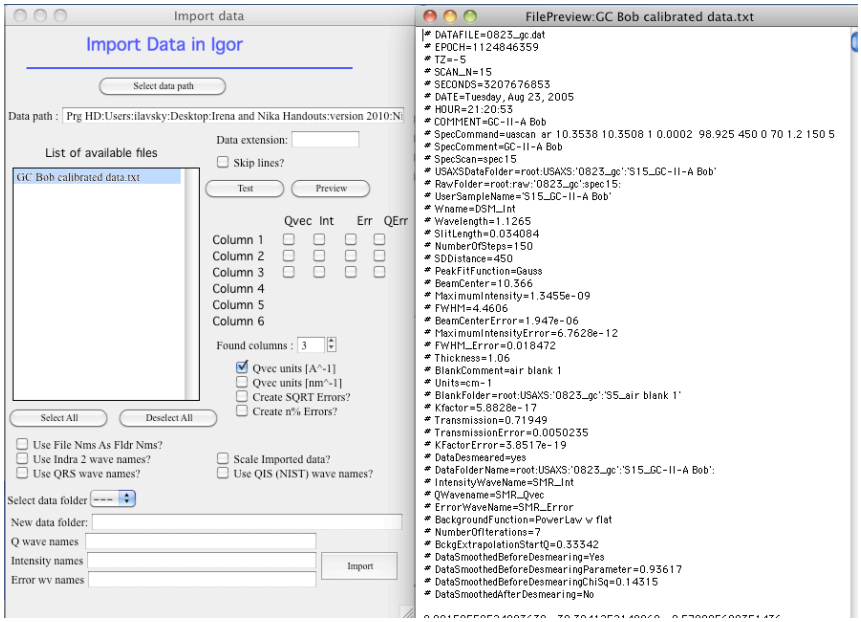
"Import ASCII". This creates new panel:



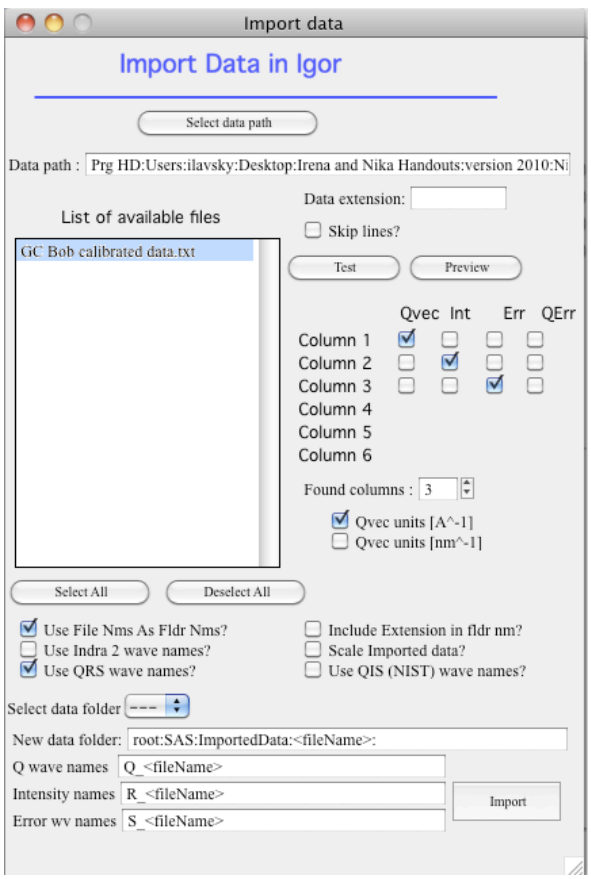
Select data path to the data:



And choose the “GC Bob Calibrated data.txt” file. The push “test” button and “Preview button”. Igor will test how many columns of data it can find and show preview of the data file:



Please fill the Import Data in Igor panel as below and then push “Import” button.



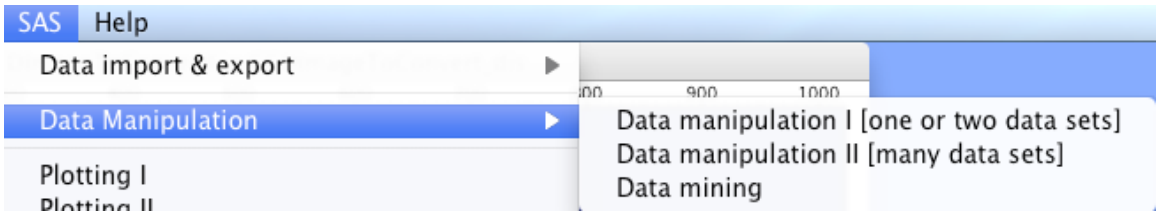
A note is made in the history area about the process:

@ Jan Ilavsky

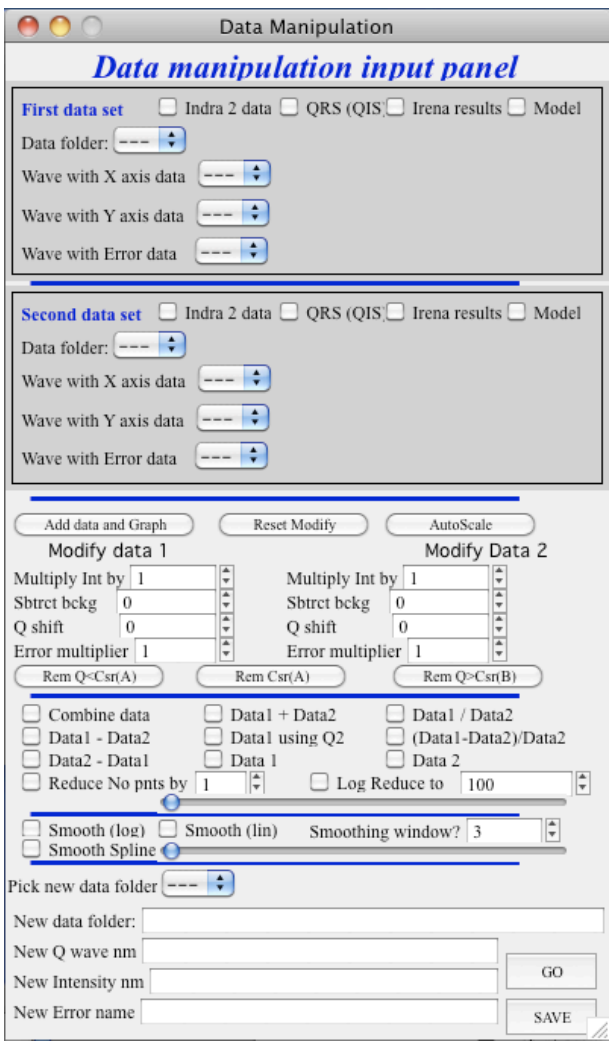
```
Imported data from :Prg HD:Users:ilavsky:Desktop:Irena and Nika Handouts:version 2010:Nika CCD images:Glassy carbon Bob Cal data:GC Bob calibrated data.txt
    Data stored in :          root:SAS:ImportedData:GC Bob calibrated data:
    New Wave names are :   R_GC Bob calibrated data    Q_GC Bob calibrated data    S_GC Bob calibrated data
Imported 1 data file(s) in total
```

Close the "FilePreview:…" notebook and "Import Data" panel.

Next we will use "Data Manipulation I" tool from SAS menu:



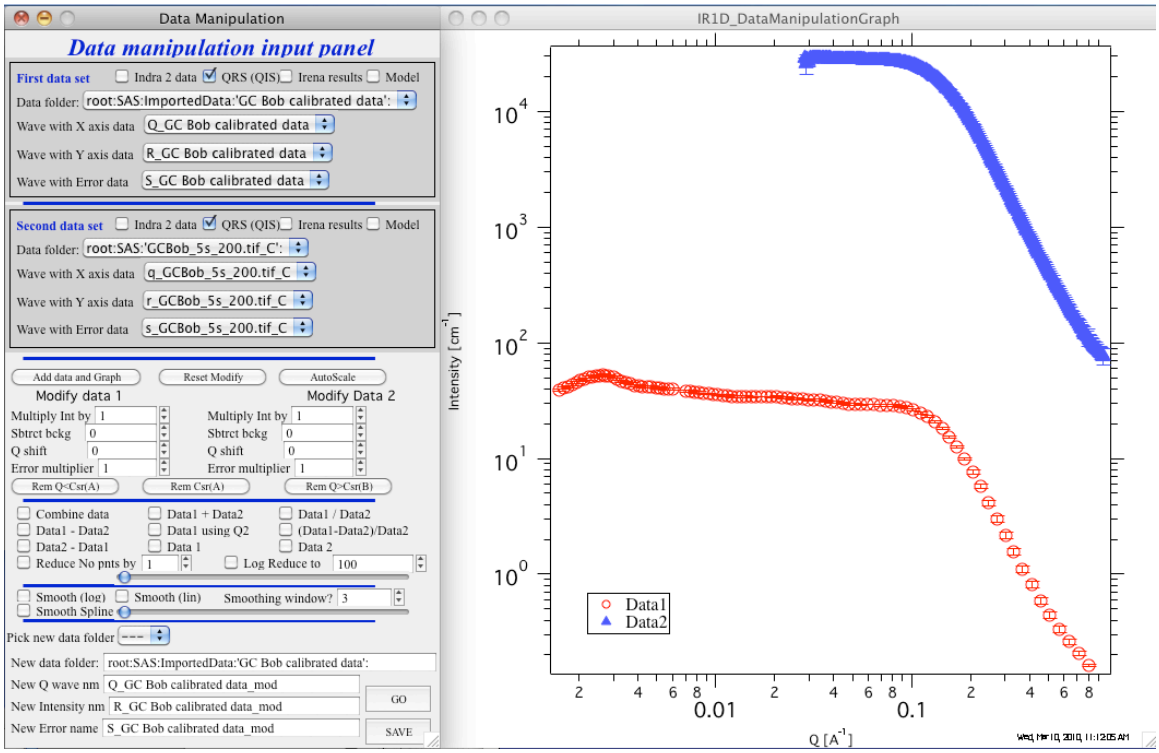
Select the command and this will create new panel:



We will use this tool for scaling the data together to get calibration constant…

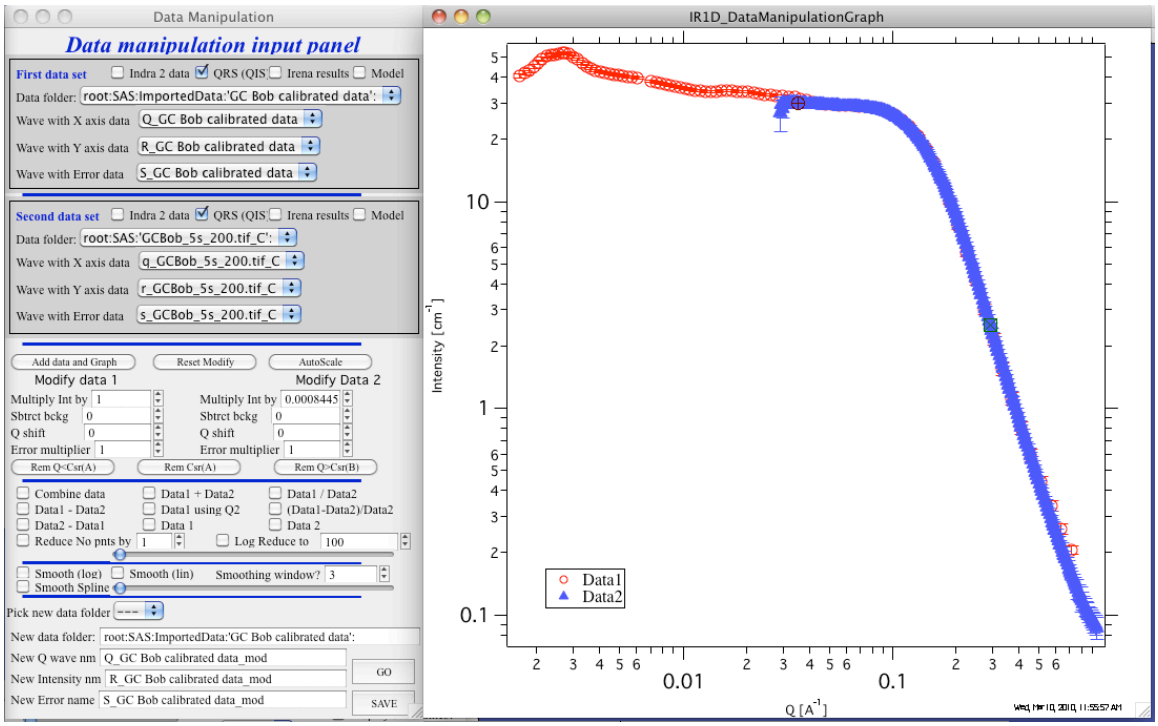
@ Jan Ilavsky

Select for both “First data set” and “Second data set” controls the “QRS (QIS)” checkbox and then select for First Data set the data we just imported and for second data set the Nika reduced data as below. Push button “Add data and Graph”:



The red data are absolutely calibrated data we are suppose to get, Blue are our arbitrary calibrated data we got. We now need to scale them together. Place cursors (A is the circle and B is the square, at the bottom of the graph) on the blue curve. The area between the cursors is going to be used for scaling. The A should be may be on point about 7 or so (to avoid the weird decrease in intensity at very low Qs), B should be around 150 or so, to avoid the area where there is significant amount of flat background in the USAXS measurement. Then push button “AutoScale”. The value in “modify Data 2”, “Multiply Int by” is about 0. 0008445. This is our scaling constant – NOTE, that in the current case, this scaling constant is time dependent and this one is for 5 seconds. For other times we need to correct by scaling by (5/time of exposure).

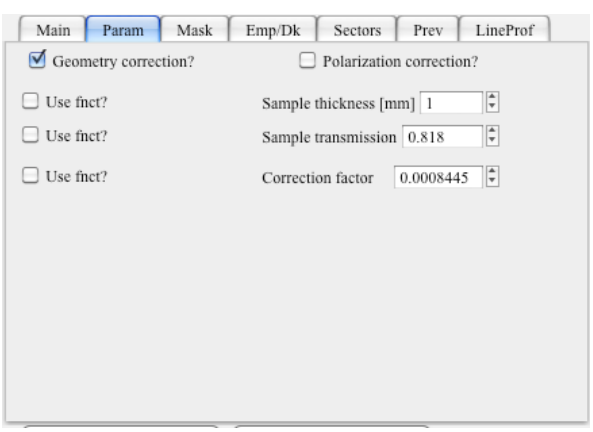
For example for 50sec measurements, the Claibration constant will be 0. 00008445 etc. This could be avoided if we used Monitor for exposures – but while in real life we do this, it would make this handout more difficult to follow….



OK, finally, we have calibration constant for our measurements. For 5 seconds it is 0.0008445 and for other times we need to scale it by (5/time of exposure).

Testing

Let's test the scaling constant… Close the "Data manipulation" panel as well as "IR1D_DataManipulationGraph" window. Then in the "Param" tab of the "Main 2D to 1D conversion panel" set the Correction factor to the above value:



and select the two measurements for Glassy carbons (GCBob and GCC16) provided and push button "Convert sel. Files 1 at time"… If you get question about overwriting, choose Yes.

You get new plot of the radial intensity:

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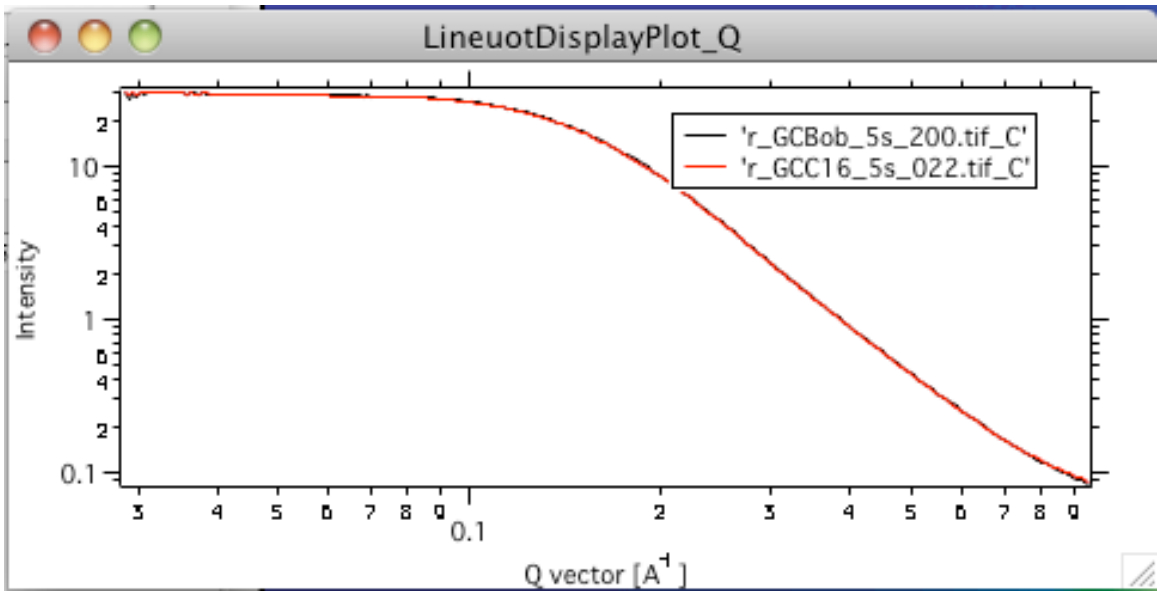
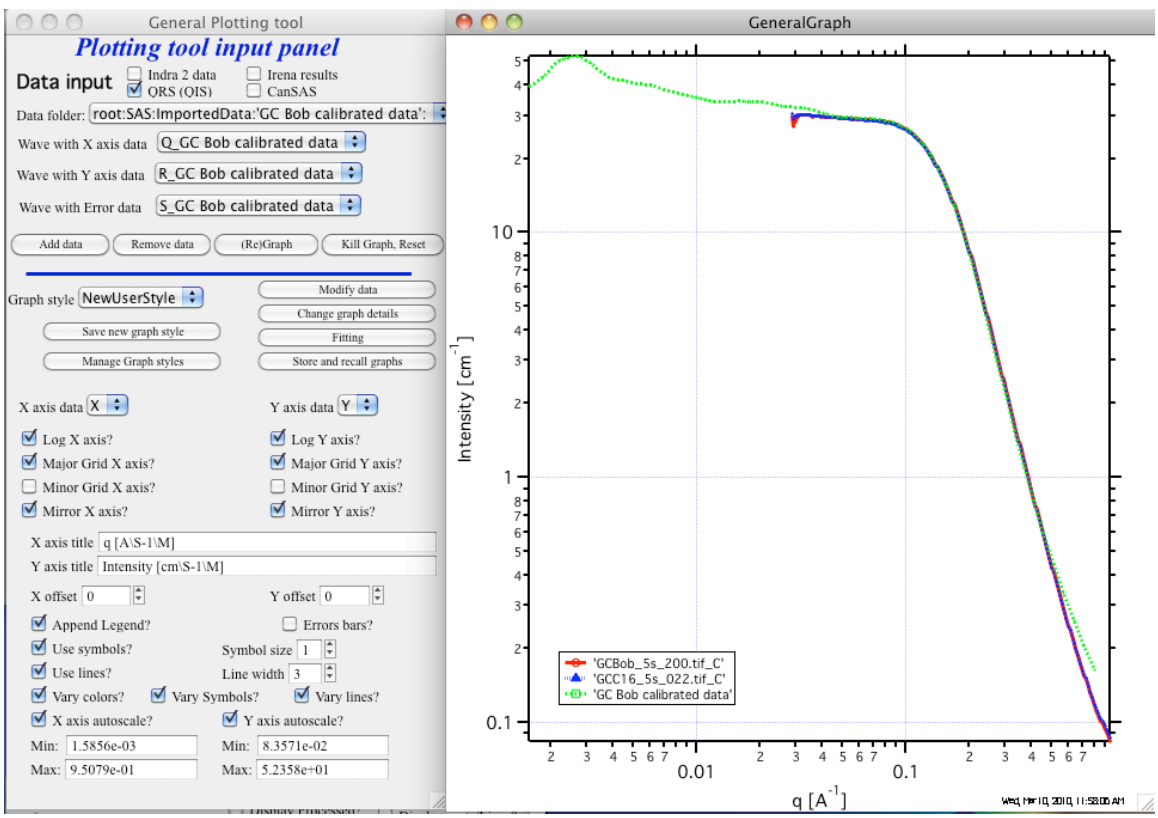


Note, both overlap (they are basically same) and now intensity at low Q is about 30 or so. That is correct. To check further, use "Plotting tool I" from SAS (Irena) package. The data are all QRS and you can now plot both Glassy Carbons Nika reduced as well as the provided standard measurement:



You can see that where the data overlap there is good agreement. AT high Q USAXS data contain more flat background (that is usual).

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This is the end of handout part I. In Part II we will see different methods of data reduction.

Please, make sure you save current Igor experiment. You will need it in the Handout part II. You can close the plotting tool and associated plot window…