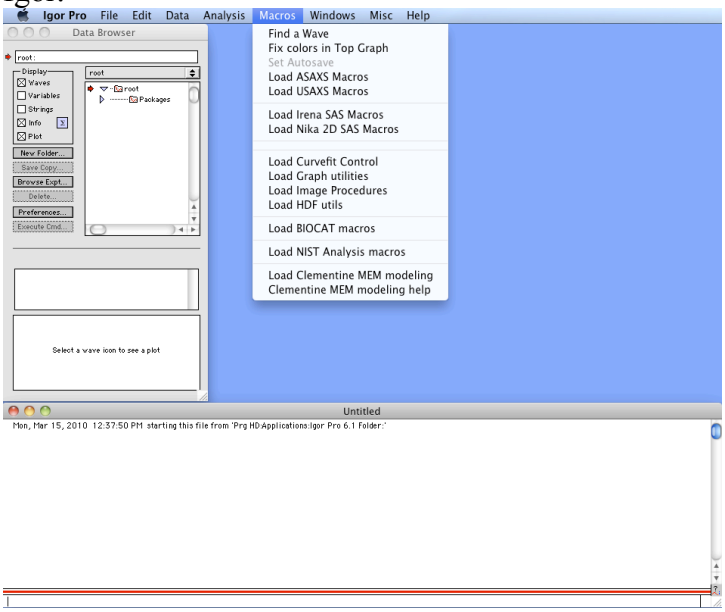
Size distribution using Sizes tool in Irena package

This handout describes how SAS data (example using provided data from USAXS instrument) can be fitted using Sizes tool in Irena (using Maximum Entropy of IPG/TNNLS methods) with size distribution of spheres. Three test data provided were measured in 2001 and represent SAS from samples of alumina polishing powders. The powders were spread on sticky tape and covered with another layer of the same tape (sticky sides towards each other). Same two tapes were subtracted as empty run. The data are not, however, calibrated as the sample thickness of these samples is not really meaningful.

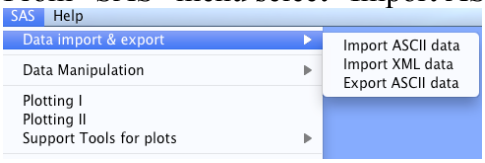
Start Igor, load macros

Start Igor Pro, from the menu "Macros" select the "Load Irena SAS Macros". This will add new menu SAS in Igor.

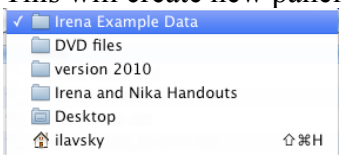


Import data

From "SAS" menu select "Import ASCII data".

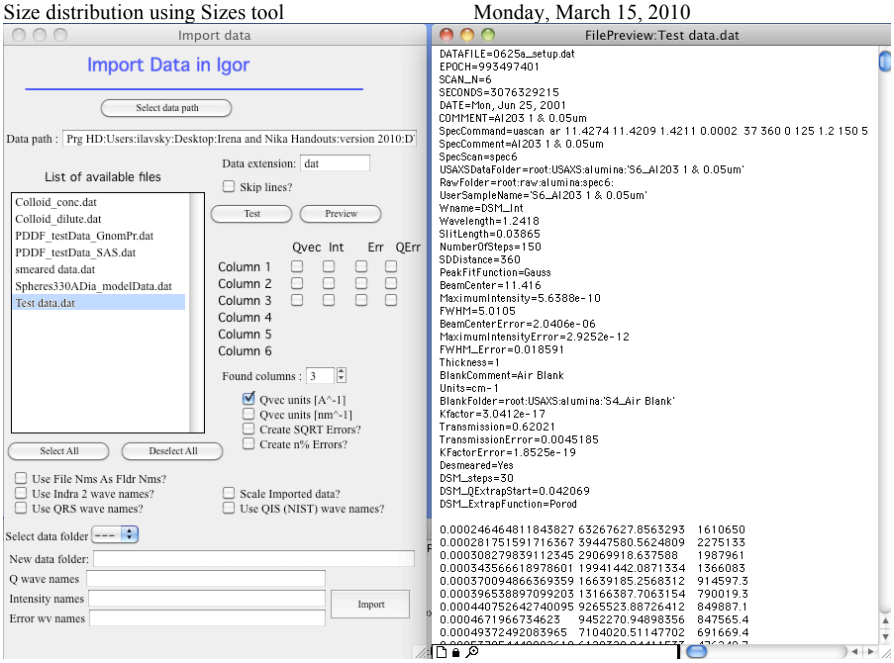


This will create new panel. Push the button "Select data path" and navigate to folder, where are the data.



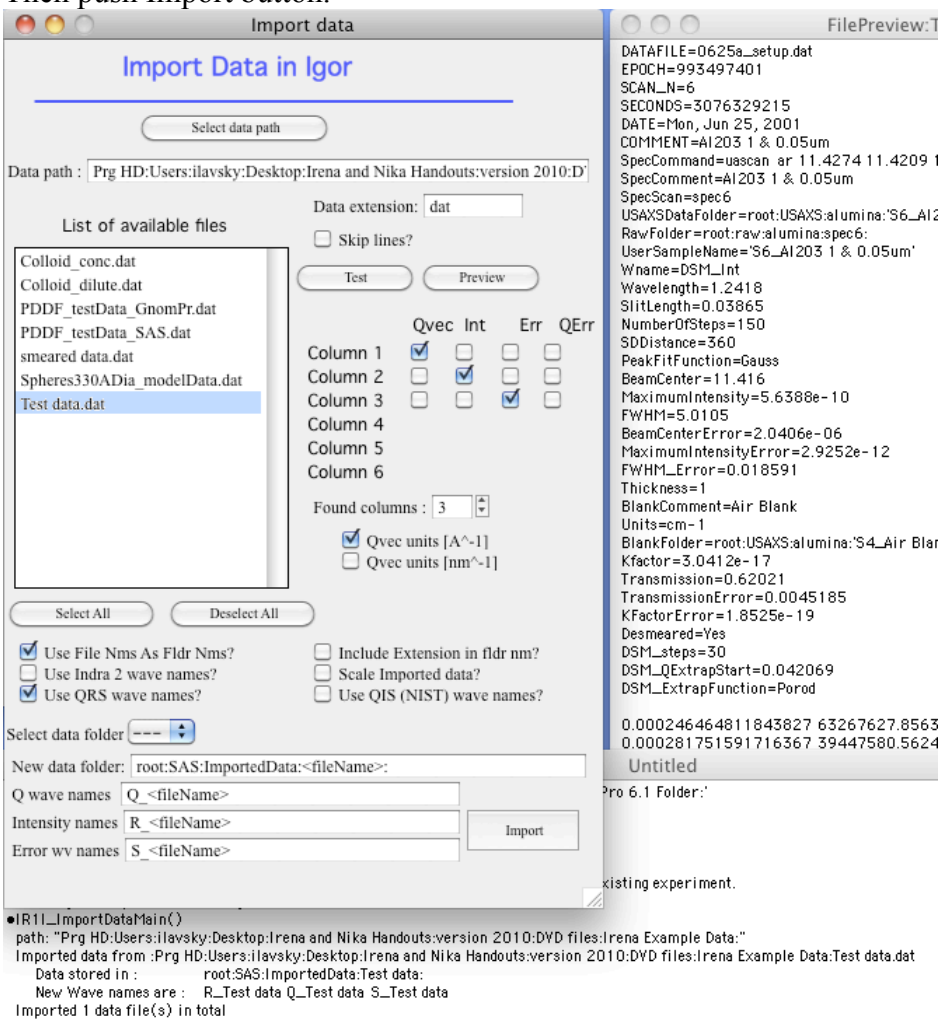
The test data provided (Test data.dat) have extension dat, we can put this in the "Data extension" field and only files with this extension will show.

Select the "Test data.dat" and push buttons "test" and "Preview". The Test will find how many columns of data are in the file and Preview will open this file for our previews in separate window.

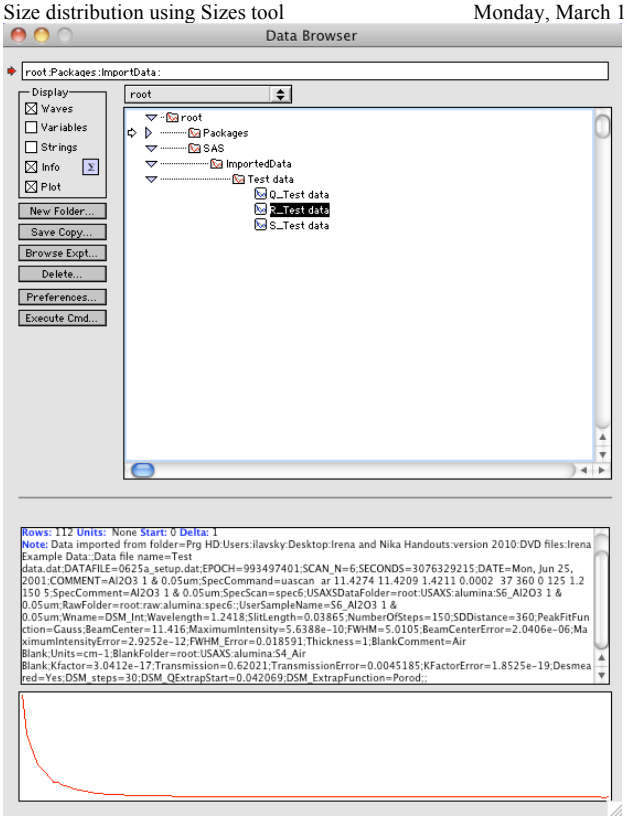


Note, that after header this files contains 3 columns – q, intensity and error estimates. Close preview and check the checkbox for column 1 as Qvec, column 2 as Int and column 3 as Err.

Select "Use File Nms as Fldr Nams?" and "Use QRS wave names". Select the file with data (Test data.dat). Then push Import button.



This will import the data into Igor – see below:



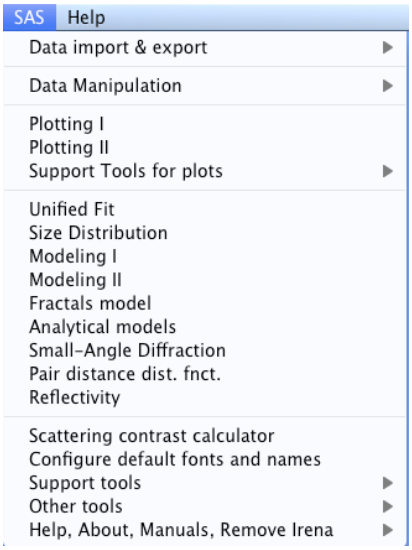
Just for information: Note, that the header from the ASCII file we had is now attached to the wave in so called wavenote – it is visible, when you select the wave in the Data browser and have the "info" checkbox checked. This is how Irena macros store additional information with waves – as wavenotes, which are text files attached to the waves.

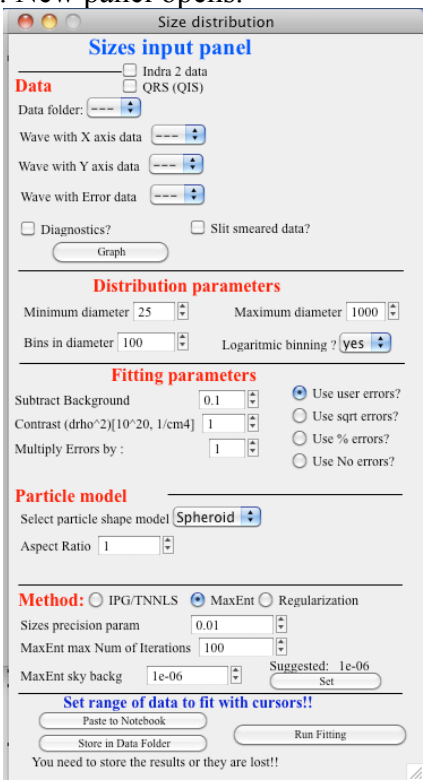
Fitting using Sizes – Maximum entropy or TNNLS methods

Setup

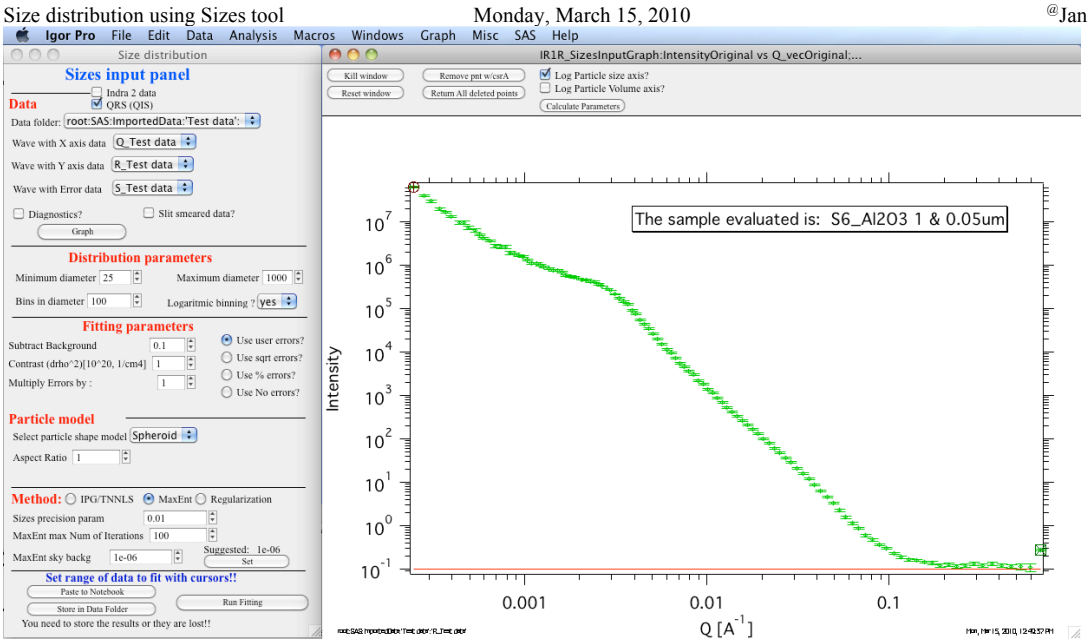
Close all import-related windows (Panel and Notebook).

From “SAS” menu select “Size distribution”. New panel opens.





Our data use “qrs” naming structure, so check the checkbox “Use qrs data structure”. The select in Folder with data popup folder “root:SAS:ImportedData:’Test data’:” – this is folder with sample of alumina powder, mixture of two sizes of Alumina powders.
The wave selection popups (“Wave with X axis data” etc.) should be populated automatically. Push “Graph” and new graph with data should appear.



Fitting parameters

The manufacturer sells these polishing powders as 1 micron and 0.05 micron (50 nm, 500 Å), so we should fit it's size somewhere between 50 A and 10 micron (10000A). In this wide size range we need to use logarithmic binning for sizes. Using 100 bins in diameters is reasonable maximum for data sets which have about 125 points – and generally for most purposes.

So set Minimum diameter to 100A, Maximum diameter to 10000, bins in diameters 100, and logarithmic binning.

Next we need to have a good background subtraction – the flat background. The red horizontal line of the graph is the flat background as is set in the control panel. The flat area at high Q of our data has intensity about 0.12, so we can change the background to that.

The material is alumina, which has contrast 1094 10^{20} cm^{-4}, but the sample was powder, so it's calibration is unsure anyways. But we can use the contrast anyway, even thought the results may not really be meaningful… So set the Contrast to 1094.

Now errors – we can relax the fitting and multiply the errors some – set for now to 5 - at the beginning and then close the errors once we verify that parameters are set correctly. This will be value we play with later…

Particle model

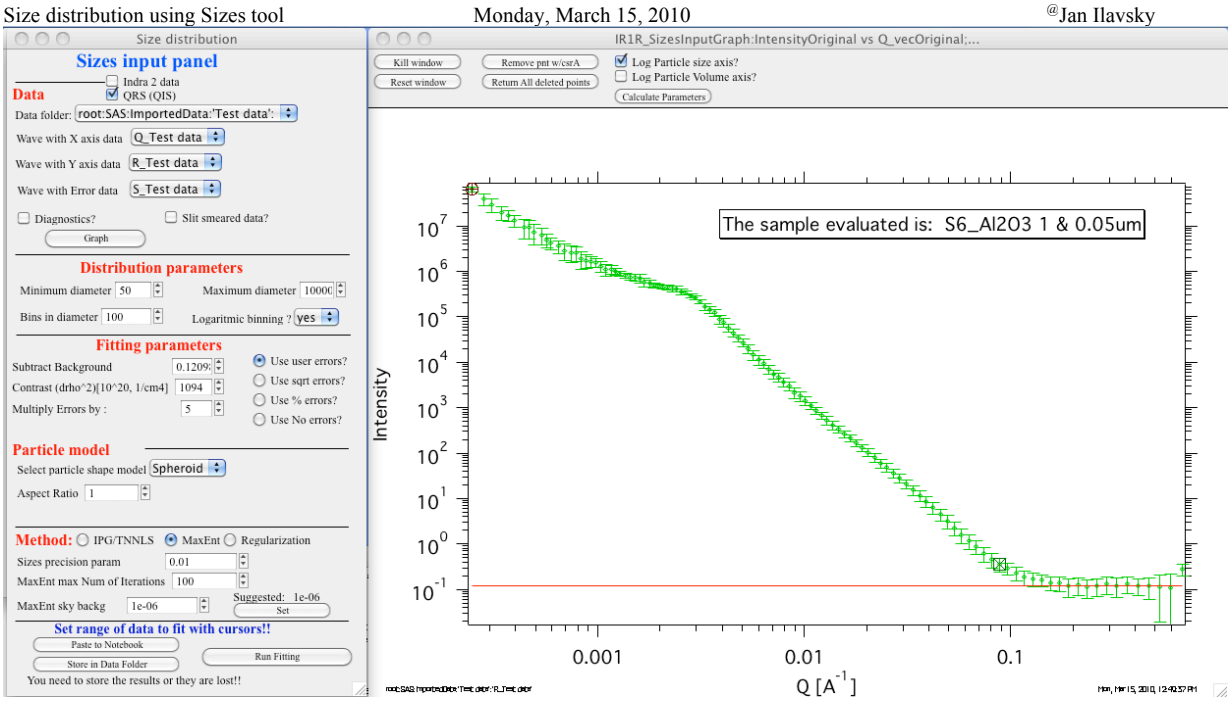
There are many form factors available as particle shapes. But most time spheres are appropriate. Spheres are spheroids with aspect ratio 1…

Method

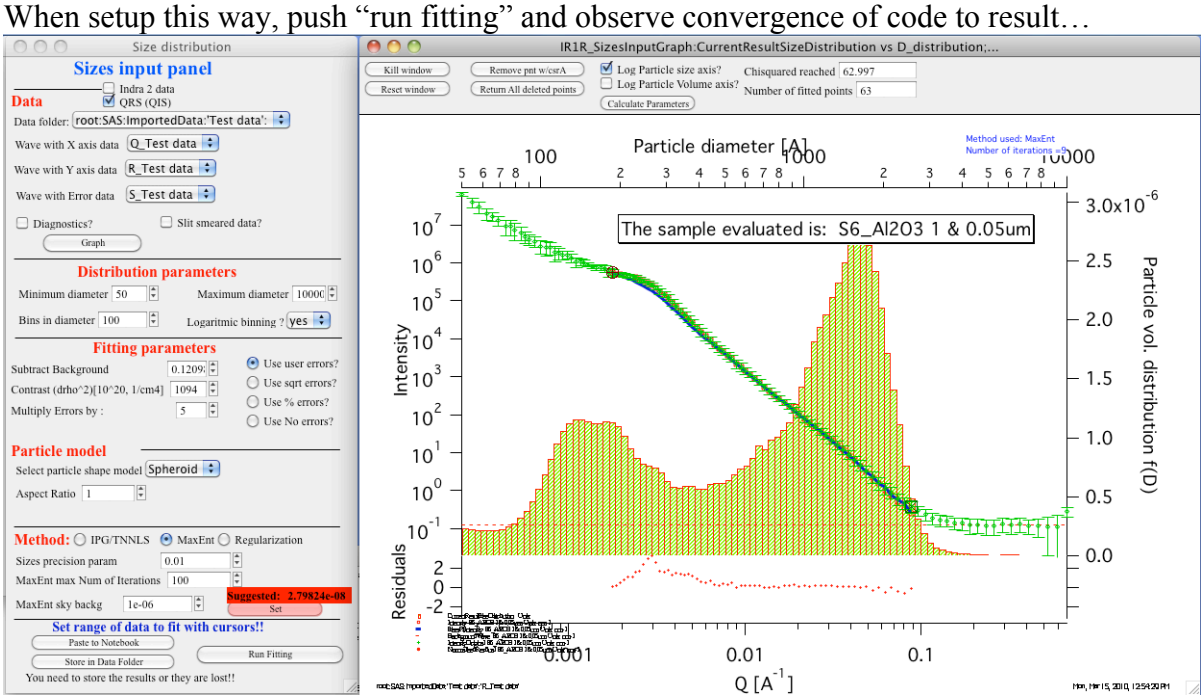
I suggest using either Maxent (default) or IPG/TNNLS method (Internal point gradient – total non-negative least square). We will go through both…

MaxEnt method

This is setup for Maxent method:



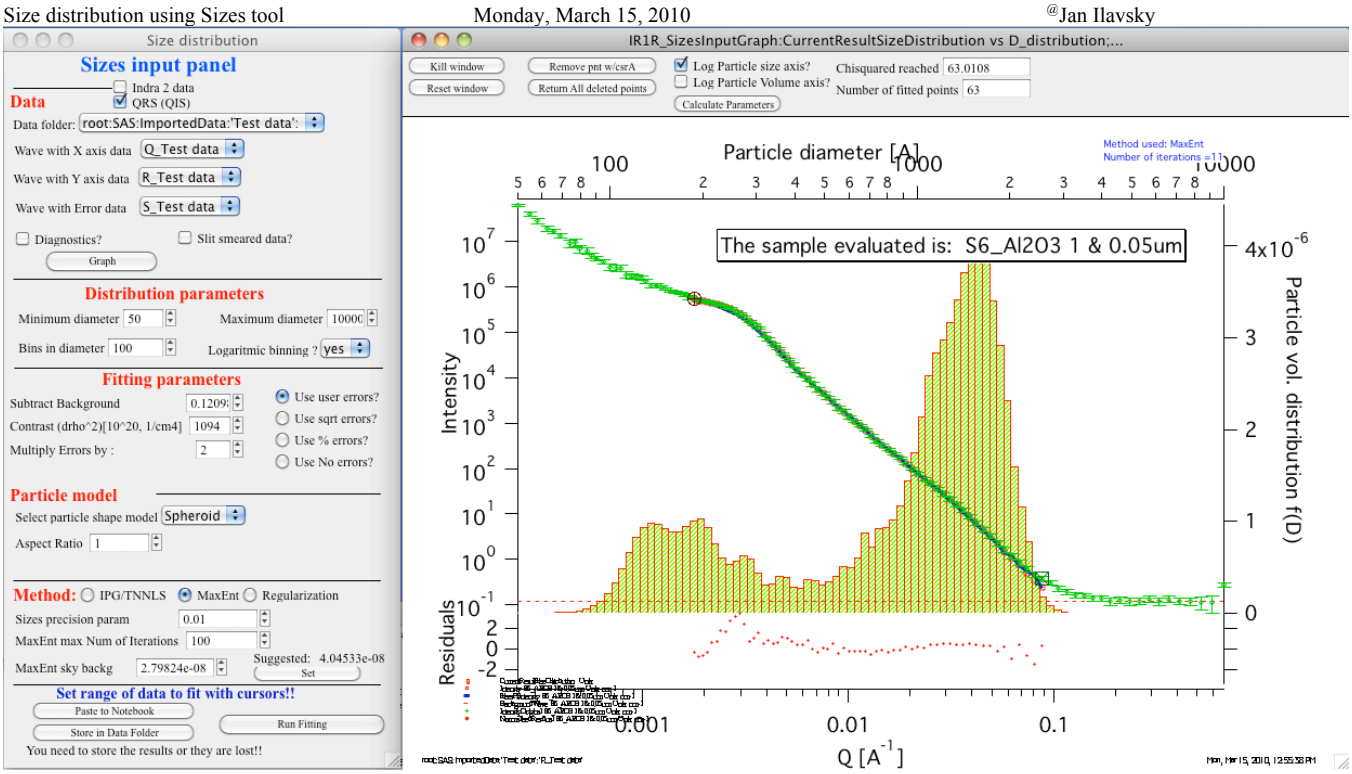
Set cursors on points 30 to 92 – points at smaller q are dominated by some really large stuff we cannot model with this method and points at higher q are dominated by background and do not bring in any more information. When setup this way, push "run fitting" and observe convergence of code to result…



Note few things:

There is red field "Suggested" at MaxEnt sky background. This parameter is internal MaxEnt parameter and under most conditions it should be set to suggested value. Some more details are in the manual of Irena… Push button "Set" to set it to suggested value.

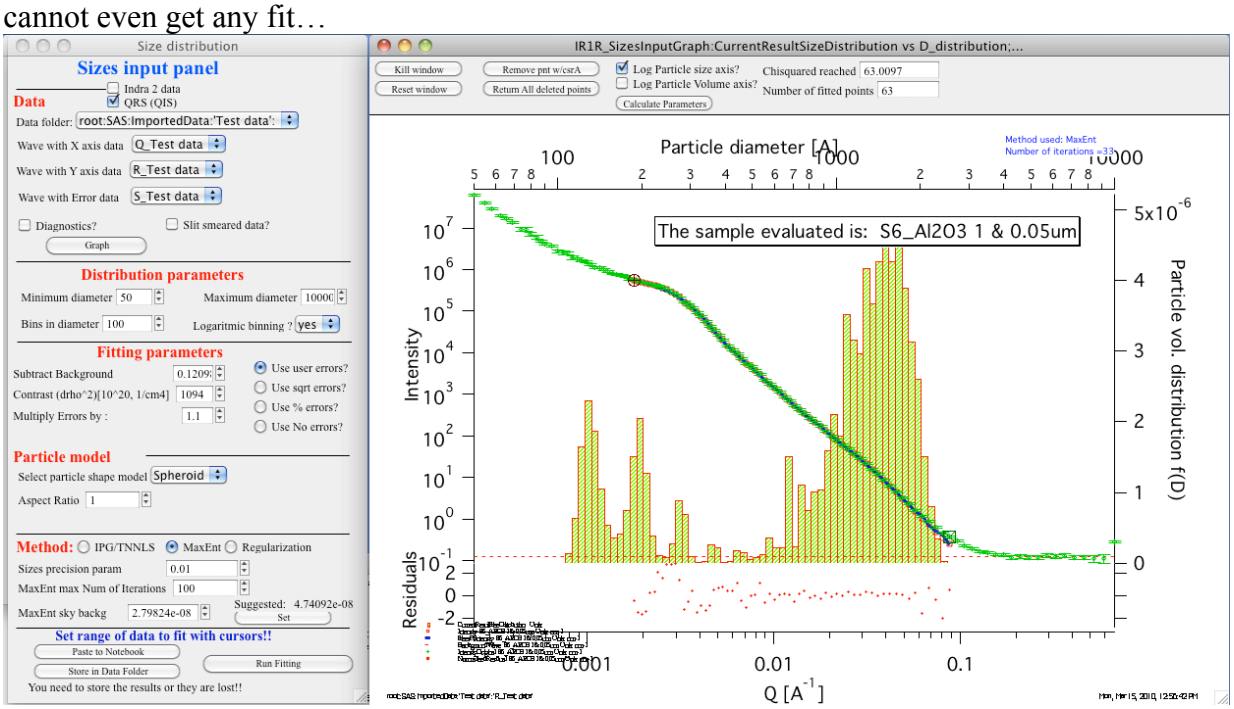
Note also that there is significant misfit between the data and model, since the blues line (fit result) is all time below the measured data. This is because we relaxed the errors by factor of 5. Let's lower that down to 2 and run fitting again.



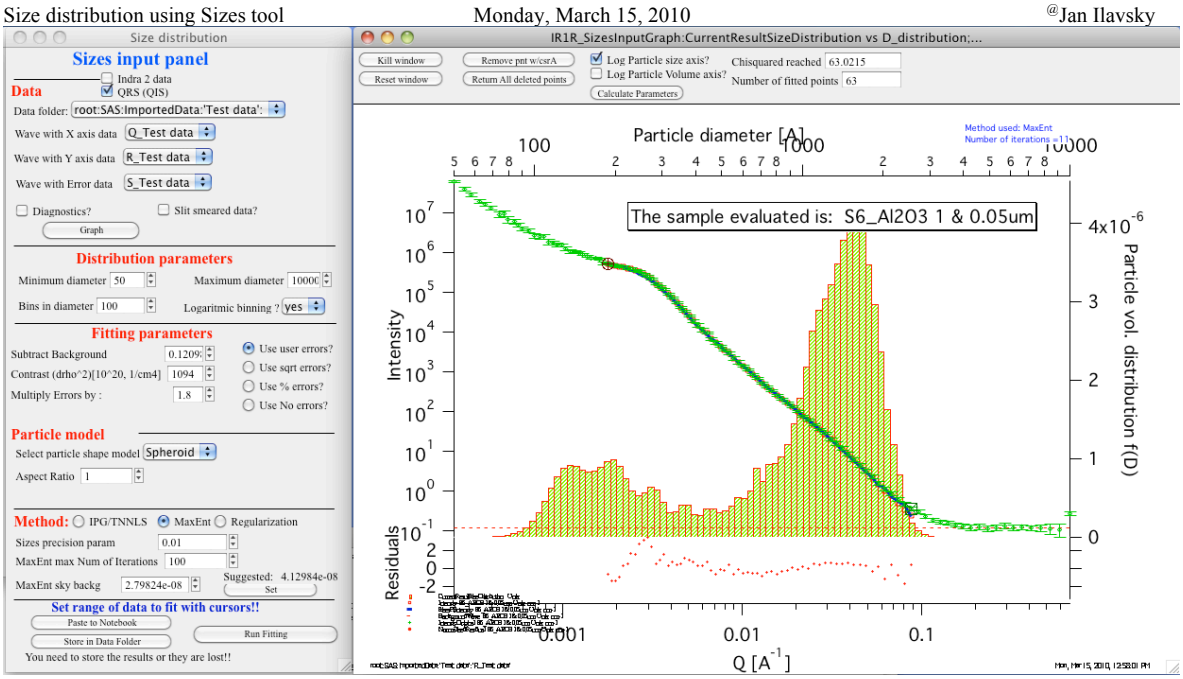
Now the fit is much closer and all seems well…

We can lower the error multiplier even more – we should reduce it as much as possible, while we reach solution quickly and it is smooth. When the solution starts being noisy, we start fitting noise in the data, not the underlying material science problem.

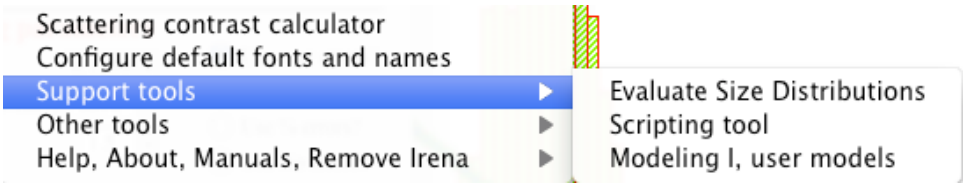
Note, that when we set errors multiplier to 1.1, the result starts getting really noisy and at lower values you cannot even get any fit…



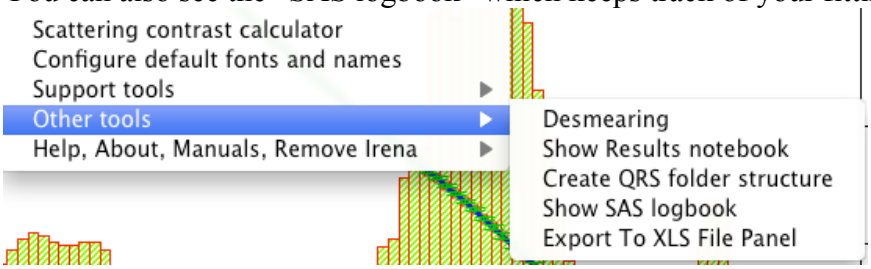
At 1.8 we get reasonable result:



Smooth distribution, reasonable result –note, that this material is clearly composed of two populations but not 50nm and 1 micron in size – one of the populations seems to be about 100 - 200Å in diameter and the other is 1000 – 2000 Å in diameter. To save data push "Store in data folder"… Results will be stored – Intensity/Q fit as well as volume and number size distributions for future use, ploting, calculations in "Evaluate Size distributions" etc.:



You can also see the "SAS logbook" which keeps track of your fitting results



SAS_FitLog: Irena Log

Normal Inches

This is log results of processing SAS data with Irena package.

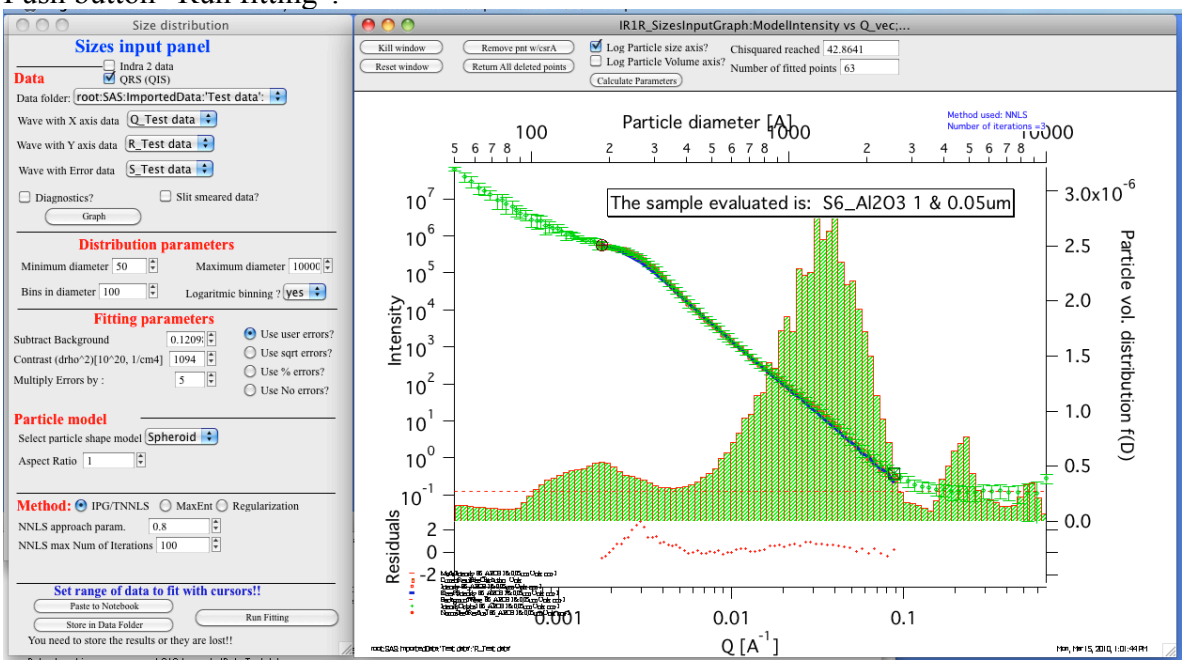
```
3/15/10,  12:43 PM

*************************************************
*************************************************
Data load record
3/15/10,  12:43 PM
File path and file name                 Prg HD:Users:ilavsky:Desktop:Irena and Nika
Handouts:version 2010:DVD files:Irena Example Data:Test data.dat

Loaded on                                       Mon, Mar 15, 2010    12:43:40 PM
Data stored in :                                root:SAS:ImportedData:Test data:
New waves named (Int,q,error) :         R_Test data Q_Test data  S_Test data
Comments and processing:
Q was in A

*************************************************
*************************************************
Sizes fitting record
3/15/10,  1:09 PM
Input data names
                                        Folder
                                root:SAS:ImportedData:'Test data':
                                Intensity/Q/error wave names  'R_Test data'             'Q_Test
data'                           'S_Test data'
                                SizesDataFrom=root:SAS:ImportedData:'Test data':
                                 SizesIntensity='R_Test data'
                                 SizesQvector='Q_Test data'
                                   SizesError='S_Test data'
                                MaxEntSkyBckg=2.7982e-08
                                      MaxEntRegular=1
                                    MaxsasNumIter=100
                                     numOfPoints=100
                                    SlitLength=0.03865
                                          Rmin=25
                                         Rmax=5000
                                        Bckg=0.12099
                                 ScatteringContrast=1094
                                          Dmin=50
                                         Dmax=10000
                                    ErrorsMultiplier=1.5
                                TicksForDiagnostics=8.5899e+06
                                 MaxEntStabilityParam=0.01
                                     NumberIterations=6
                                      MaxEntNumIter=0
                                        AspectRatio=1
                                 FractalRadiusOfPriPart=0
                                   FractalDimension=10
                                     CylinderLength=0
                                       TubeLength=0
                                   TubeWallThickness=0
                                 TubeCoreContrastRatio=0
```

IPG/TNNLS

Most of the setup is same as for MaxEnt method. Change to IPG/TNNLS by selecting appropriate checkbox next to Method:.

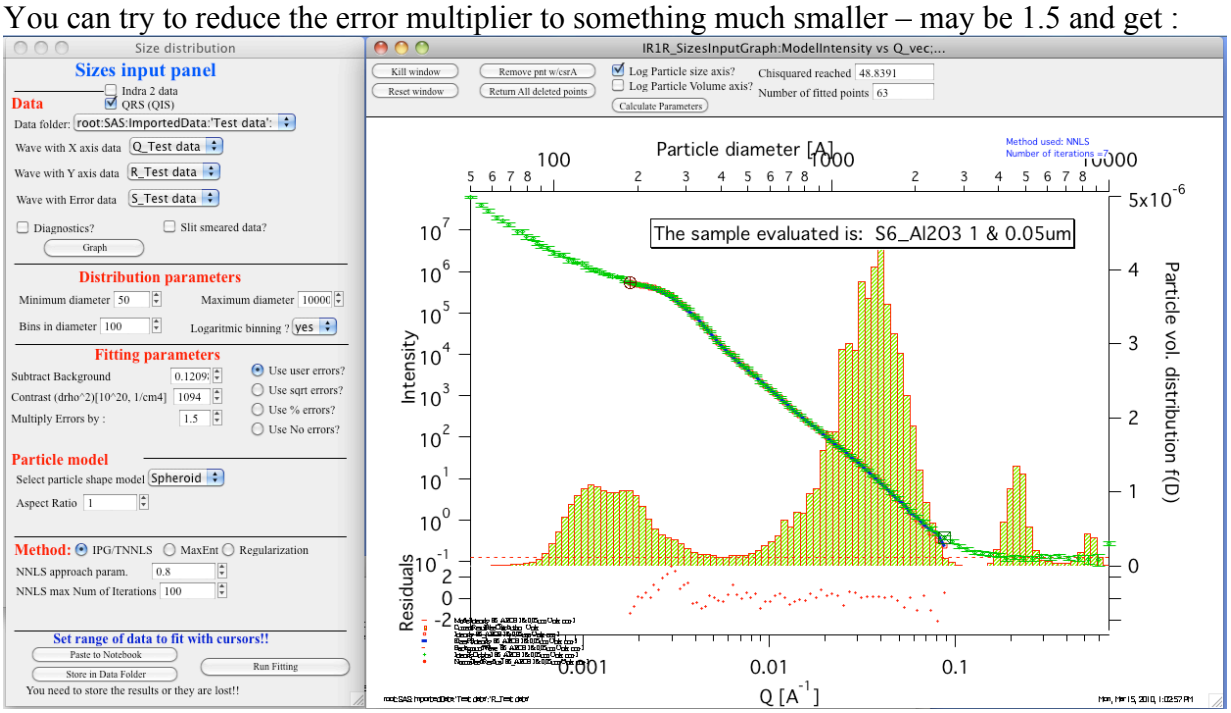
New and different parameters appear. Generally, these parameters should be appropriate for most problems. Note, that there may be different appropriate error multiplier needed to get a solution. Change to 5 again to start with…

Push button "Run fitting".



The code converges quickly to solution above. Note, that in this case we see many peaks – physically, the first two peaks are the result, the peaks at larger sizes are some "ringing peaks" which are artifact of the form factor and internal Bessel functions.

You can try to reduce the error multiplier to something much smaller – may be 1.5 and get :



It may be difficult to get rid of these ringing peaks – this method may not be the best one for this sample…

Note, that the real results (peaks at 100-200A and 1000 – 2000 A) are very similar for both methods.

Comments:

Success of any appropriate method is not easy to predict. If IPG/TNNLS methods works, it provides smoother results, which is probably more correct. The MaxEnt does not have some artifacts of the IPG method, but often produces more noisy results. By comparing both and picking the better one, you can choose.