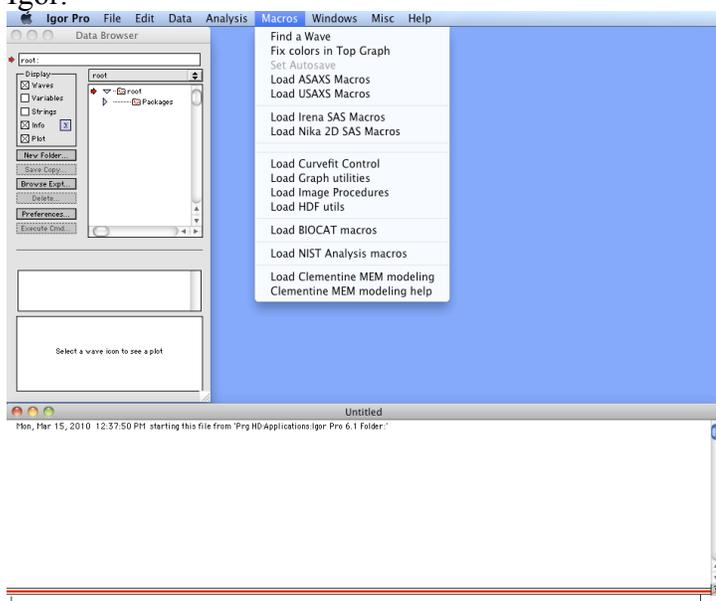


## Unified fit tool in Irena package

*This handout describes how SAS data (example using provided data from USAXS instrument) can be fitted using Unified fit tool in Irena and further, how log-normal size distribution of spheres can be extracted from the results. 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.*

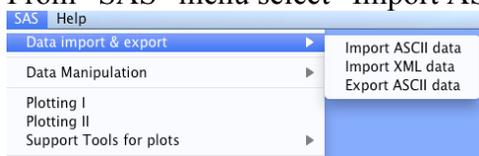
### Start Igor, load macros

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”.

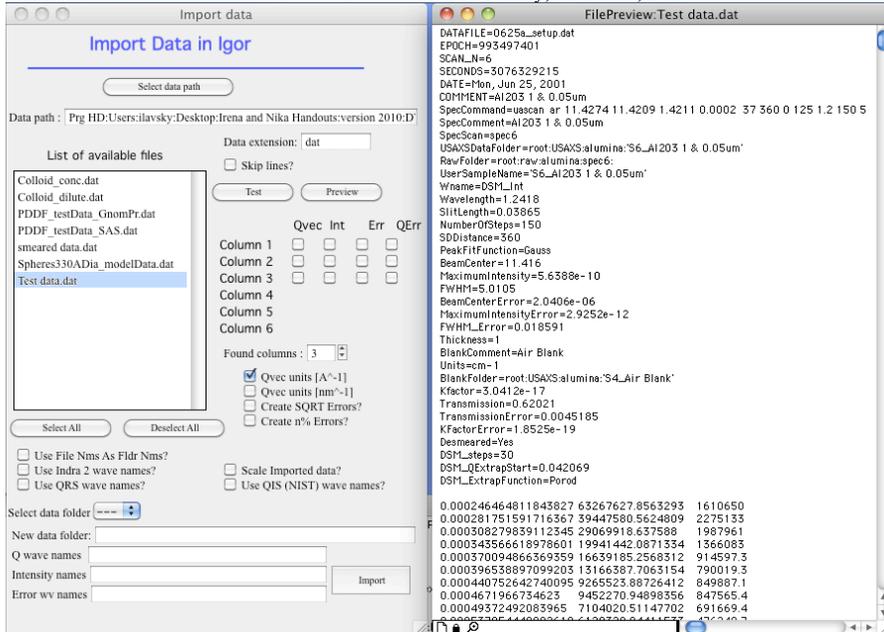


This will create new panel. Push the button “Select data path” and navigate to folder, where are the data.

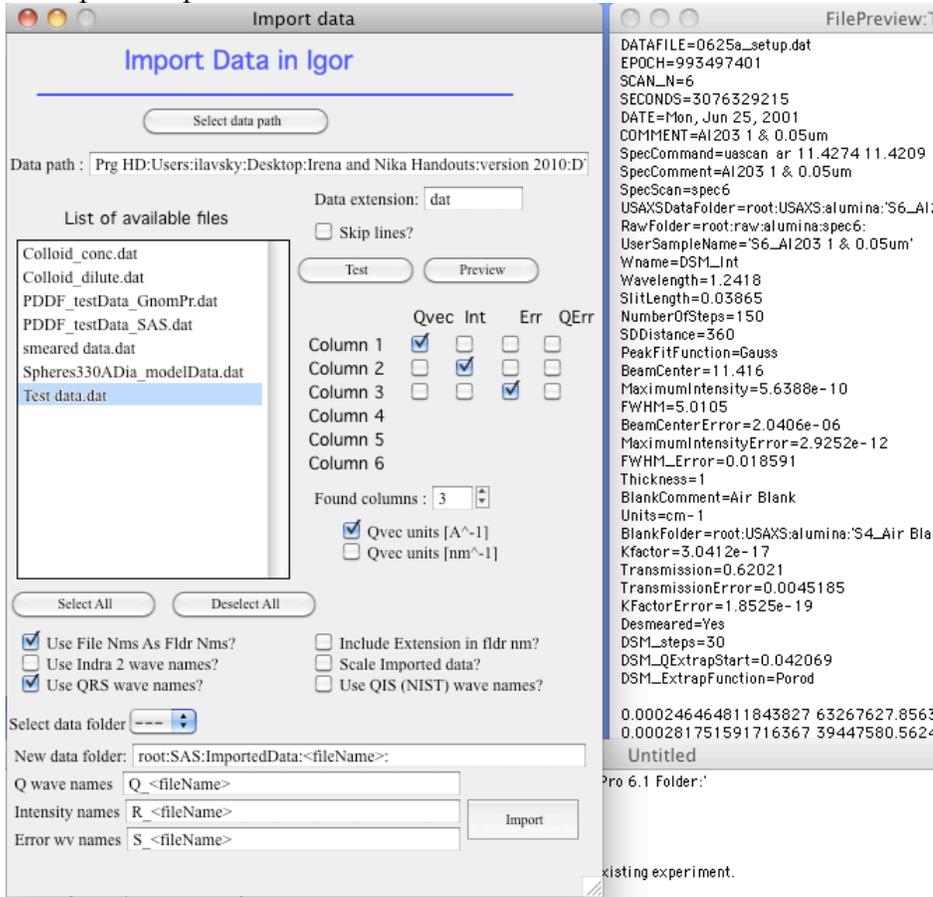


The test data provided (Test data.dat) have extension dat, we can put this in the “Data extension” field and only files with this extension will show.

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 Nms?” and “Use QRS wave names”. Select the file with data (Test data.dat). Then push Import button.



```

●IR11_ImportDataMain()
path: "Prg HD:Users:ilavsky:Desktop:Irena and Nika Handouts:version 2010:DVD files:Irena Example Data:"
Imported data from :Prg HD:Users:ilavsky:Desktop:Irena and Nika Handouts:version 2010:DVD files:Irena Example Data:Test data.dat
Data stored in : root:SAS:ImportedData:Test data:
New Wave names are : R_Test data Q_Test data S_Test data
Imported 1 data file(s) in total
  
```

This will import the data into Igor – see below:

root.Packages.ImportData:

Display

- Waves
- Variables
- Strings
- Info
- Plot

New Folder...  
Save Copy...  
Browse Expt...  
Delete...  
Preferences...  
Execute Cmd...

root

- root
  - Packages
    - SAS
      - ImportedData
        - Test data
          - 0\_Test data
            - 2\_Test data
              - S\_Test data

Rows: 112 Units: None Start: 0 Delta: 1

Notes: Data imported from folder=Prig HD:Users:ilavsky.Desktop:Irena and Nika Handouts:version 2010.DVD files:Irena Example Data: Data file name=Test data.dat:DATAFILE=0625a\_setup.dat:EPOCH=993497401:SCAN\_N=6:SECONDS=3076329215:DATE=Mon, Jun 25, 2001:COMMENT=Al2O3 1 & 0.05um:SpecCommand=uascan ar 11.4274 11.4209 1.4211 0.0002 37 360 0 125 1.2 150 5:SpecComment=Al2O3 1 & 0.05um:SpecScan=spec6:USAXSDataFolder=root:USAXS:alumina.S6\_Al2O3 1 & 0.05um:RawFolder=root:raw:alumina:spec6:UsesSampleName=56\_Al2O3 1 & 0.05um:Wname=DSM\_Int:Wavelength=1.2418:SlitLength=0.03865:NumberOfSteps=150:SDDistance=360:PeakFitFunction=Gauss:BeamCenter=11.416:MaximumIntensity=5.6388e-10:FWHM=5.0105:BeamCenterError=2.0406e-06:MaximumIntensityError=2.9252e-12:FWHM\_Error=0.018591:Thickness=1:BlankComment=Air Blank:Units=cm-1:BlankFolder=root:USAXS:alumina.S4\_Air Blank:Kfactor=3.0412e-17:Transmission=0.62021:TransmissionError=0.0045185:KFactorError=1.8525e-19:Desmeared=Yes:DSM\_steps=30:DSM\_QExtrapStart=0.042069:DSM\_ExtrapFunction=Parod:.

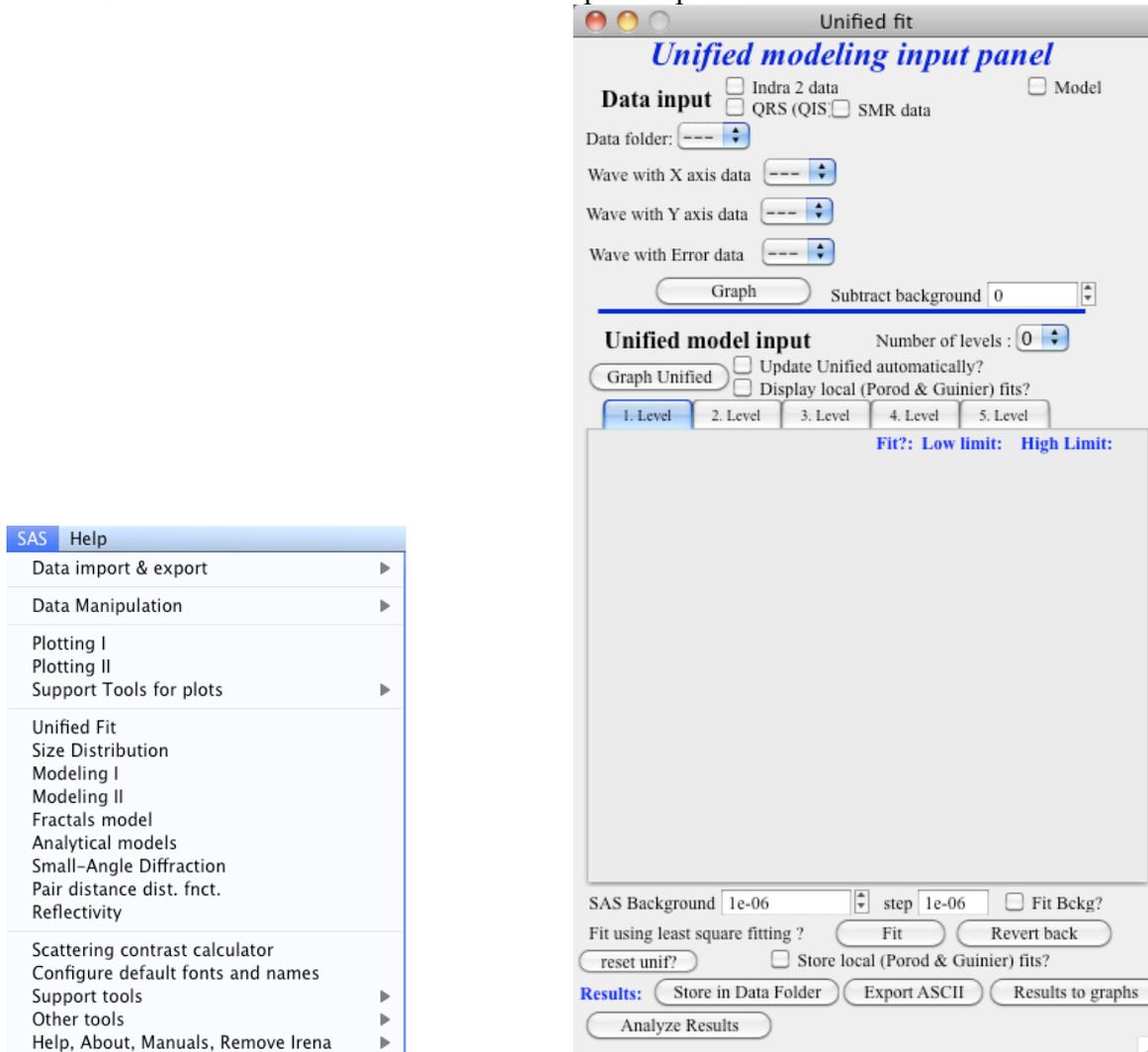
Just for information: Note, that the header from the ASCII file we had is now attached to the wave in so called wavenote – it is visible, when you select the wave in the Data browser and have the “info” checkbox checked. This is how Irena macros store additional information with waves – as wavenotes,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

## Using Unified Fit tool

### Setup

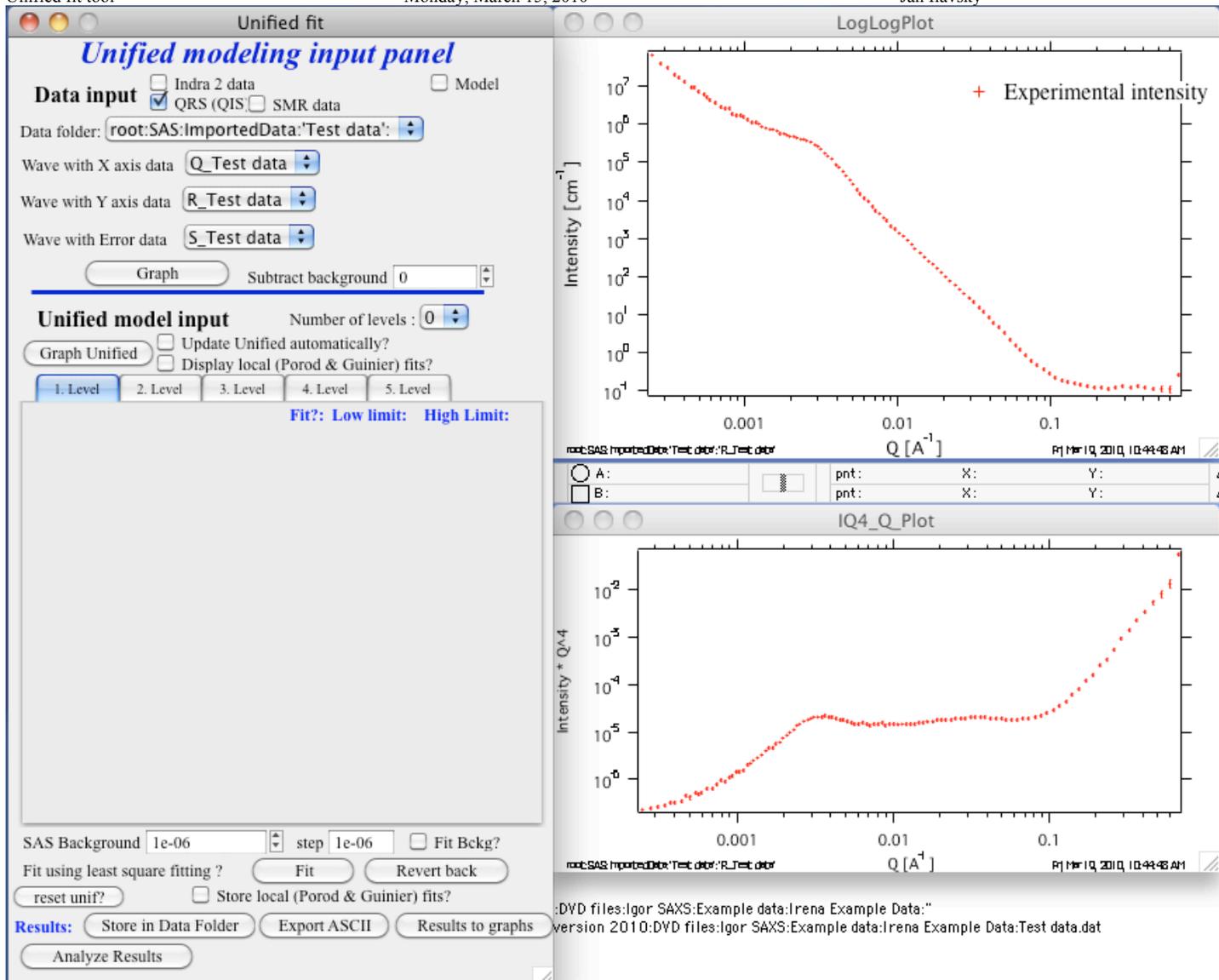
Close all import-related windows (Panel and Notebook).

From “SAS” menu select “Unified Fit”. New panel opens.



Select data: Check “QRS” data type, pull on “Data Folder” and select the one with our data, all should fill in automatically.

Next Push “Graph” button:



Next we need to start fitting the data themselves.

Note, that in Unified you need to compose each of the “levels” from high-Q to low-Q region. Basically, we MUST start from the small features and go to large ones.

Also, it is important to understand, if the features are independent features (like population of two size of powder here) or if these are scattering from the same population on different length scales (like agglomerates of small particles, which scatter as primary particles as well as agglomerates). This will be addressed later.

Now, let's start with the fine stuff... Select “Number of Levels “ to be 1, check the “Update Unified automatically” for convenience:

**Unified modeling input panel**

**Data input**  Indra 2 data  Model  
 QRS (QIS)  SMR data

Data folder:

Wave with X axis data:

Wave with Y axis data:

Wave with Error data:

Subtract background:

---

**Unified model input** Number of levels:

Update Unified automatically?  
 Display local (Porod & Guinier) fits?

1. Level 2. Level 3. Level 4. Level 5. Level

**Level 1 controls** Fit?: Low limit: High Limit:

G:

Rg:

Rg step:   G step:

Is this mass fractal from lower level? Surf / Vol:

B:

P:

P step:   B step:

RgCutoff:

k factor:

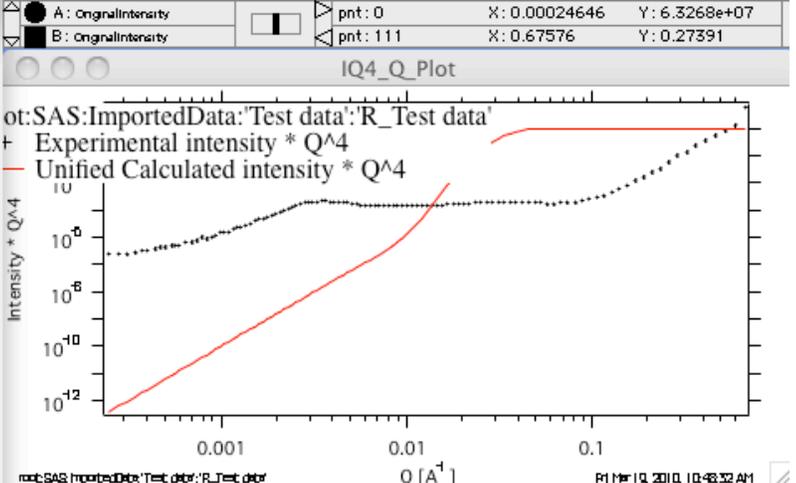
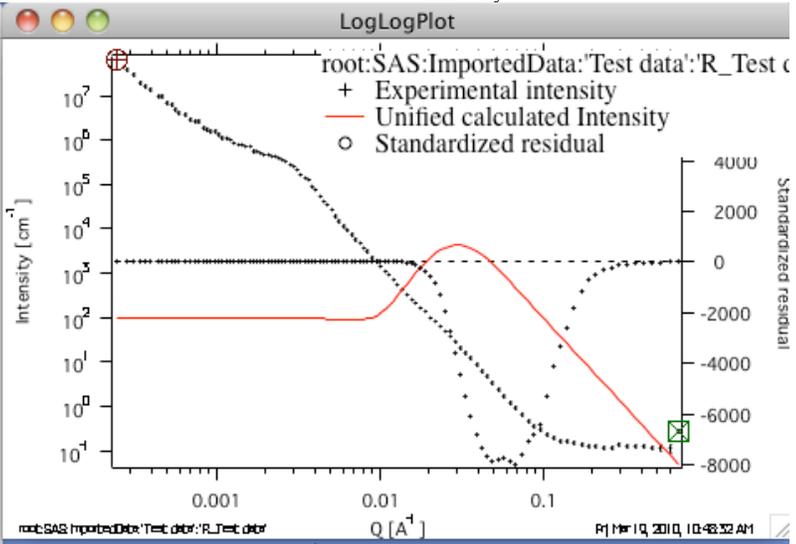
Is this correlated system?

SAS Background:   step:   Fit Bkg?

Fit using least square fitting?

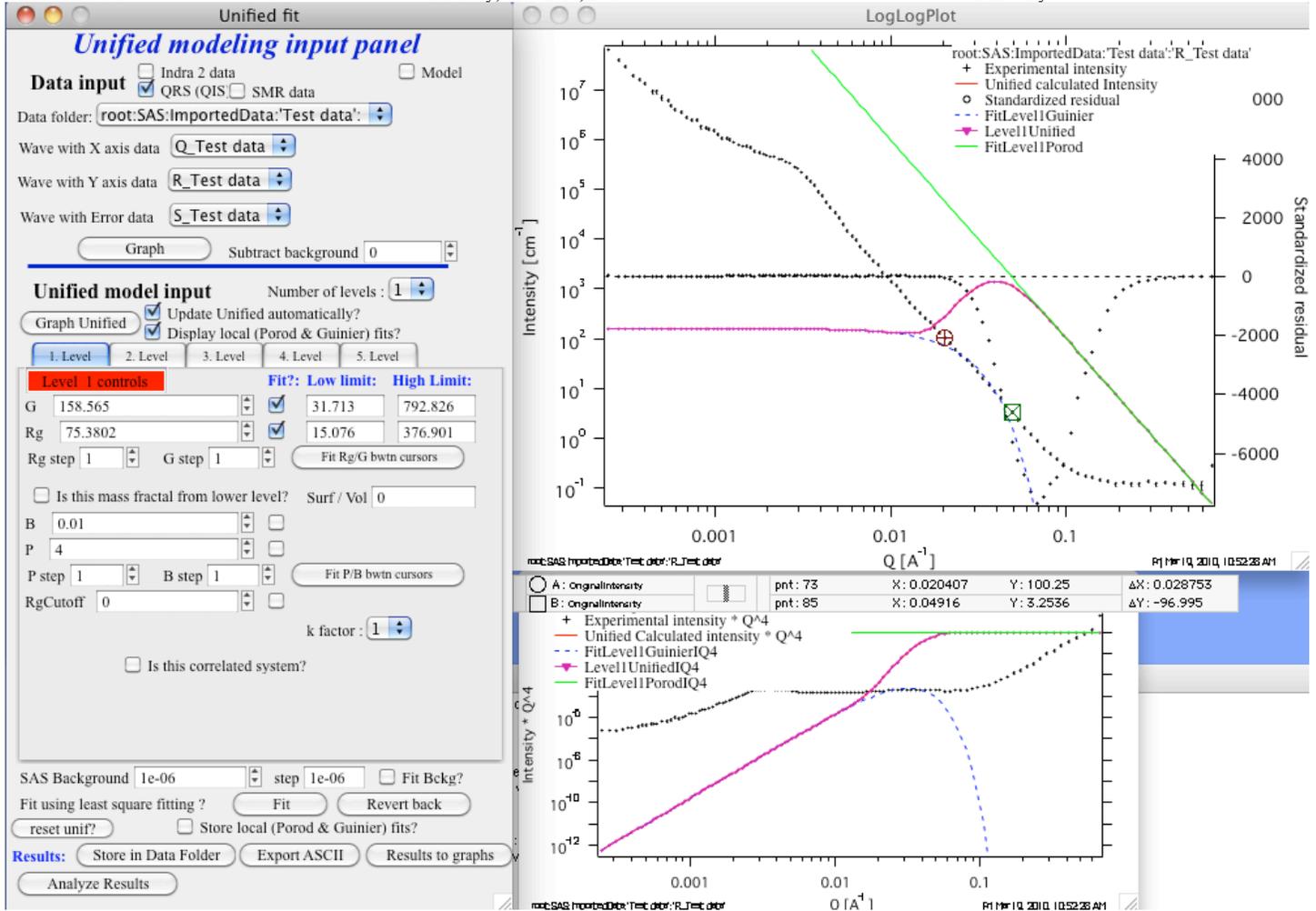
Store local (Porod & Guinier) fits?

**Results:**

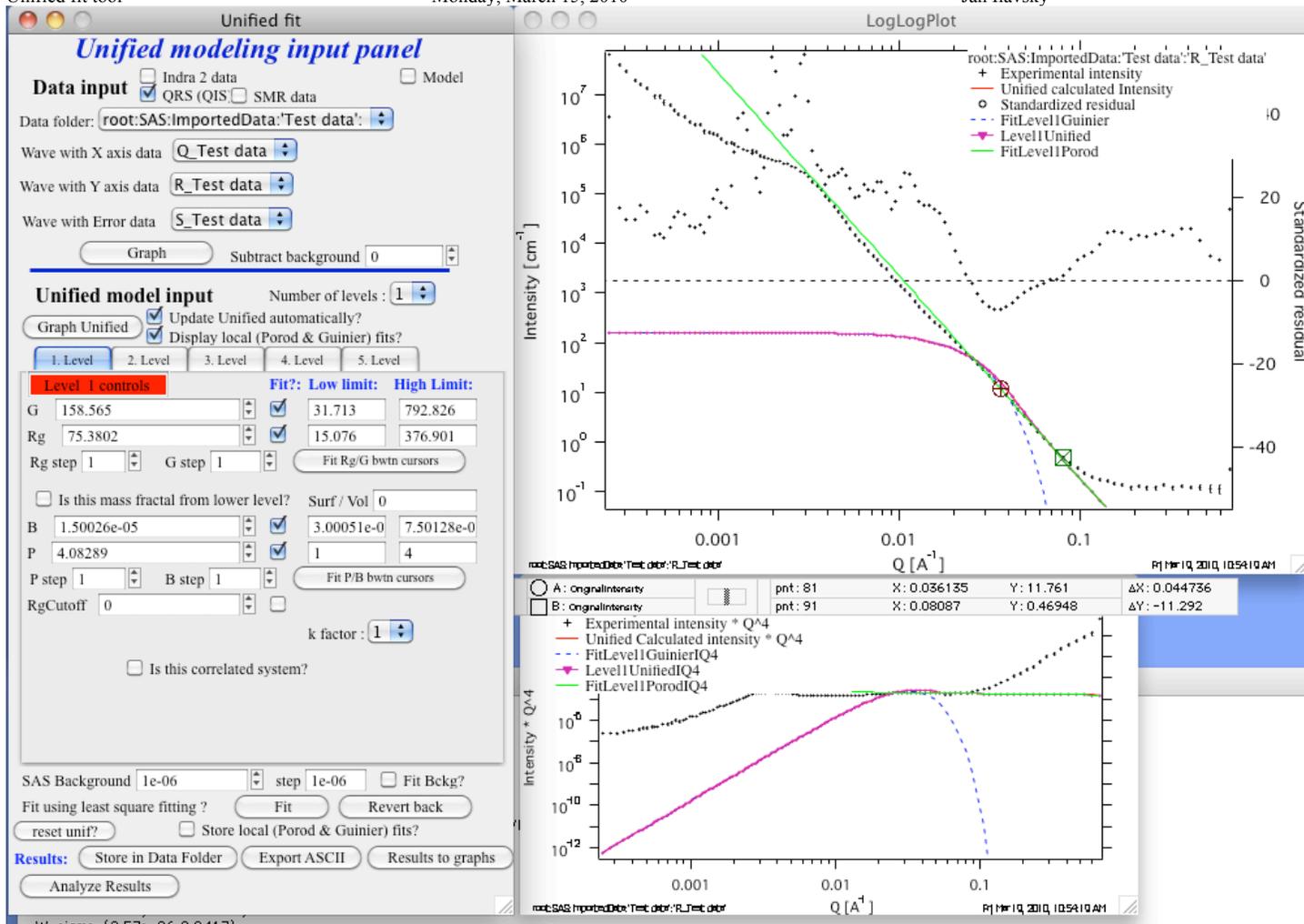


Comment: Note, the font in the legend is inappropriate for my screen here, you can use “Configure default fonts and names” in the SAS menu to change it. You need to graph again the data for these changes to take effect.

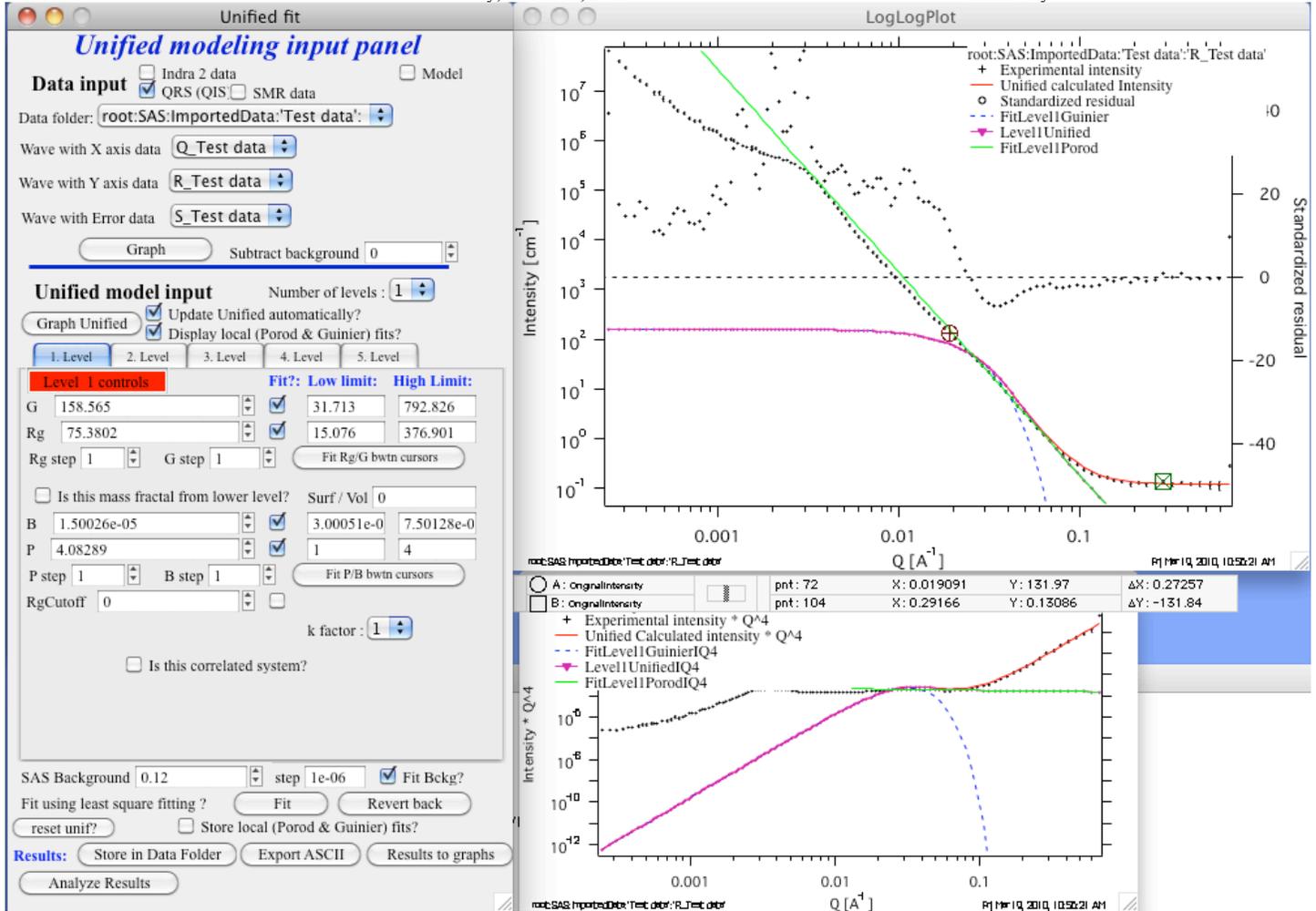
We need to fit now the Guinier area to the first “hump” around 0.03 Å<sup>-1</sup>. Put cursors on points (black +, measured data) around 73 and 85, select “Fit?” for Rg and G, and push button “Fit Rg/G bwn cursors”:



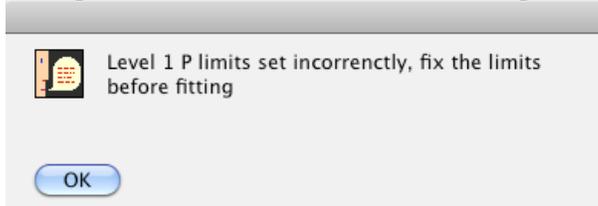
Next we need to fit power law slope. Move cursors to points around 81 and 91, select “Fit?” for B and P, and push Fit P/B bwtwn cursors” button:



Now we can fit whole 1<sup>st</sup> level, to do this move cursors to points around 72 and 104, keep all of the “Fit?” checkboxes selected. Also, let’s add some flat background in the “SAS Background” field and check “Fit Bckg?” checkbox. Background around 0.12 should be good starting value:



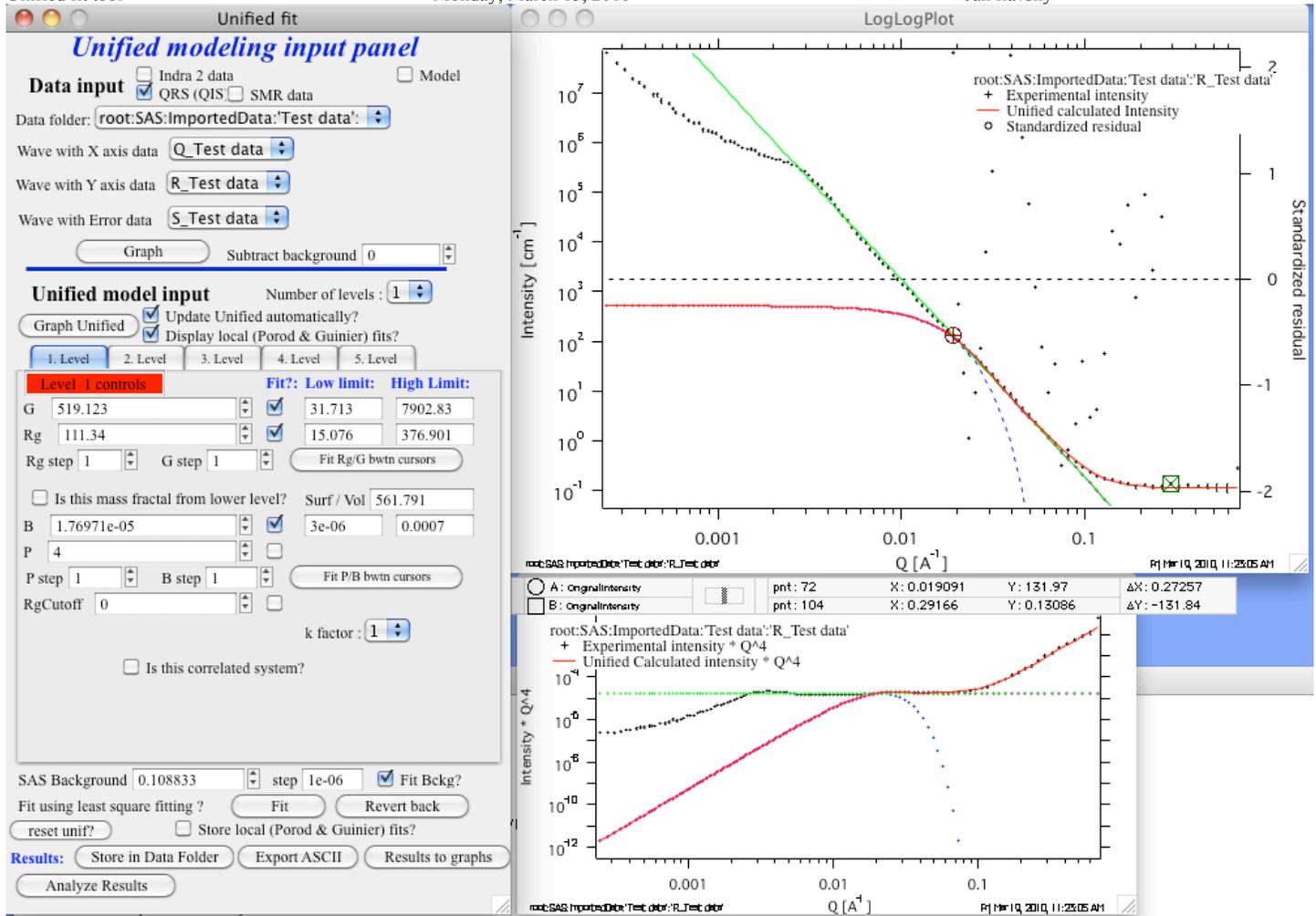
Now push the button “Fit” and here is a problem:



The code is checking, what the fitting limits are and we have starting value outside current fitting limits. The fitting limits are not enforced when we use “Fit Rg/G” and “Fit B/P” buttons, But when we use global “Fit” button, this can happen.

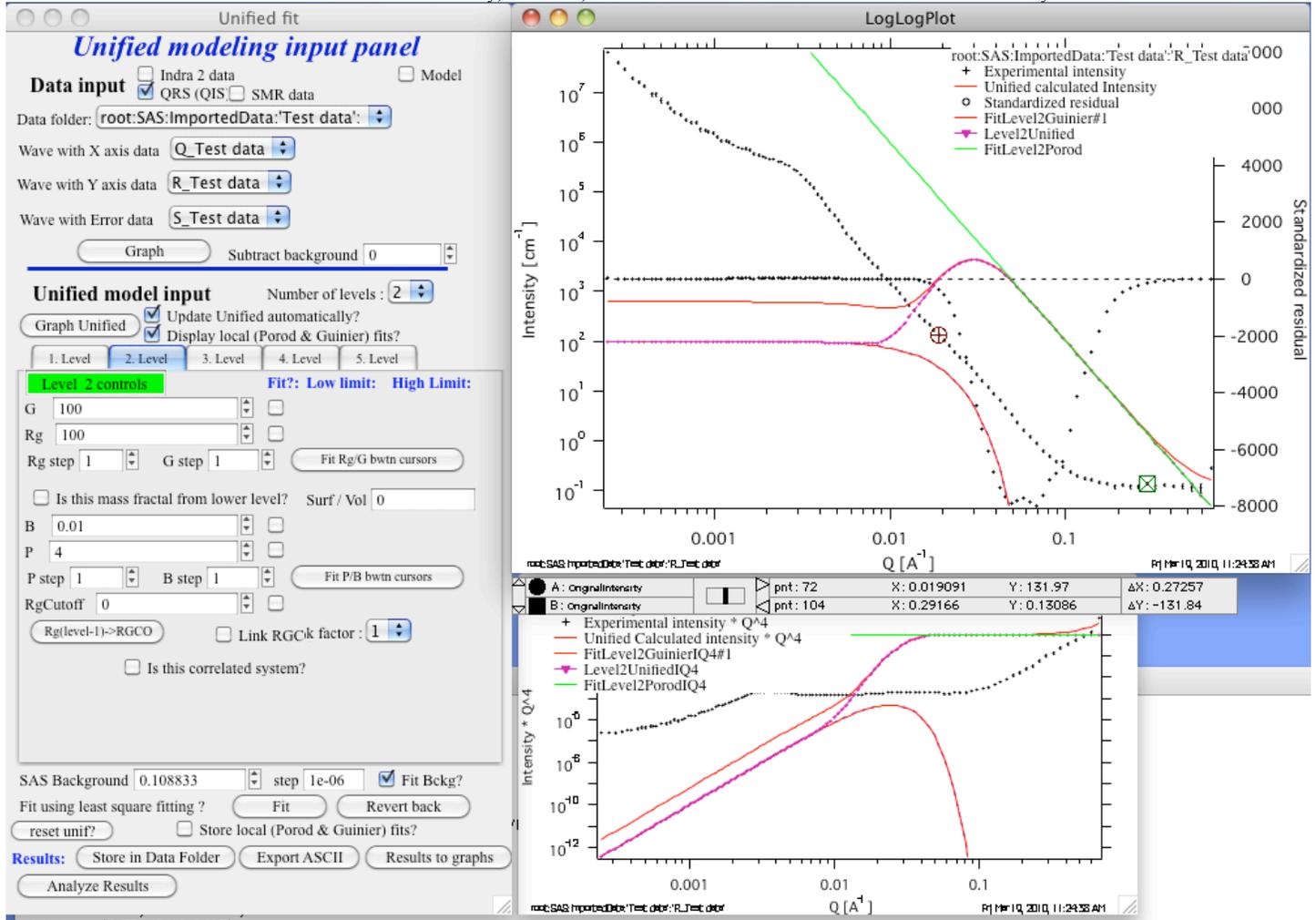
Note, that the current value for P is over 4 – about 4.08. That is not physical since 4 is smooth particles, above 4 we would have to have diffuse interface and that is not likely. In this case we have inter relationship between Porod’s power law slope and background, which are coupled. I suggest we set P to 4 and uncheck the “Fit?” checkbox next to it.

Change fitting limits as necessary and then push the fir button again.

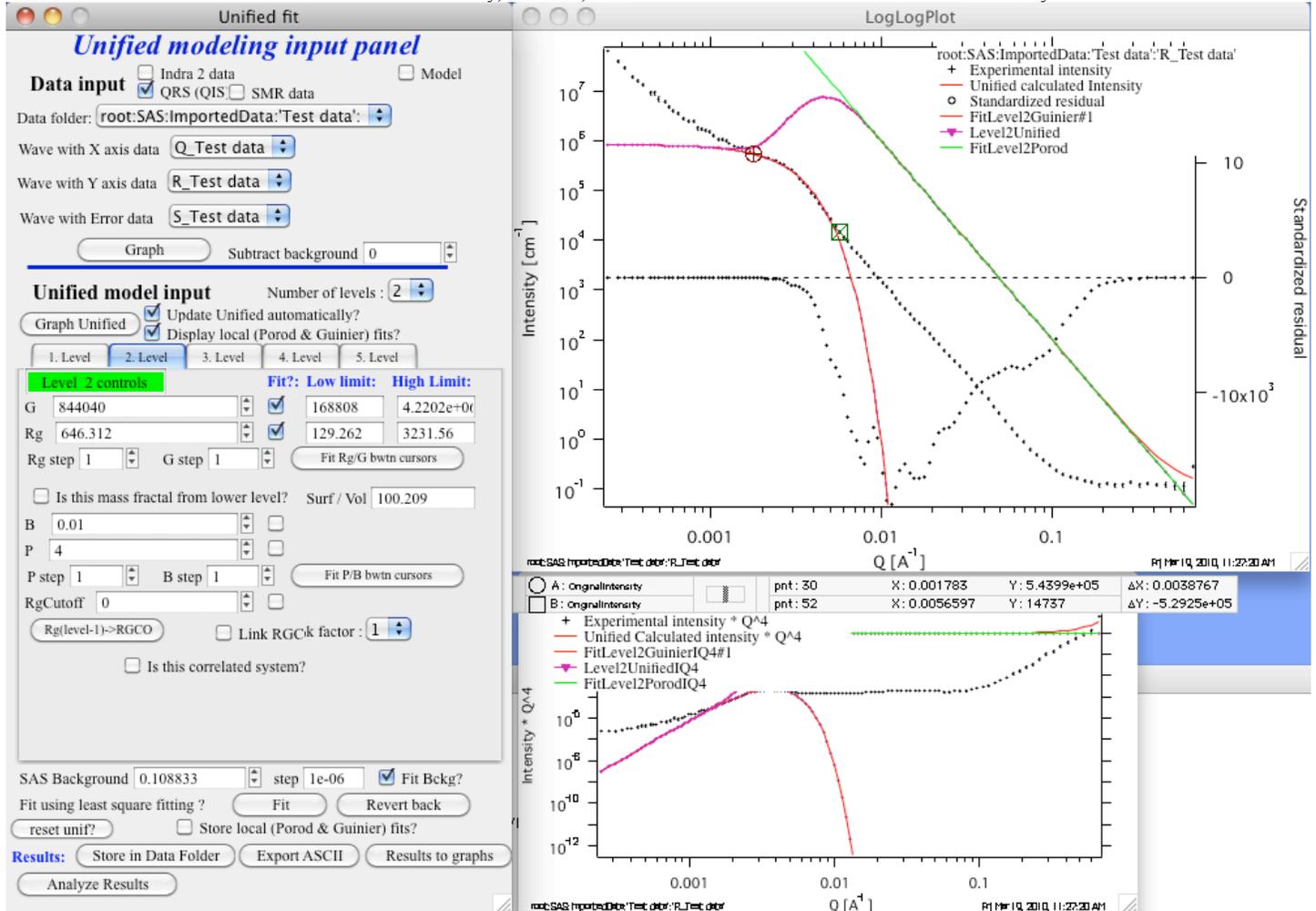


That looks good for the fine particles.

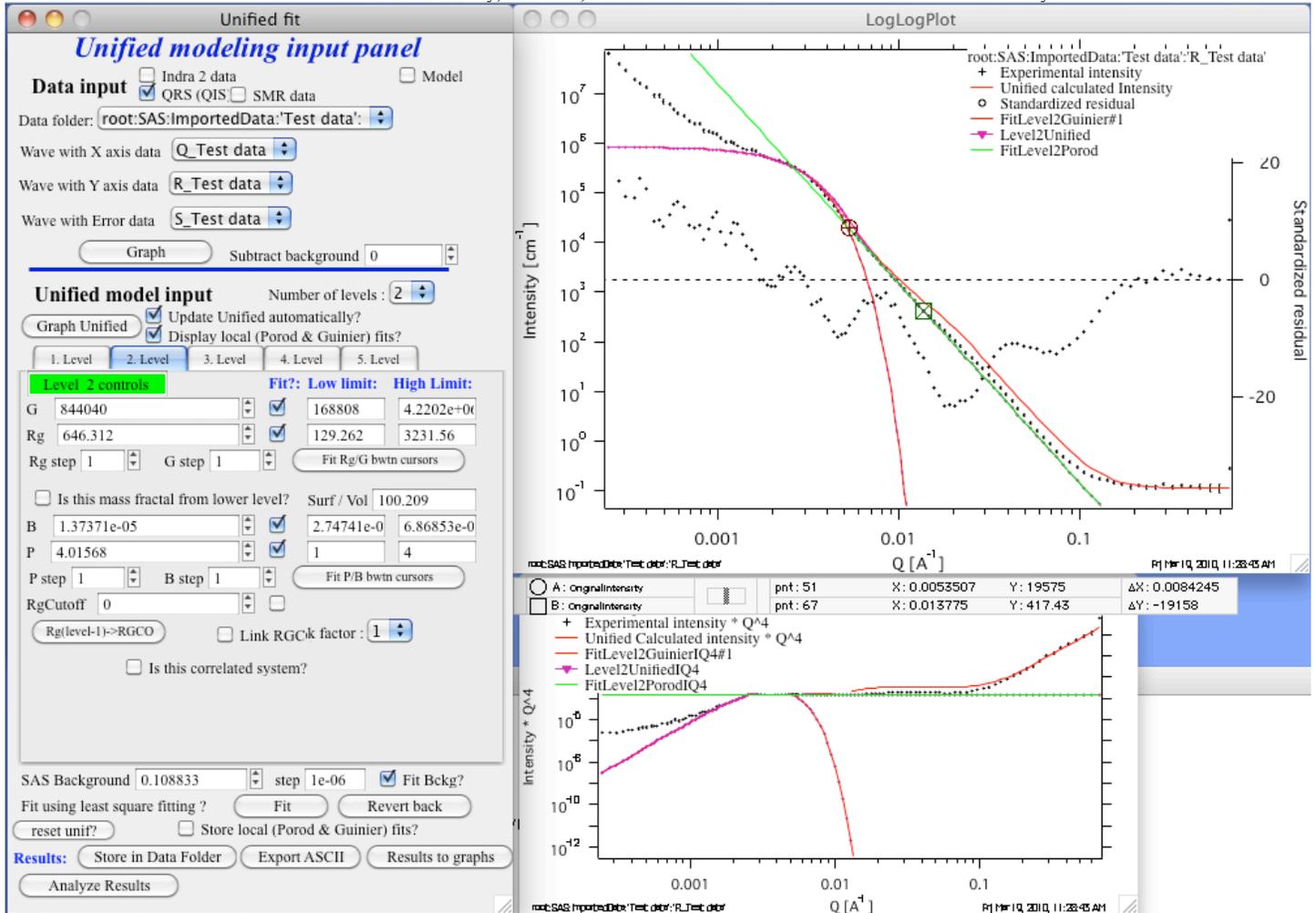
Next we need to model the larger particles.  
 Select "Number of levels" as 2 and click on second tab:



Now we need to fit the Rg/G again, but in order for the fit to work we need to move starting conditions closer. Therefore, we need to increase G to about 10<sup>6</sup> and Rg to may be 1000. Then we can select with cursors points about 30 to 52, select "Fit?" next to G and Rg, and push "Fit Rg/G" button:

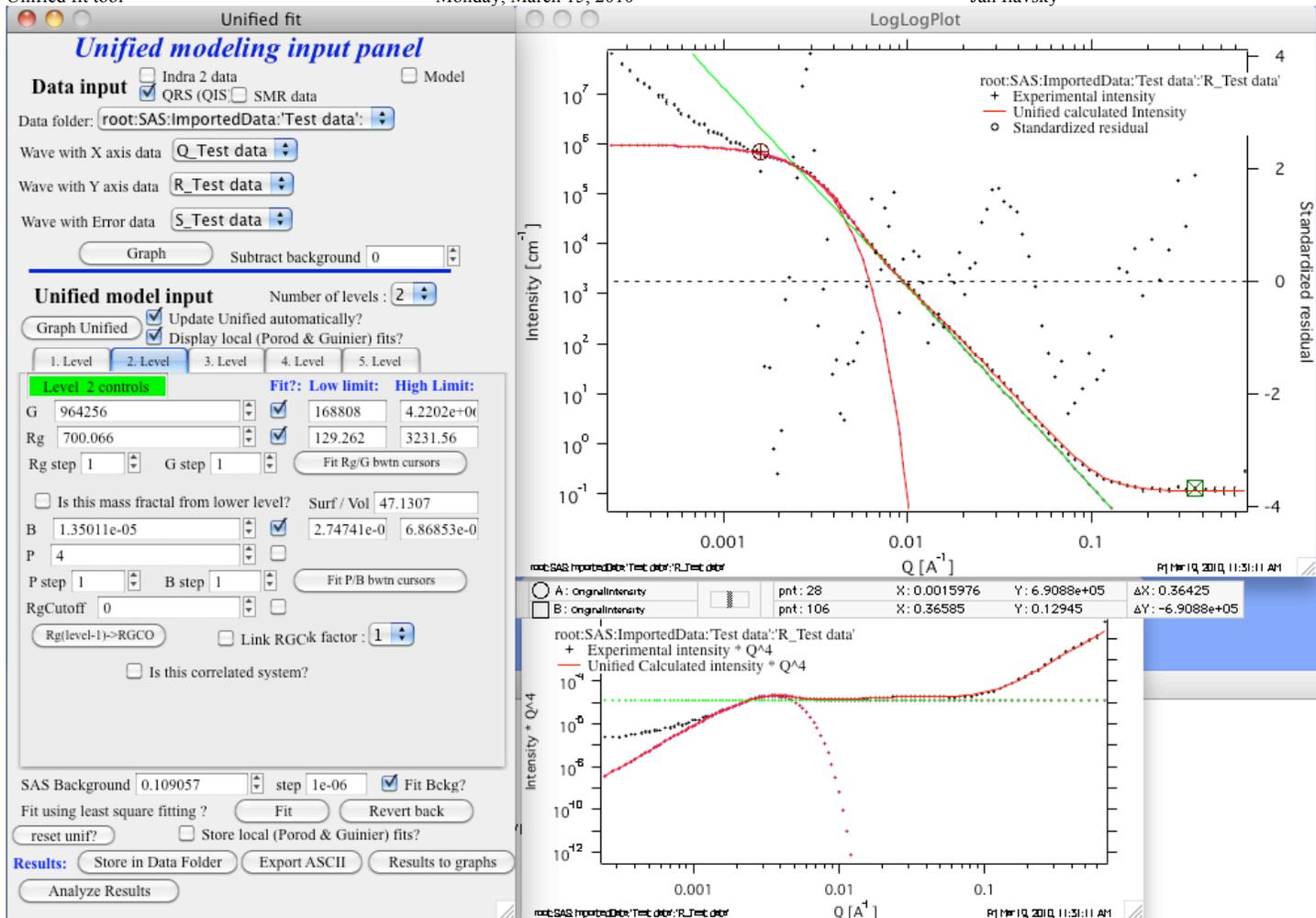


Now we need to fit Power law slope, so check the checkboxes "Fit?" next to B and P. select with cursors range may be 51 to 67 or so. The "Fit P/B".



Now we need to optimize all parameters at once. Note, that the P is about 4 – and that is correct, these are relatively smooth particles with sharp interfaces. To reduce number of fitting parameters, we can set for this level the P also to 4 and uncheck the “Fit?” checkbox.

Now, select with cursors points about 28 to 106 and push button “Fit”.

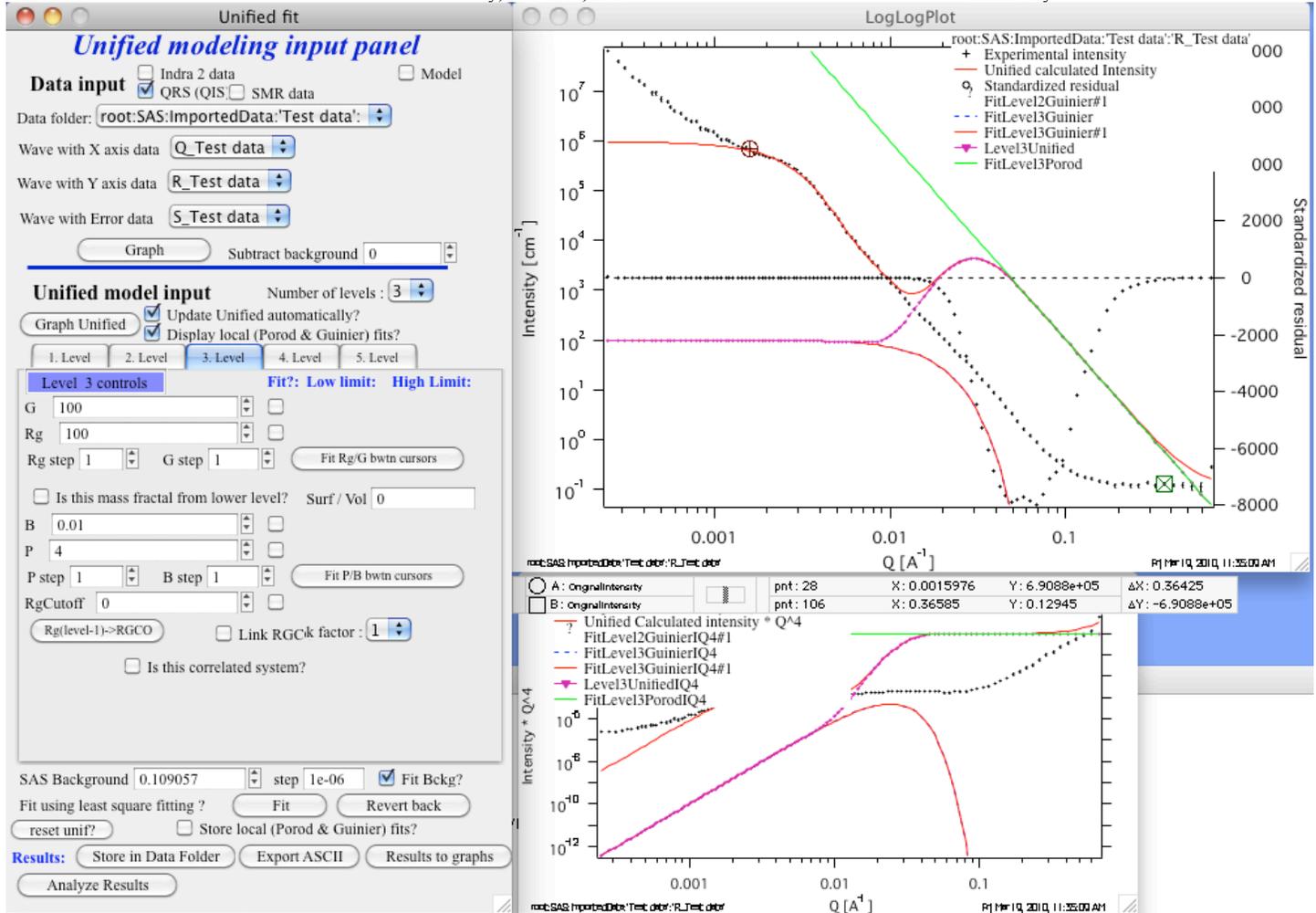


Next we need to decide what to do with the low-Q power law slope. There are many things this can be, but let's assume, this is agglomeration of the large Alumina particles.

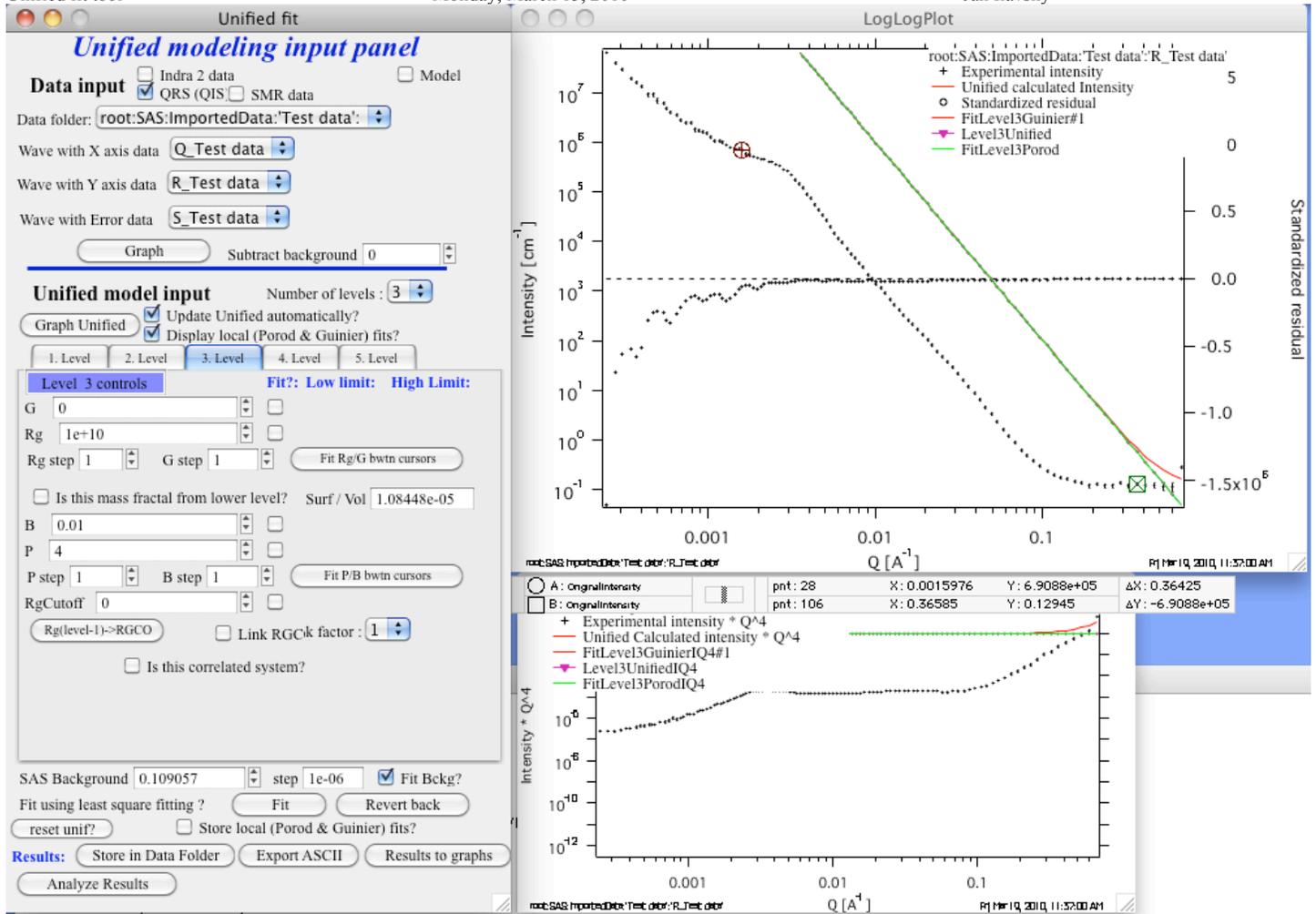
In this case there are some more things one needs to realize:

If this is agglomeration of particles with Rg about 700A (the large Alumina particles, Level 2), then it's scattering needs to end at around Rg~700. For this Unified contains parameter called "Rg cut off" (RGCO). Since we do not see the Guinier area for this population (too large, it would be at too small Q values for what we measured), we can model ONLY the power law slope.

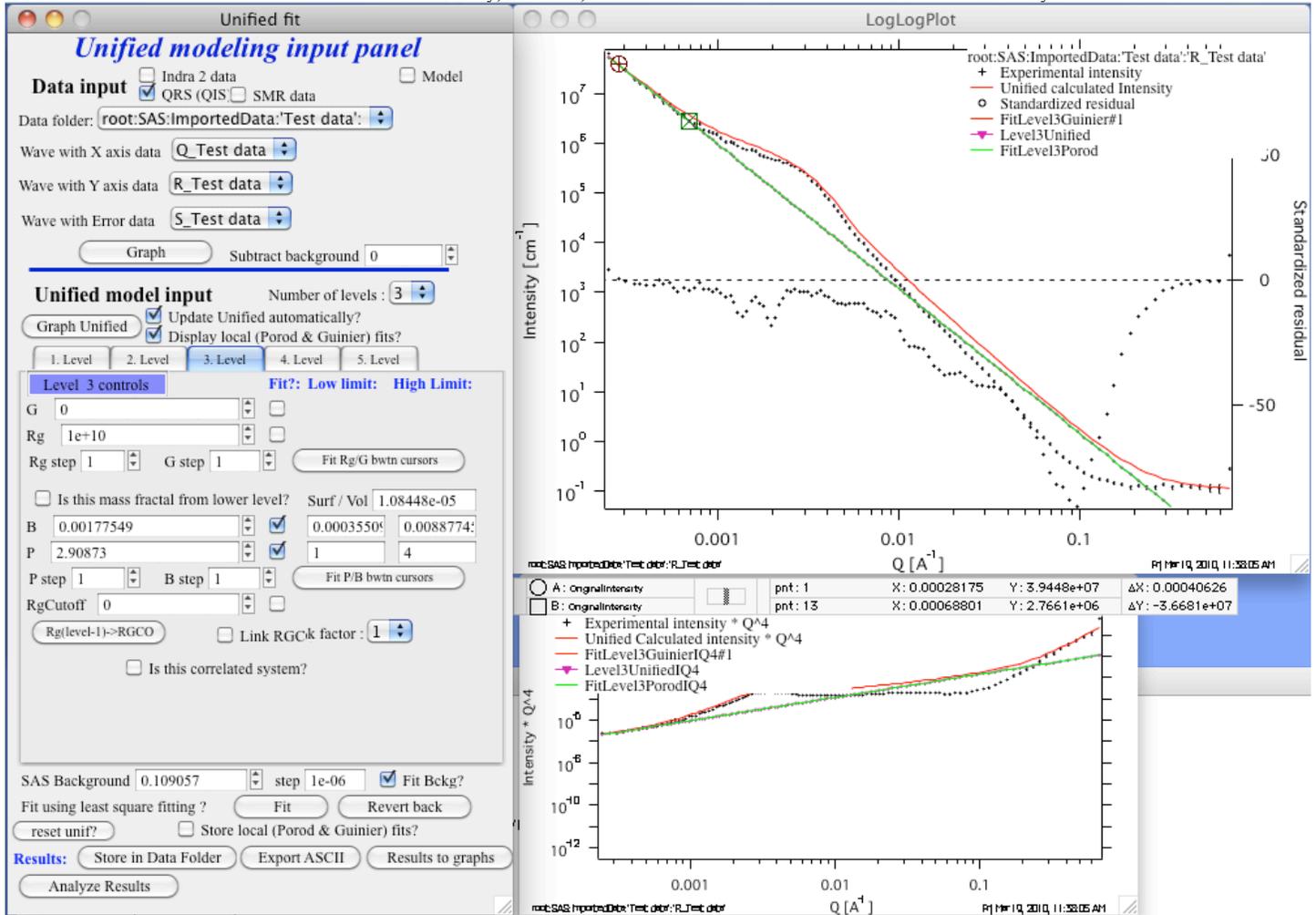
To model this, we add another level to our Unified model (set "Number of levels" to 3) and select third tab:



Now, let's remove the Guinier area from this level (NOTE: This must be last level used, you cannot add level 4 when you do this). To do so, set G to 0, Rg is set automatically to very large number. At that moment, the level represents ONLY power law slope.



And now select points 1 – 13 with cursors, check the “Fit” checkbox next to B and P and push “Fit P/B” button:

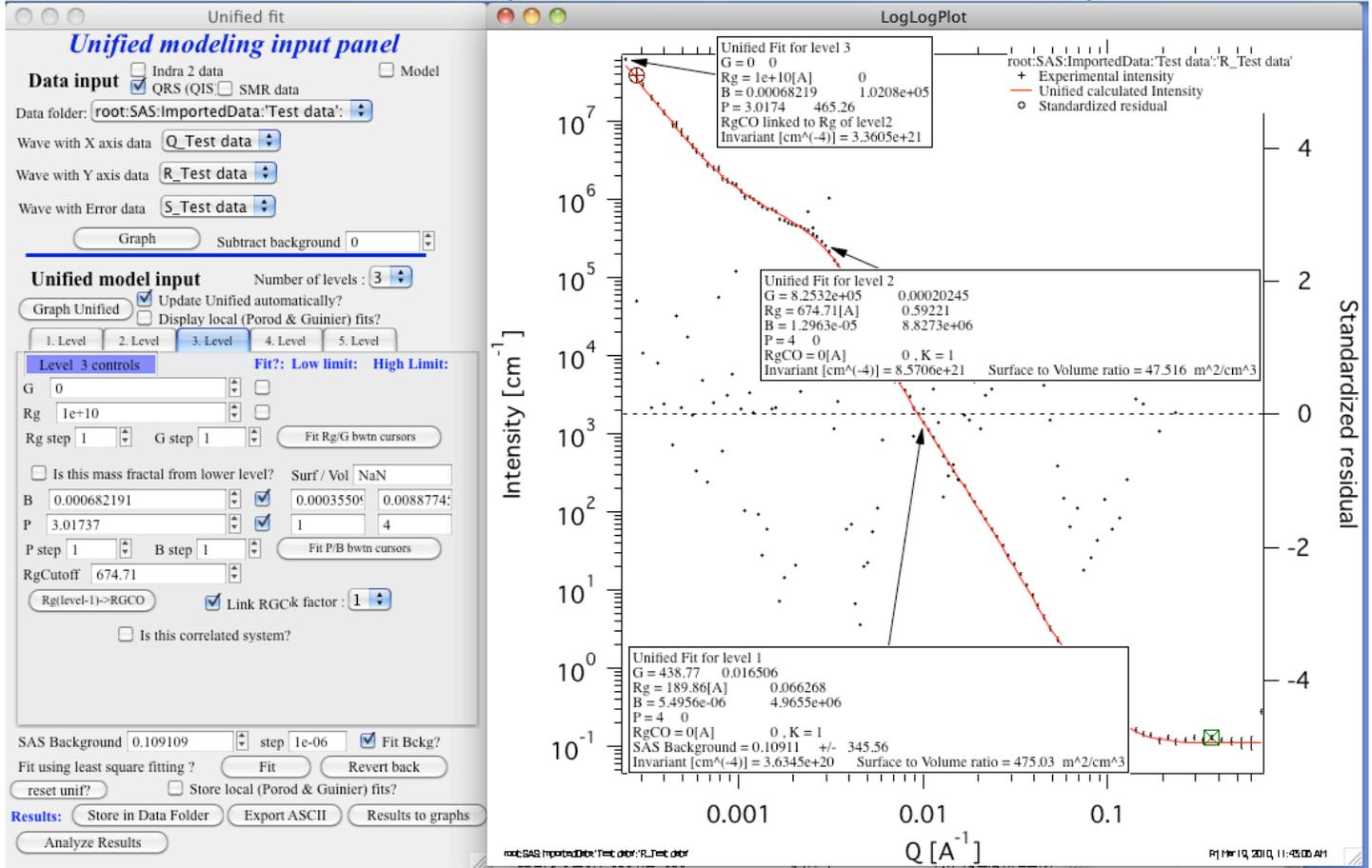


Note, that the fitted power law slope line fits reasonably well at low Q but at higher Q values it crosses measured data. This is very common for “level-type” structures, where small particles create fractal like structure on larger length scales. This is exactly where the Rg cut off comes in place. Note, that by default the RGCO is set to 0 for each level. That means there is no cut off for the scattering.

But, if (like here) the level 3 is actually agglomerate of Level 2, you can use the button “Rg(level-1)->RGCO” to copy Rg from level 2 in the RGCO of level 3. Or you can type manually 700 in RgCutOff field. Or, in this case you can use the checkbox to link them together and then they will be kept in sync during fitting. That may be the best. Check the checkbox “Link RGCO”.

Next, select all of the data (points 1 to 106) and use “Fit” button to finalize the fit. Uncheck the “Display Local fits” and push also buttons “Results to graphs”

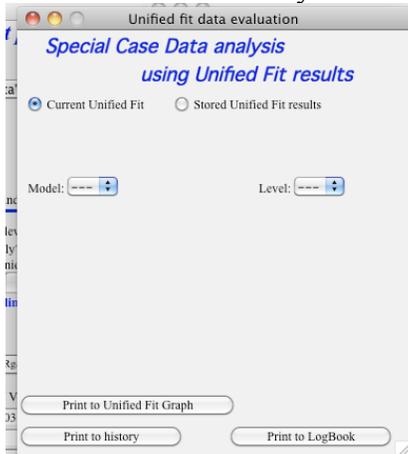
Here is the result:



You have now full description of your system... you have for each population Rg (~size), prefactor G (~volume), B (which is Porod's constant if P=4), P (power law slope), invariant and Surface to Volume ratio (if P=4). This is the basic set of results from Unified fit.

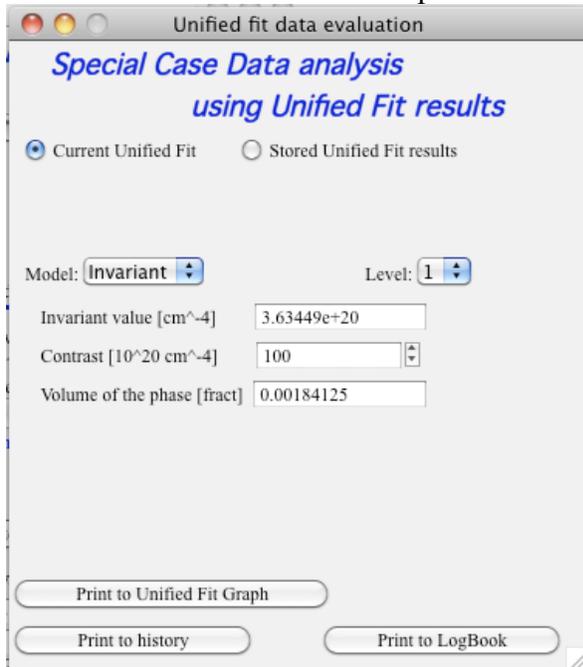
You can now save the results back to folder using "Store in Data Folder" button. If you do so, you can then compare results later, reload the fit into Unified fit tool etc.

You can also do "Analyze Results" to get further analysis.



This tool enables you to analyze some special cases in Unified...

1. Invariant. If the data intensity is absolutely calibrated and you know the contrast of the phase, this tool will calculate the volume of that phase in absolute (fractional volume) units.



Unified fit data evaluation

*Special Case Data analysis  
using Unified Fit results*

Current Unified Fit     Stored Unified Fit results

Model: **Invariant**    Level: **1**

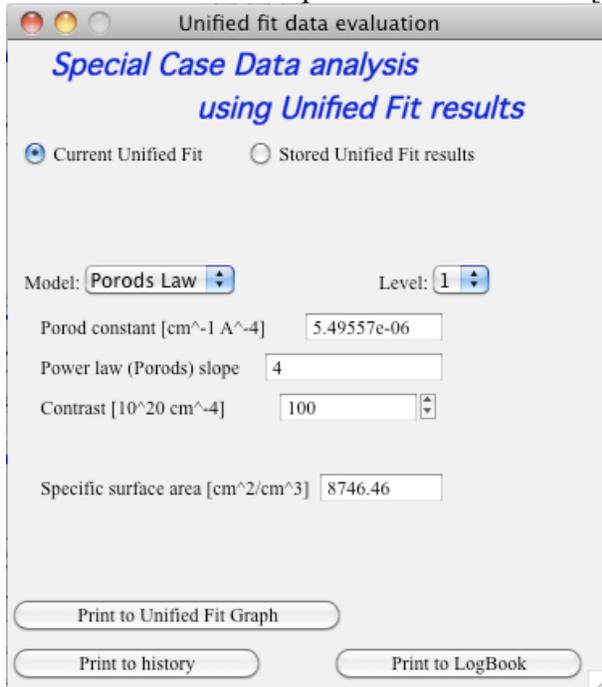
Invariant value [ $\text{cm}^{-4}$ ]    3.63449e+20

Contrast [ $10^{20} \text{cm}^{-4}$ ]    100

Volume of the phase [fract]    0.00184125

Print to Unified Fit Graph    Print to history    Print to LogBook

2. Porod's law. If the intensity is absolutely calibrated,  $P=4$ , and you know the contrast of the phase, the tool will calculate the specific surface area in [ $\text{cm}^2/\text{cm}^3$ ]



Unified fit data evaluation

*Special Case Data analysis  
using Unified Fit results*

Current Unified Fit     Stored Unified Fit results

Model: **Porods Law**    Level: **1**

Porod constant [ $\text{cm}^{-1} \text{A}^{-4}$ ]    5.49557e-06

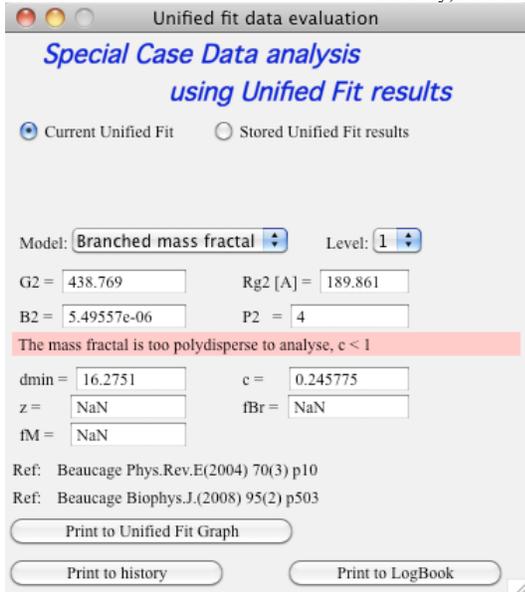
Power law (Porods) slope    4

Contrast [ $10^{20} \text{cm}^{-4}$ ]    100

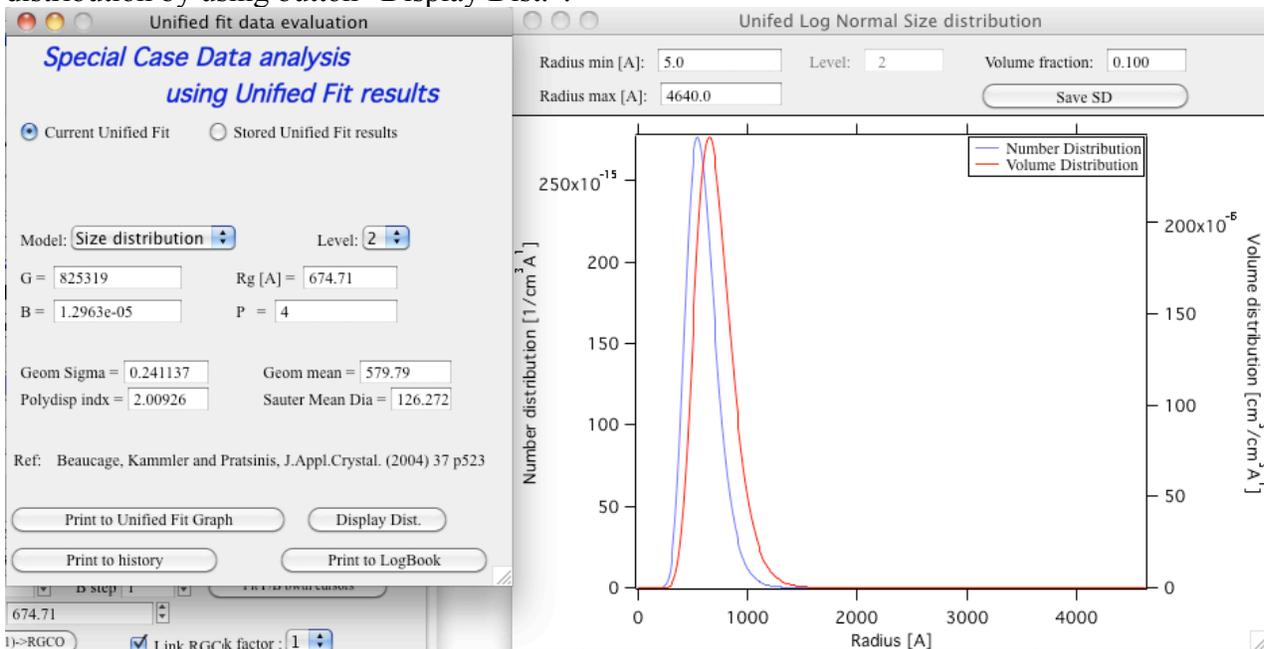
Specific surface area [ $\text{cm}^2/\text{cm}^3$ ]    8746.46

Print to Unified Fit Graph    Print to history    Print to LogBook

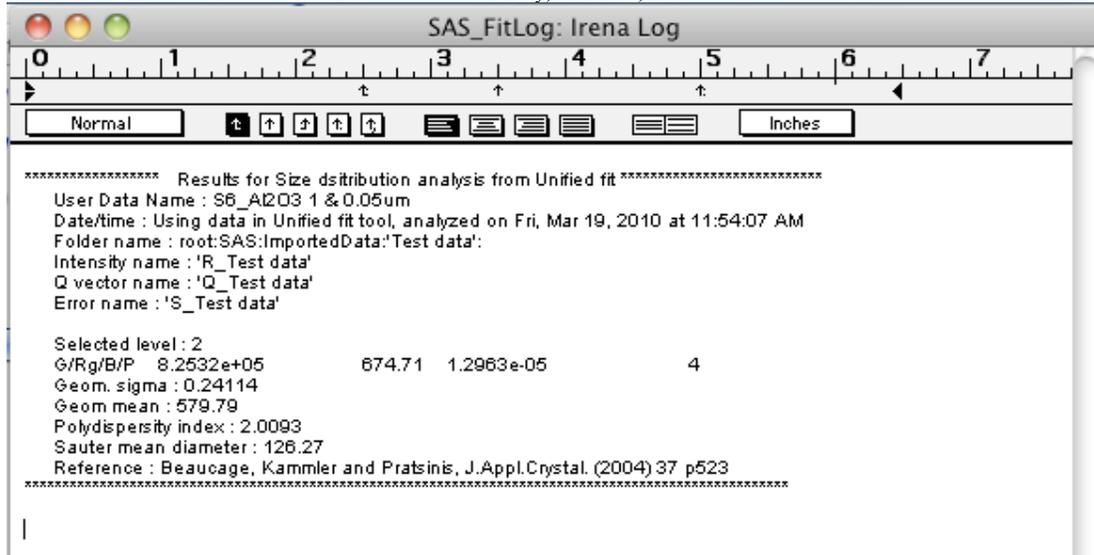
3. Branched mass fractal. If you have mass fractal data, you can use this tool to extract some specific parameters of this fractal. Please, read the publications referenced in the panel to figure this out. It is really interesting and useful theory when applied correctly.



- Size distribution. Greg Beaucage also developed method how to estimate parameters of log-normal size distribution from the Unified fit. The tool can calculate parameters (as defined in the referenced publication) and generate (and store) the volume and number size distribution. You can visualize the distribution by using button “Display Dist.”:



You can also print the results to history area as well as into the LogBook...



You can save the calculated size distribution into the folder with data and then visualize it with other Irena tools – and compare it with Size distribution, modeling etc.

For more details please read the manual and original references. Unified fit seems easy, but there are some special tricks, which are kind of built in, and it is important to understand the limitations and specifics.