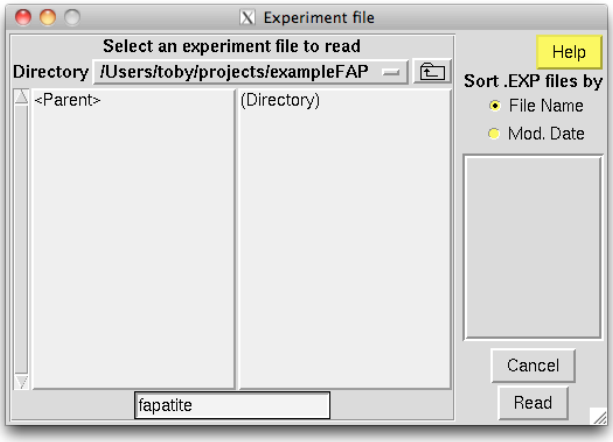
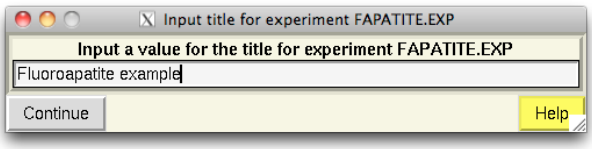
Start EXPGUI

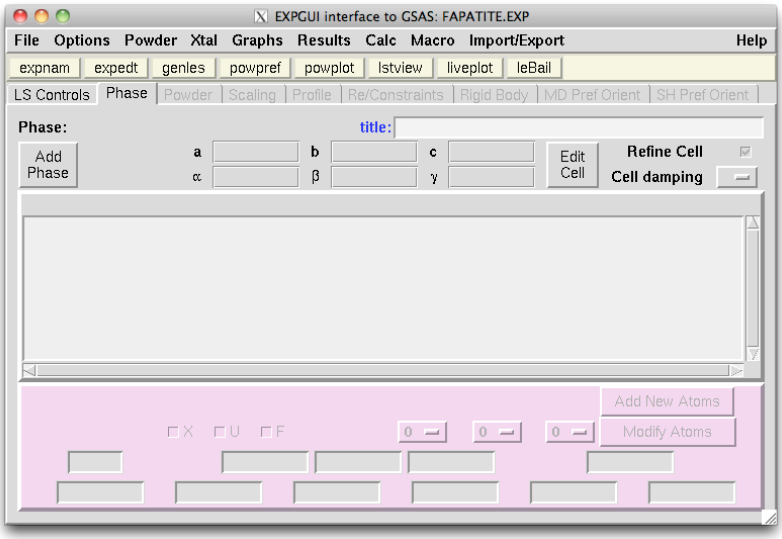



Enter a name for the experiment file (your choice). Press Read. Since this is a file we want to create we get a warning box:

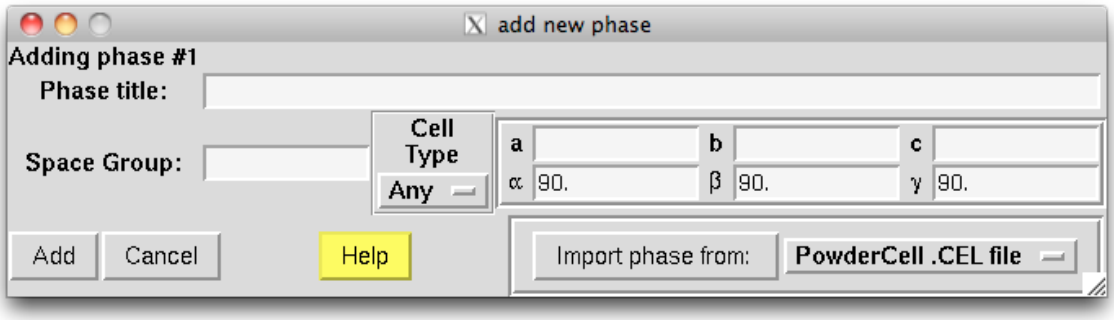
Press Create and enter an arbitrary title (for your own use):



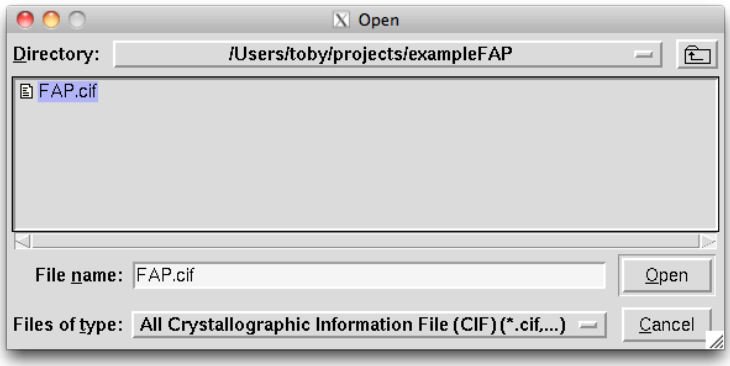

Press Continue. EXPGUI then opens and brings you to the phase panel (this is a hint). Note that almost all other tabs are disabled (also a hint).



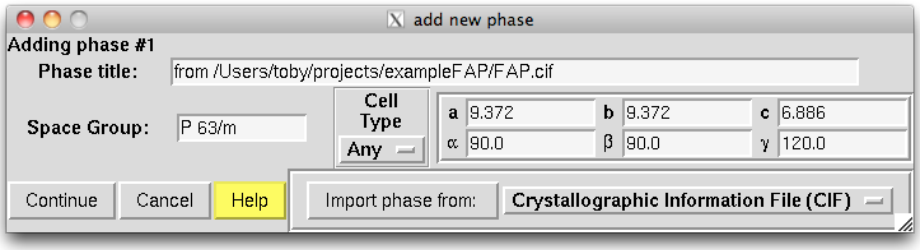

Press Add Phase to enter the structural information. Note that we are going to read a cell and atoms in one step, but this can be done in different steps. This information can be read from a file or be typed into windows.



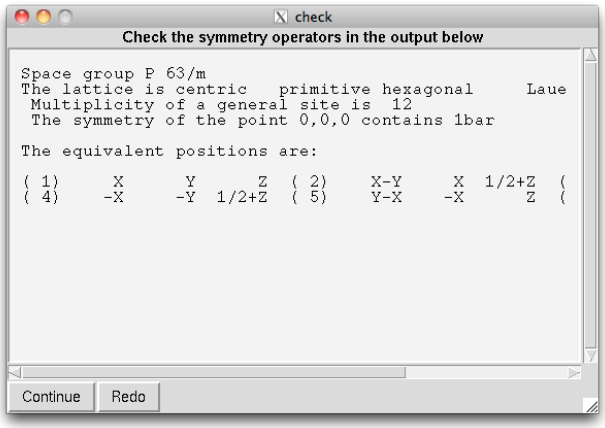

Here we read from a CIF file by changing PowderCell to CIF. This brings up a window where we can select the CIF file (note this window will appear different depending on the operating system):



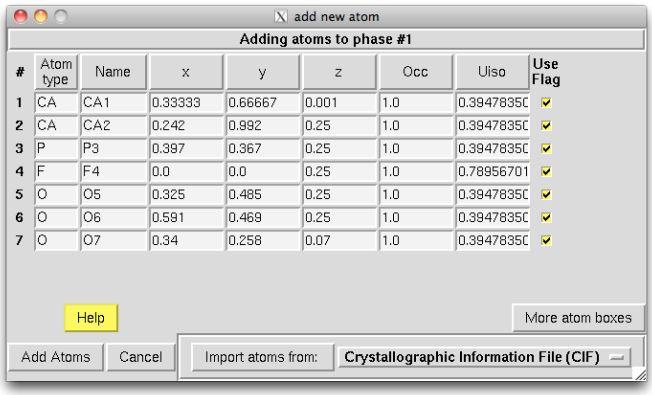

Press Open. Lattice/space group information is loaded into the window for us:




Look this information over, it is not always correct – particularly the space group (at least one space is needed here between the P and the 6, for example). Here all is OK so, press Continue. EXPGUI shows the generated symmetry operations:

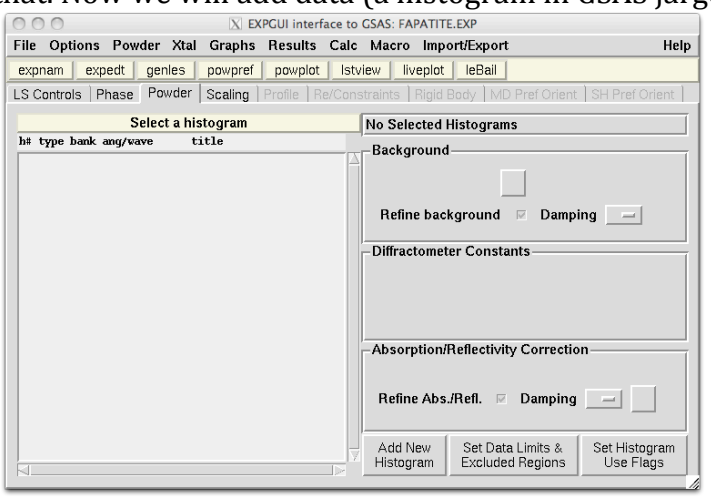


It is a good idea to check these against the International Tables Volume A. These are what was wanted, so we press Continue. Next we see the atoms:

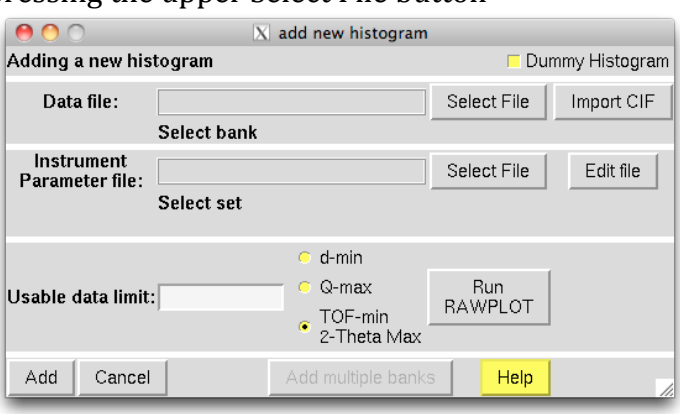



| # | Atom type | Name | x | y | z | Occ | Uiso | Use Flag |
|---|---|---|---|---|---|---|---|---|
| 1 | CA | CA1 | 0.33333 | 0.66667 | 0.001 | 1.0 | 0.3947835( | ✓ |
| 2 | CA | CA2 | 0.242 | 0.992 | 0.25 | 1.0 | 0.3947835( | ✓ |
| 3 | P | P3 | 0.397 | 0.367 | 0.25 | 1.0 | 0.3947835( | ✓ |
| 4 | F | F4 | 0.0 | 0.0 | 0.25 | 1.0 | 0.78956701 | ✓ |
| 5 | O | O5 | 0.325 | 0.485 | 0.25 | 1.0 | 0.3947835( | ✓ |
| 6 | O | O6 | 0.591 | 0.469 | 0.25 | 1.0 | 0.3947835( | ✓ |
| 7 | O | O7 | 0.34 | 0.258 | 0.07 | 1.0 | 0.3947835( | ✓ |

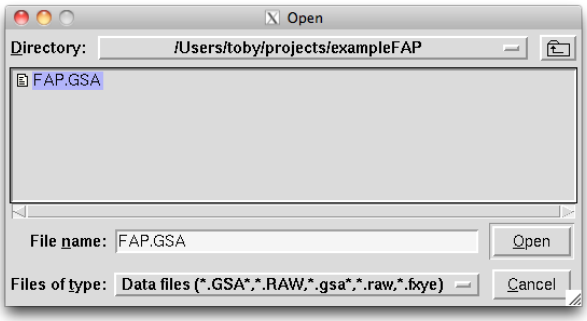
Note that these Uiso values are very large! This CIF must have them wrong. We can change them now, but we will deal with this later. Otherwise the input is OK, so press Add Atoms to continue. Now the Phase panel has been filled out with our first (and in this case, only) phase. Note we can now access the Powder panel. So lets do that. Now we will add data (a histogram in GSAS jargon).



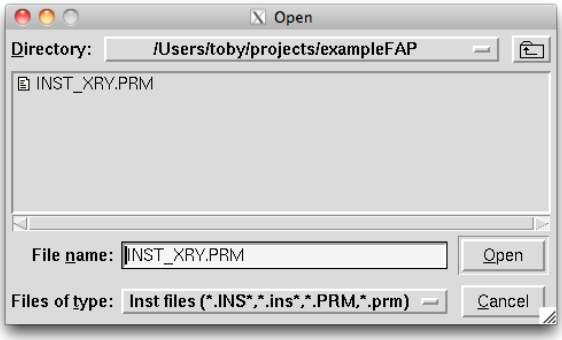

Press Add New Histogram (at bottom). Here we will enter the data file name by pressing the upper Select File button



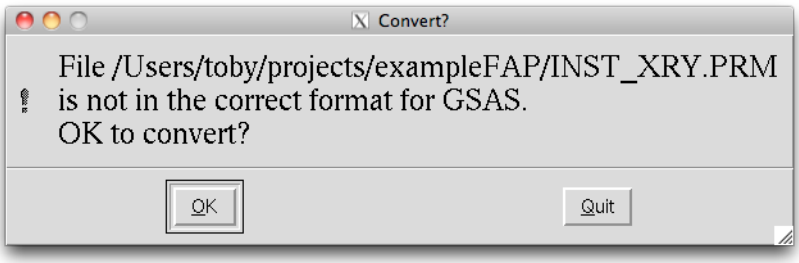

Select the FAP.GSA data file:



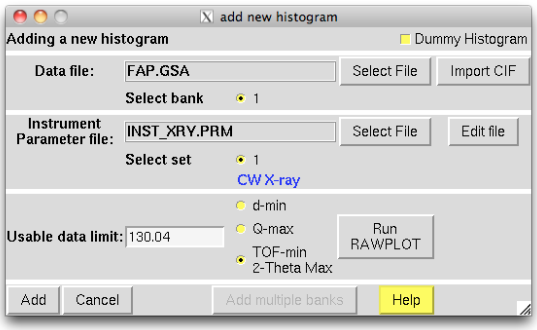

Then press Open. We now need an instrument parameter file to specify the data type, provide default peak widths etc. We will use a GSAS-distributed file for this. Press the lower Select file button and select file INST_XRY.PRM



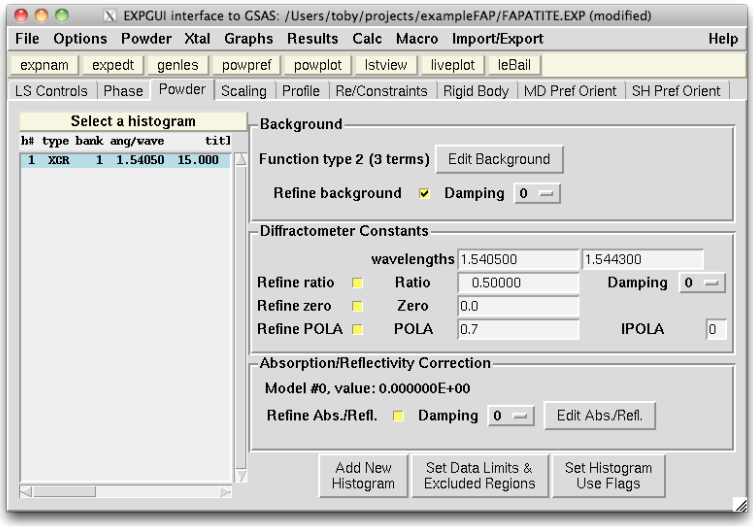

The press Open. You may get a warning that you can ignore (just press OK):



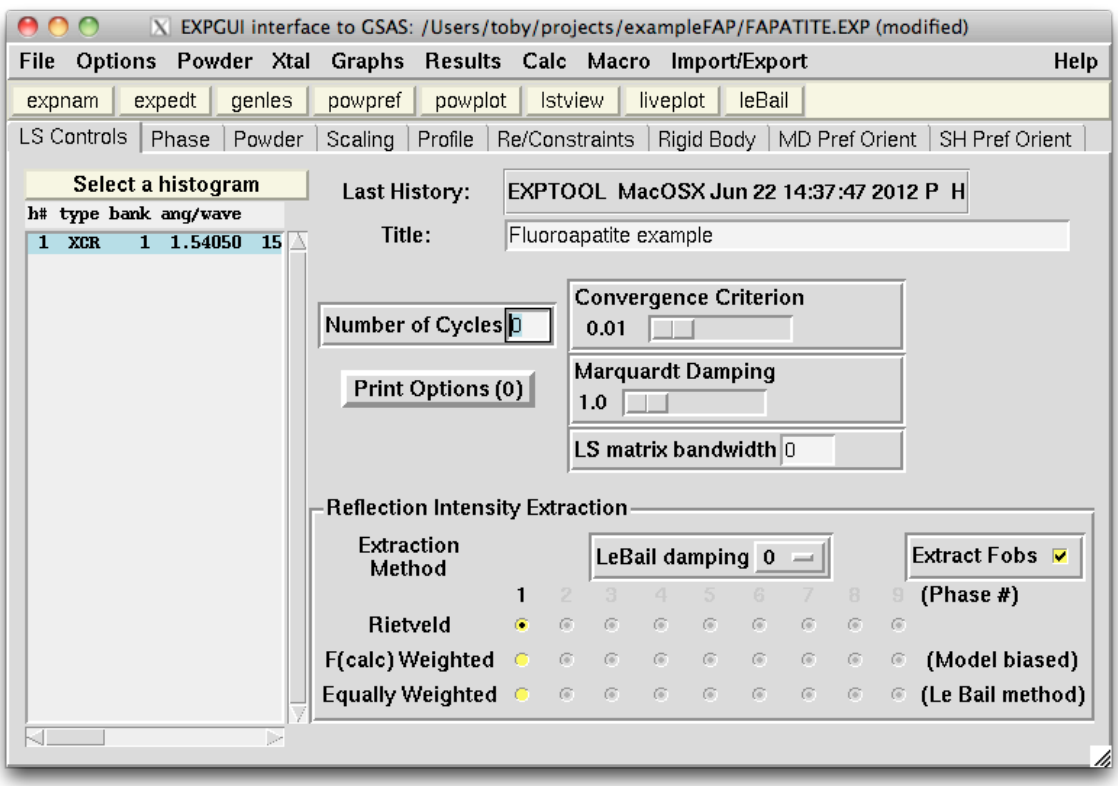

The powder histogram information is now ready to be added to the experiment, but before we do that we might want to change the data limit to use less data. One can use RAWPLOT to look at the data, but here lets use all the data (to 130 degrees).



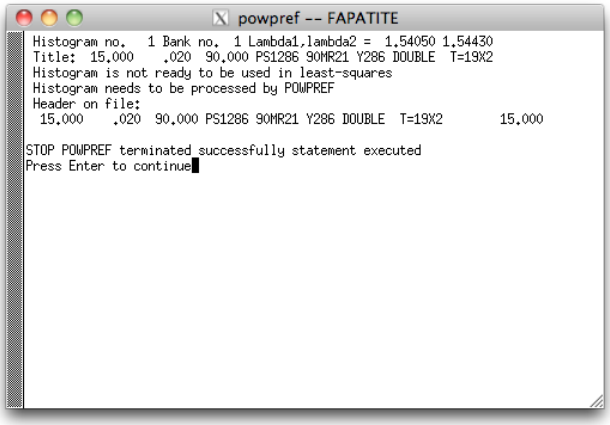

Press Add to add the data. We see this in the Histogram panel:



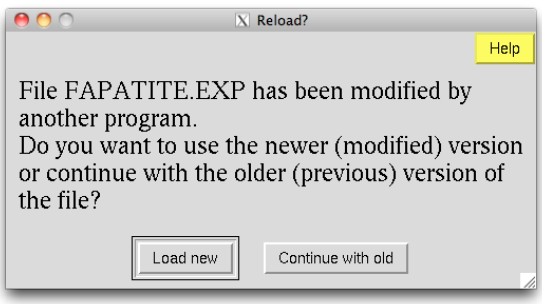

Before we do any refinement, we should see what the pattern looks like. On the controls panel set the number of cycles to zero:



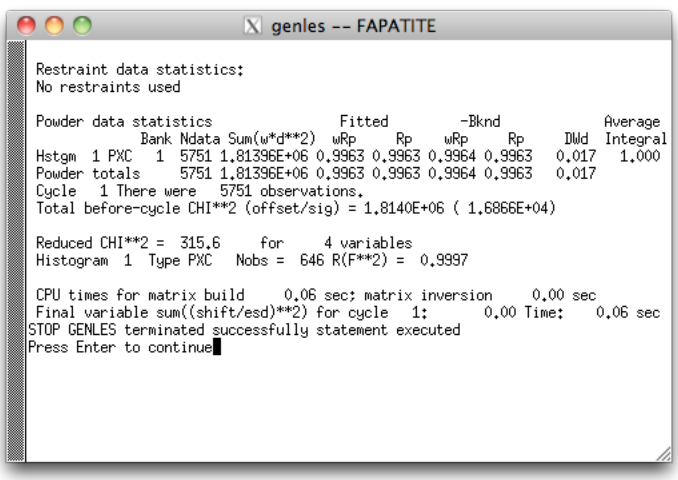

To start running press the powpref button



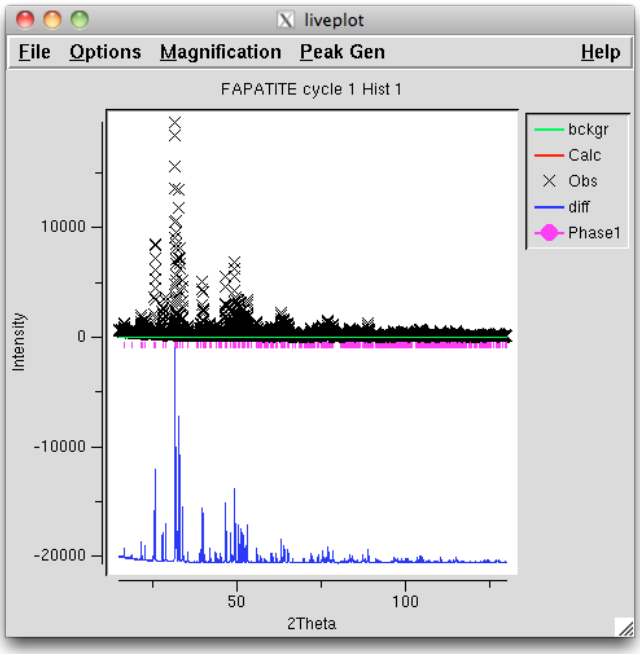

Press Enter in the window to continue. The EXP file has been changed by the POWPREF program, so we press Load New



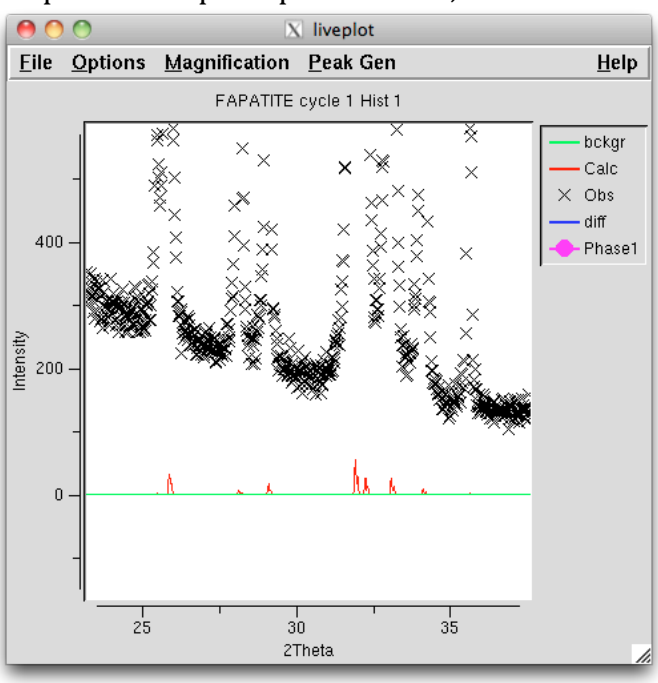

Then press the genles button to start that program. With 0 cycles, it will compute the powder intensities, but not refine any parameters.



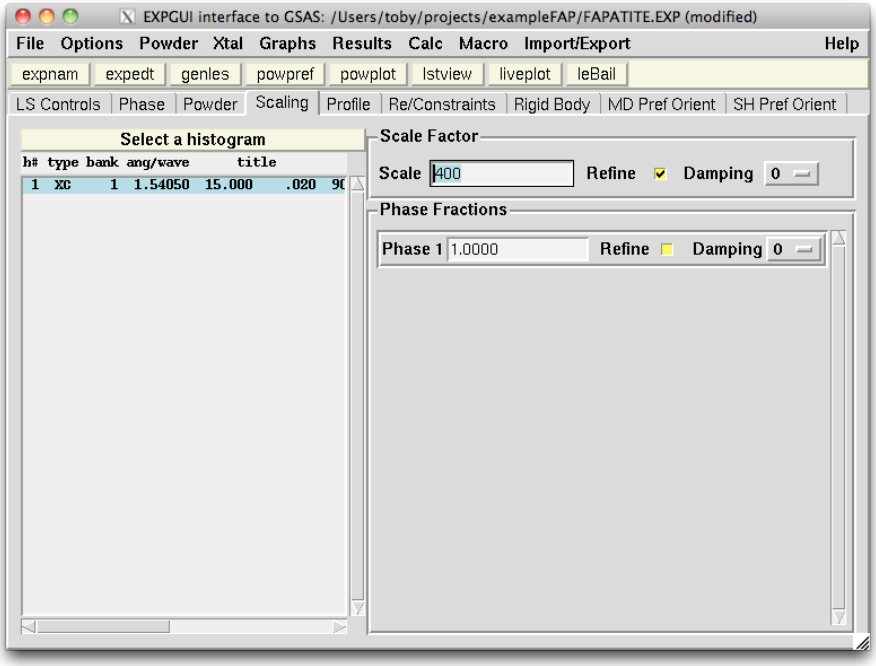

As before press Enter and then Load New in the next window. Now press the liveplot button to see a plot. We see the plotted data, but no computed intensity.



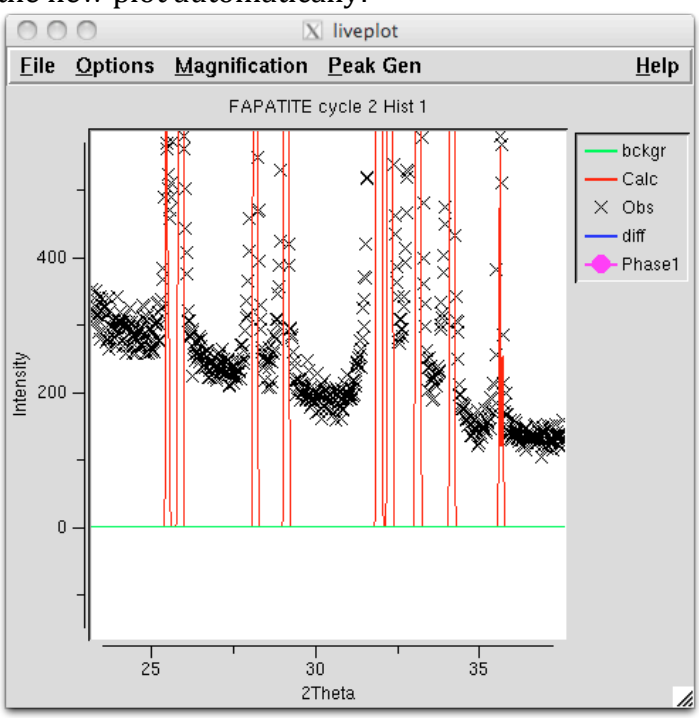

If we zoom in enough (drag a box with the left mouse button to zoom in; click with the right to zoom out), we see that there are computed intensities but they are computed with perhaps 50 counts, while the raw data have 20,000 counts.



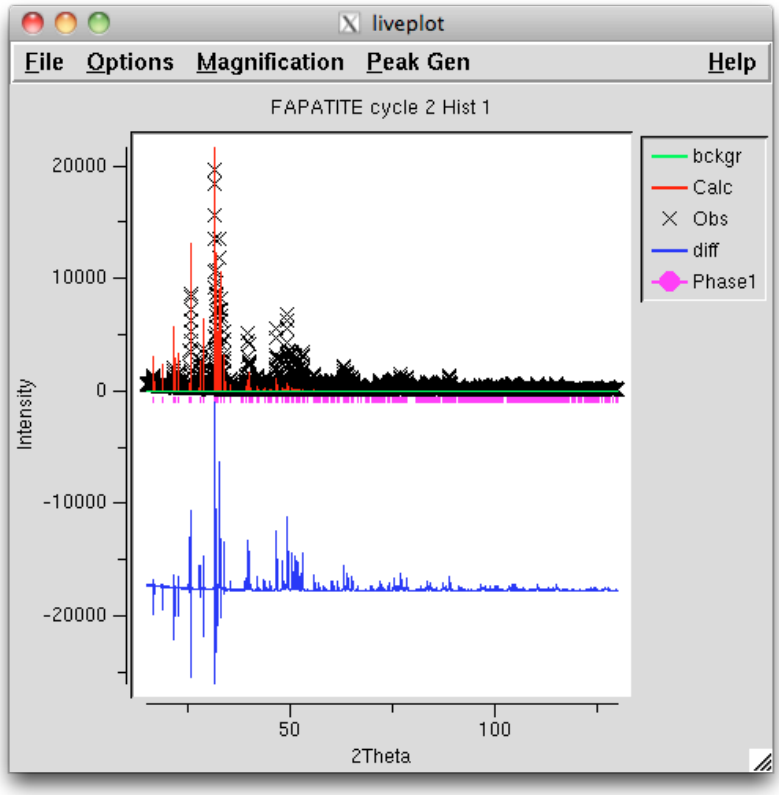

While we could refine the scale factor, lets instead multiply the scale factor by 20,000/50 (400) using the scaling panel:



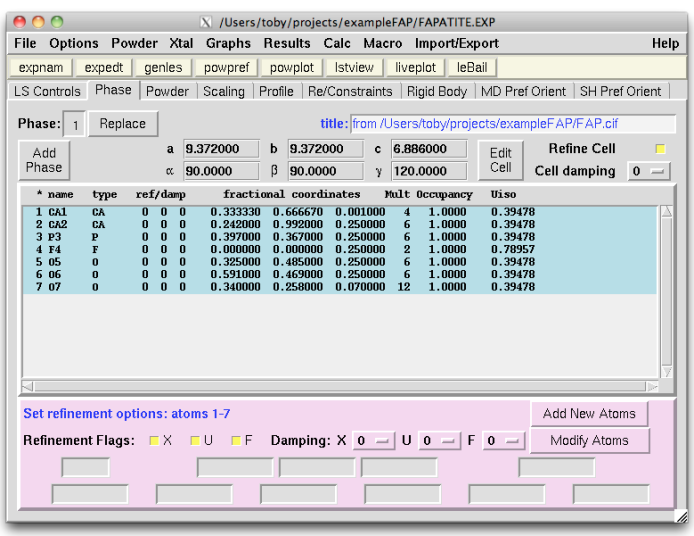

Then press Genles, press Enter and Load New as before. Liveplot updates to show us the new plot automatically:



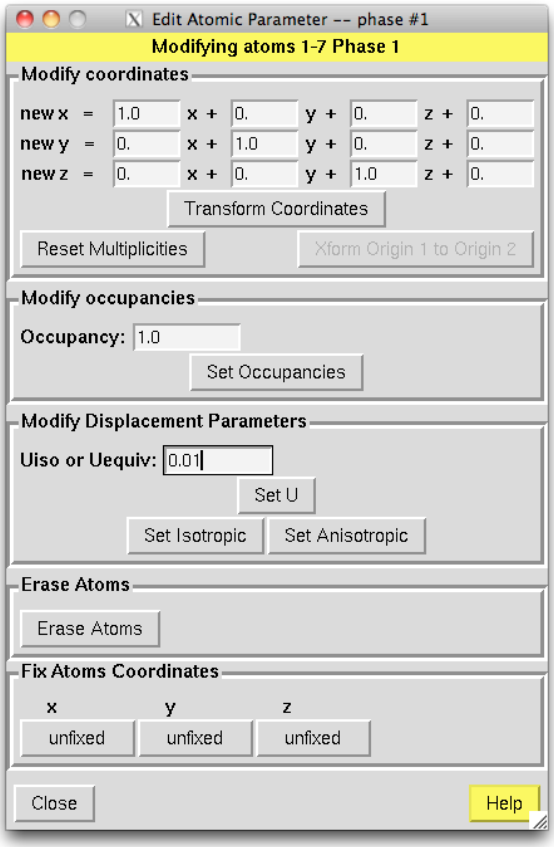

Or when we zoom out:



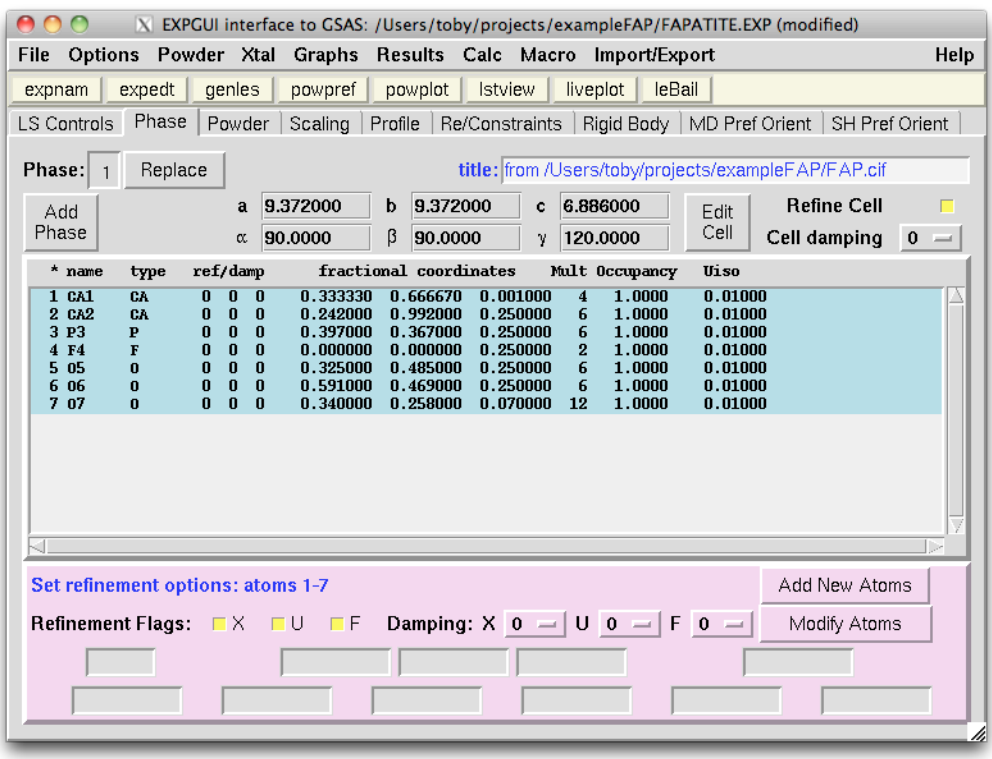

Note that peaks are more or less lining up. The intensity falls off way too fast (this is due to the Uiso values being too high. Lets change them. On the phase panel drag the mouse over all atoms:



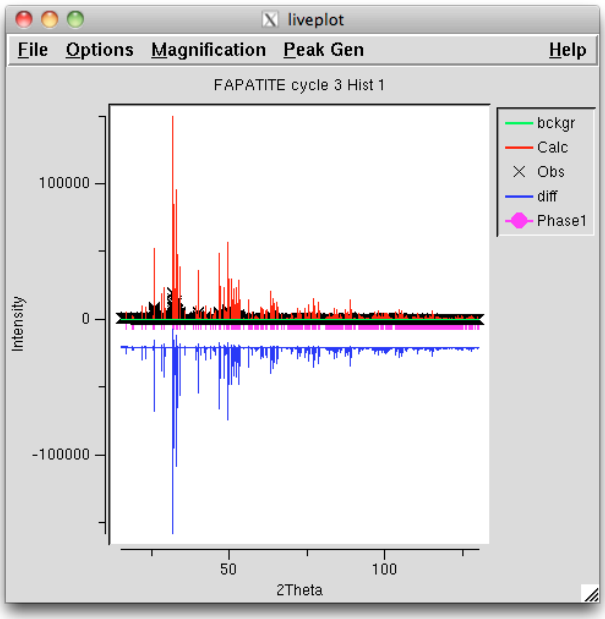

Press Modify Atoms. Change the Uiso value in the new window to 0.01



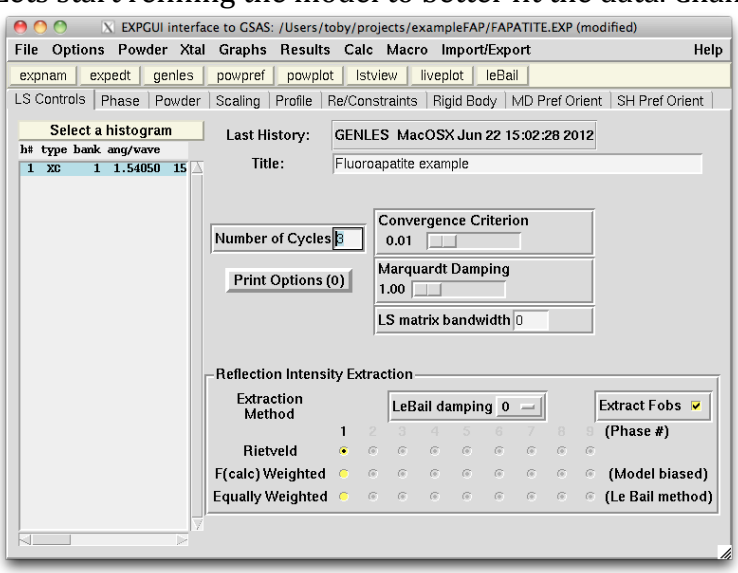

Press Set U and then Close. Note the Uiso values are now changed on the phase panel:



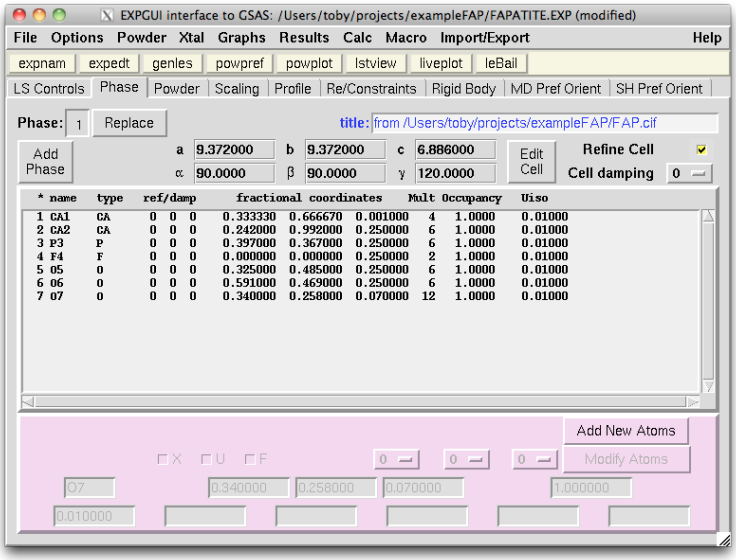
Recompute with Genles, as before. Liveplot now shows much better intensities, though now our scale factor is probably too big.



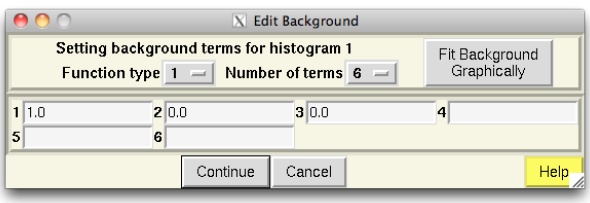

Lets start refining the model to better fit the data. Change the number of cycles to 3



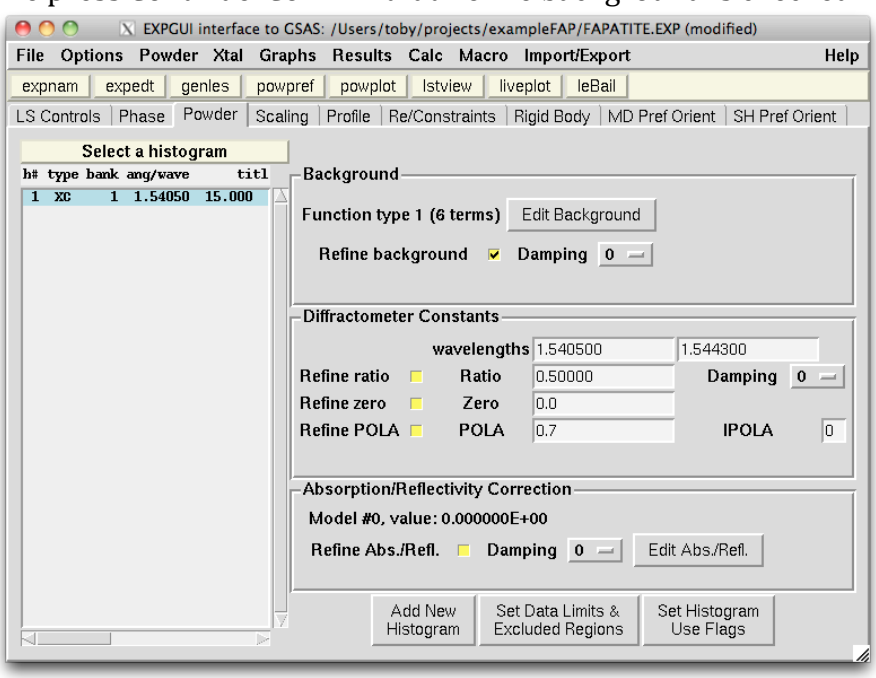

Use the refine cell option on the phase panel




Change the background to type 1 with six parameters. Select the Powder Panel, use Edit Cell. One the edit cell window select function type 1, and number of terms 6.




The press Continue. Confirm that Refine background is checked.




Now lets refine by pressing the genles button.
The GENLES output shows the $\chi^2$ dropping from 6813 to 74

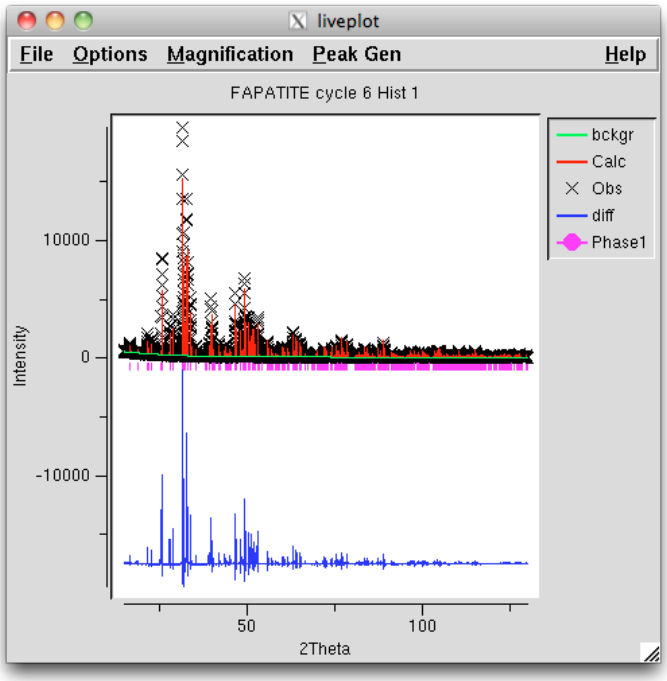
genles -- FAPATITE

```
Restraint data statistics:
No restraints used

Powder data statistics                Fitted         -Bknd             Average
             Bank Ndata Sum(w*d**2)  wRp     Rp    wRp     Rp     DWd  Integral
Hstgm  1 PXC   1  5751 3.91213E+07 4.6269 2.8865 4.6352 2.8899   0.397   1.000
Powder totals     5751 3.91213E+07 4.6269 2.8865 4.6352 2.8899   0.397
Cycle   4 There were   5751 observations.
Total before-cycle CHI**2 (offset/sig) = 3.9121E+07 ( 3.6501E+05)

Reduced CHI**2 =  6813.     for     9 variables
Histogram  1  Type PXC   Nobs =  646 R(F**2) =  2.9591

CPU times for matrix build     0.06 sec; matrix inversion     0.00 sec
Final variable sum((shift/esd)**2) for cycle   4:    5421.44 Time:    0.06 sec

Restraint data statistics:
No restraints used

Powder data statistics                Fitted         -Bknd             Average
             Bank Ndata Sum(w*d**2)  wRp     Rp    wRp     Rp     DWd  Integral
Hstgm  1 PXC   1  5751 4.86594E+05 0.5160 0.4190 0.6357 0.5303   0.379   1.000
Powder totals     5751 4.86594E+05 0.5160 0.4190 0.6357 0.5303   0.379
```

genles -- FAPATITE

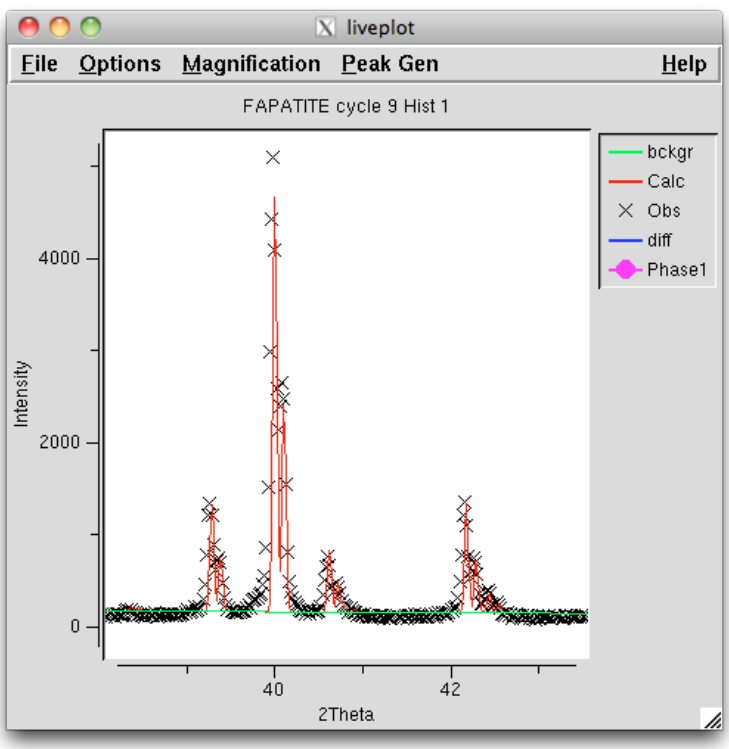
```
Reduced CHI**2 =  84.74     for     9 variables
Histogram  1  Type PXC   Nobs =  646 R(F**2) =  0.4534

CPU times for matrix build     0.06 sec; matrix inversion     0.00 sec
Final variable sum((shift/esd)**2) for cycle   5:     250.52 Time:    0.06 sec

Restraint data statistics:
No restraints used

Powder data statistics                Fitted         -Bknd             Average
             Bank Ndata Sum(w*d**2)  wRp     Rp    wRp     Rp     DWd  Integral
Hstgm  1 PXC   1  5751 4.25375E+05 0.4825 0.3839 0.5928 0.4812   0.339   1.000
Powder totals     5751 4.25375E+05 0.4825 0.3839 0.5928 0.4812   0.339
Cycle   6 There were   5751 observations.
Total before-cycle CHI**2 (offset/sig) = 4.2537E+05 ( 3.9158E+03)

Reduced CHI**2 =  74.08     for     9 variables
Histogram  1  Type PXC   Nobs =  646 R(F**2) =  0.4432

CPU times for matrix build     0.06 sec; matrix inversion     0.00 sec
Final variable sum((shift/esd)**2) for cycle   6:     246.65 Time:    0.06 sec
STOP GENLES terminated successfully statement executed
Press Enter to continue
```

Liveplot will also show the fit looks better.

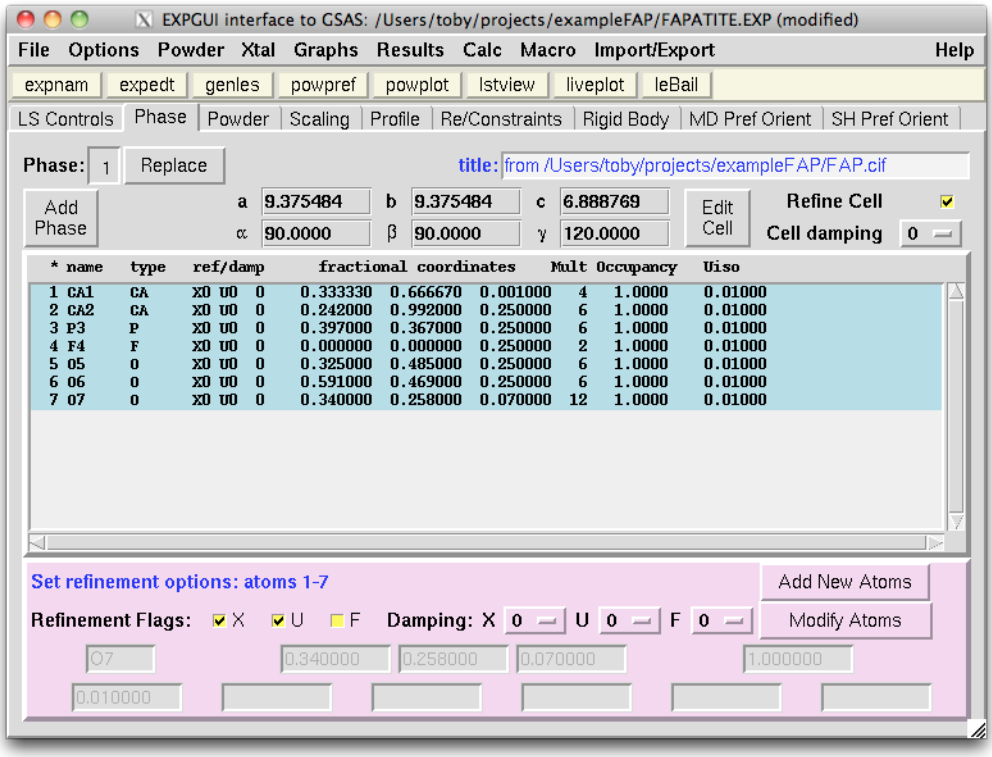



Run powpref and genles again (because the cell dimensions changed a fair amount). $\chi^2$ drops to 61.

Zooming in on the pattern in liveplot shows the match to the intensities are not bad. The peak shape is a bit narrow, but not bad.

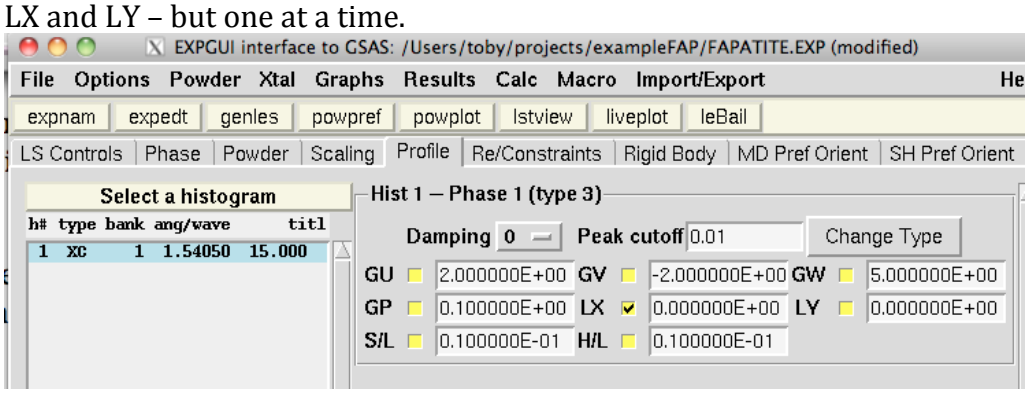



Select to refine the atom positions (as allowed by symmetry) and Uiso values by selecting all atoms and click on X and F below the atoms:

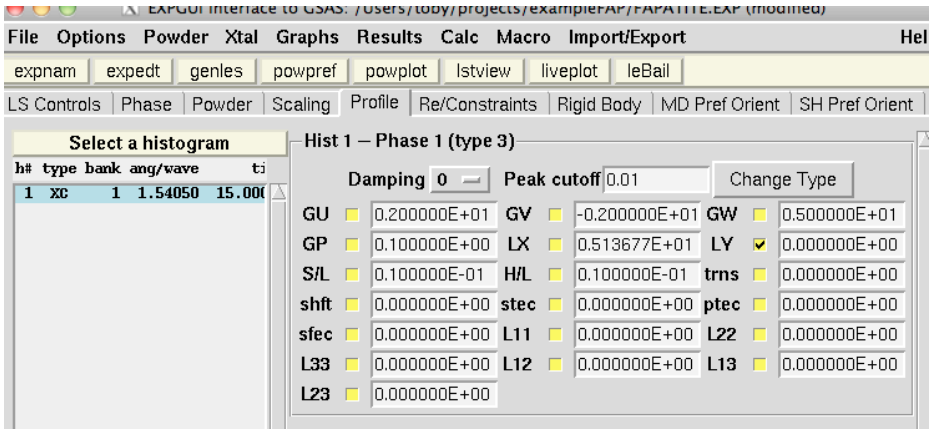



Press genles to refine. The agreement improves a bit. If this structure were a bit more complex, we might have initially grouped some Uiso values or only vary some of the heavy atoms.

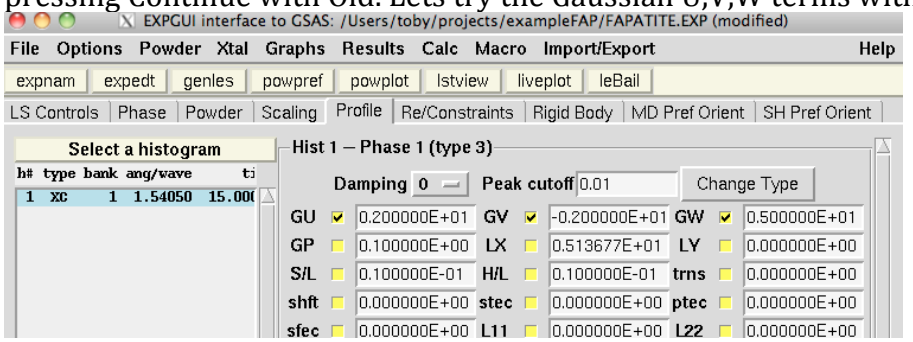
Now lets improve the peak shapes. The initial profile is not fitting the tails of the pattern well (see the liveplot example above), so let's refine the two Lorentian terms LX and LY – but one at a time.



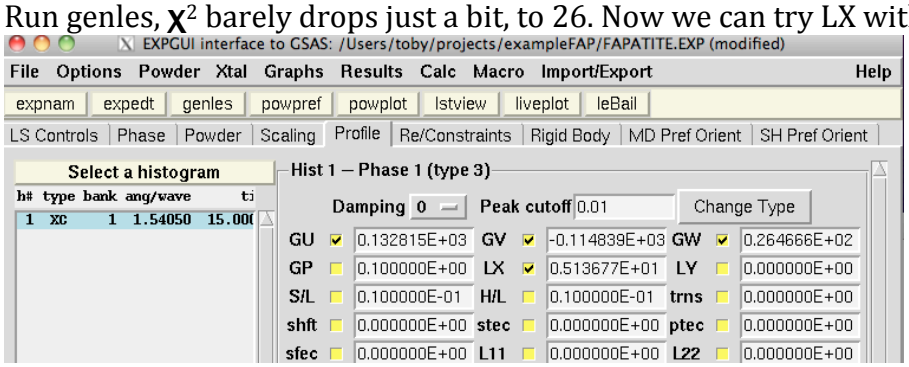

Click on LX and then genles. $\chi^2$ drops to 29.
Try the same with LY turning LX off




Run genles, $\chi^2$ barely drops, so we do not need this. We could reject the change by pressing Continue with Old. Lets try the Gaussian U,V,W terms without LX or LY:

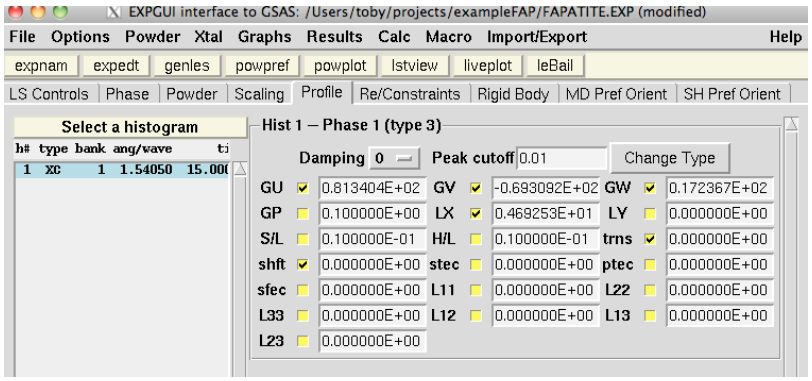



Run genles, $\chi^2$ barely drops just a bit, to 26. Now we can try LX with those terms




since the peak shape has been changed a fair amount, lets run powpref and then genles. $\chi^2$ drops quite a bit, to 17. It is a good idea to run powpref and genles again, but not much changes.

Since this is Bragg-Brentano (lab) data, lets correct for sample shift and transparency (since the sample is not that absorbing). Turn on shft and trns:




lets run genles. $\chi^2$ drops quite a bit, to 5. Again since peaks have moved, lets run powpref and then genles again. $\chi^2$ even more, to 3.2. Doing that again causes no improvement.

There are probably a few more things we should try in this fit, but it is mostly complete.