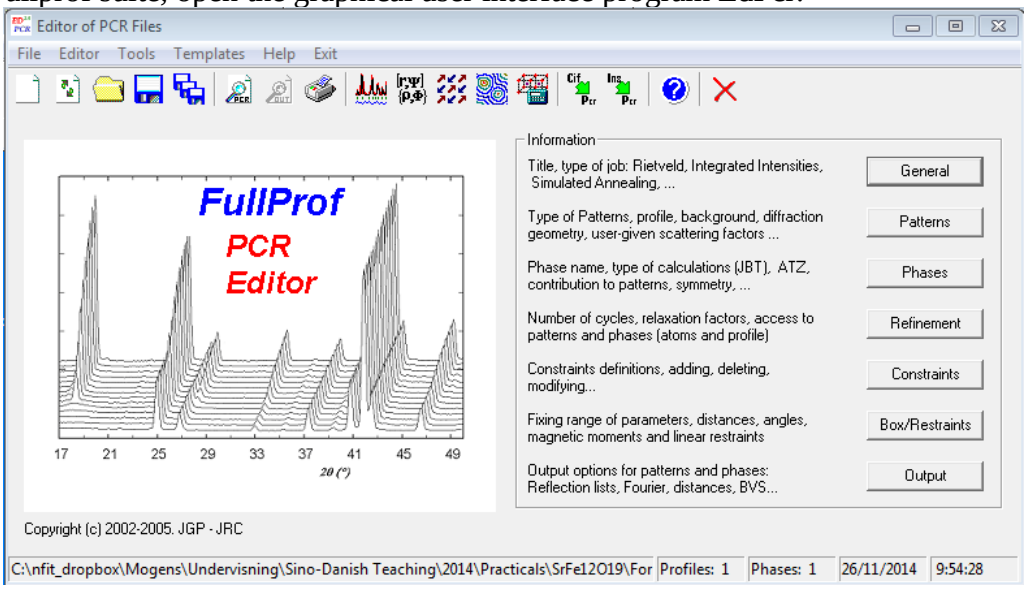
TUTORIAL: Introduction to size refinements in Fullprof

This is a short introduction to Fullprof to be used for the refinement of powder X-ray diffraction data. The Fullprof program can be downloaded from: https://www.ill.eu/sites/fullprof/ and the files needed for the tutorial is found on the moodle.

Example of Fe:

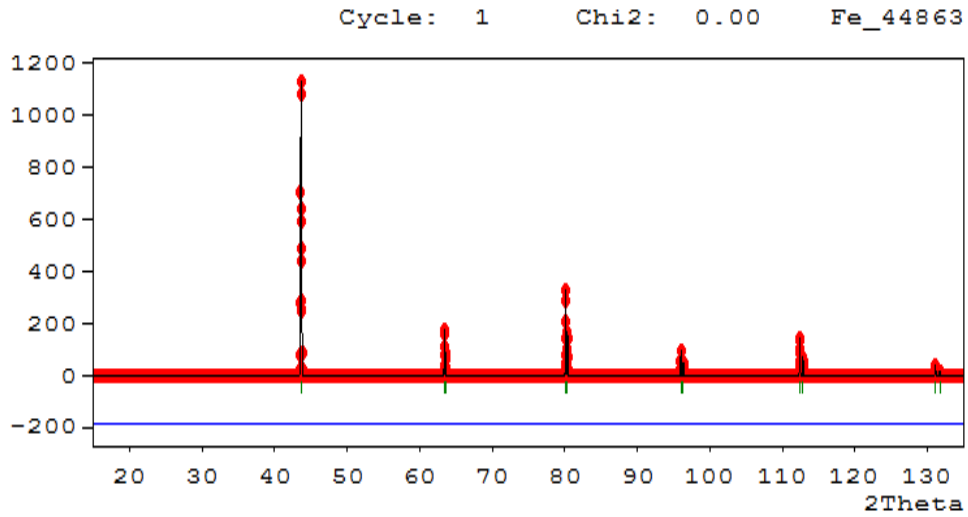
This example is meant as an easy example of investigating the impact on a powder diffraction pattern of iron by changing the scale factor and unit cell parameter in the model.
From Fullprof suite, open the graphical user interface program EdPcr.



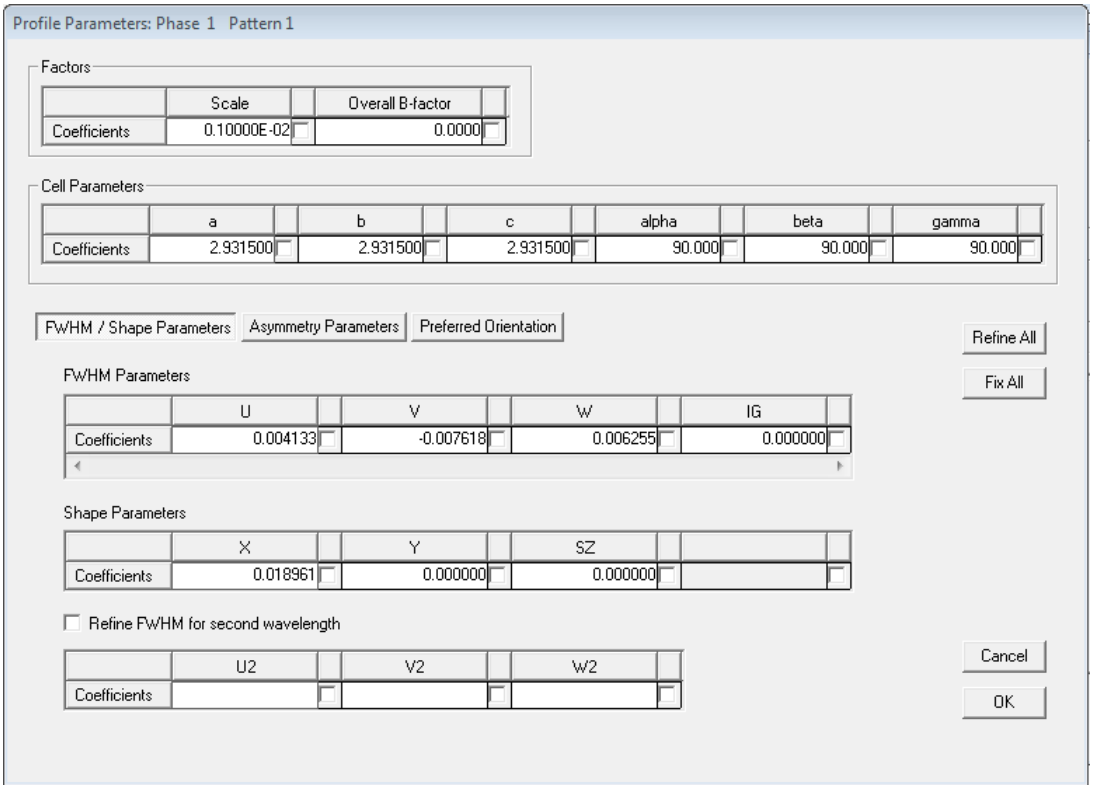
The information about the crystal structure is held in a *.pcr file – which is imported by clicking  .

First, we wish to look at some model data – of iron – therefore you should open the `Fe.pcr` file.

You can now click on  to see how the powder pattern of Fe should look like:



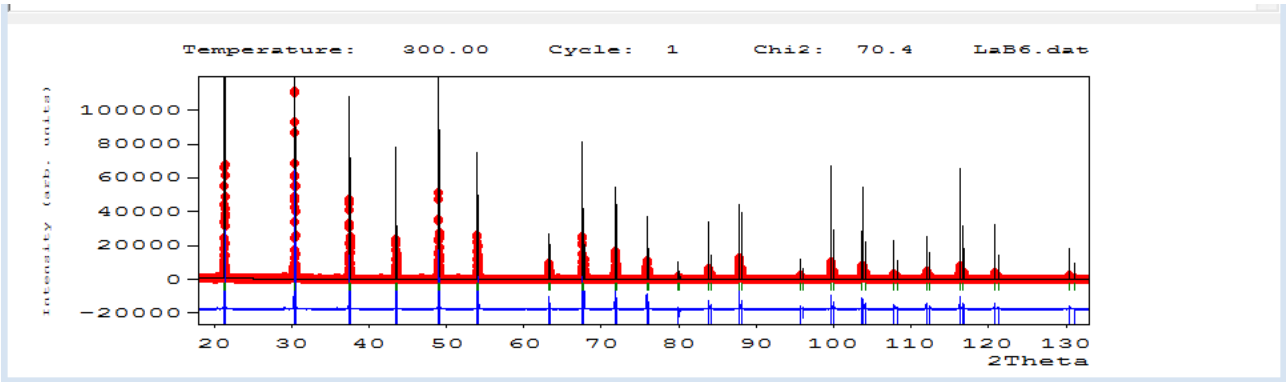
To change the unit cell parameter, go into the Refinement and click the bottom Profile, which will produce the following screen:



1) Here you can now try to change the number in "Scale" and click "OK" and "OK" again and then  to see the effect of the change you have made.
2) Likewise can you try to change the unit cell parameters a,b,c – the structure is cubic so they should always have the same value.
3) You can also attempt to change the peak profile parameters U, V, W, which are Gaussian parameters, while X, Y are Lorentzian parameters. The parameter describing the size is actually Y.

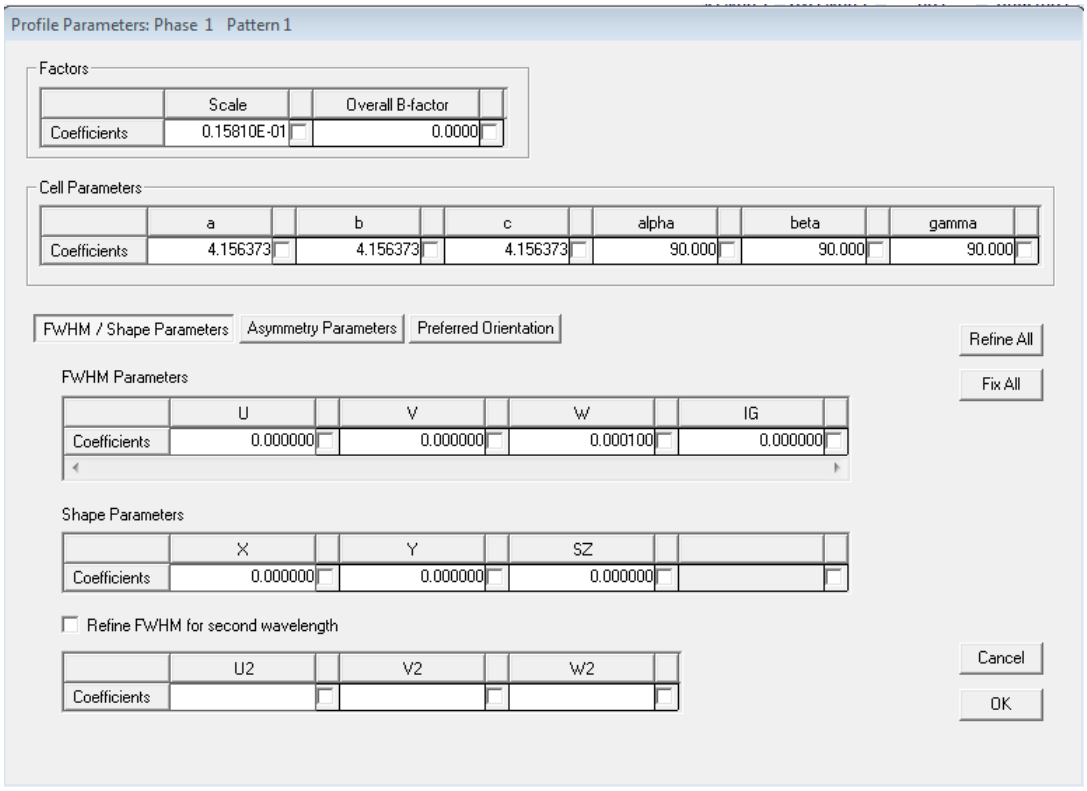
What happens to the diffraction peak if you pretend to heat up the sample and increase the unit cell parameters?
After having looked at different parameters we will change to look at some real data.

Example of LaB_6:
LaB6 is a NIST (National Institute of Standards and Technology, Washington US) standard – the specific samples are send to many laboratories in order to have the same reference measurement. The sample is made so it has no peak broadening due to size, therefore the samples is to calibrate the instrument broadening.
Open EdPcr and go into the correct library and open `LaB6.pcr`
Click the refine bottom  to see how the model fits the data.



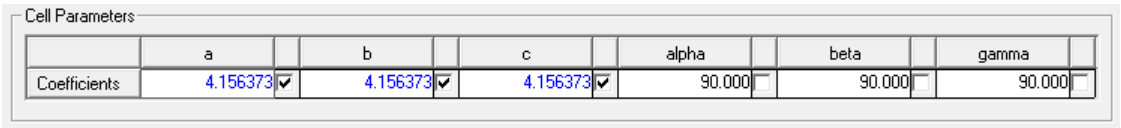
The peak intensity is much too high – if you zoom into the peaks you will also see that the peak width is too small – I basically made the peak width almost zero. Now we need to do a proper refinement of this data. So we need to refine the scale factor, after that the unit cell parameters and finally we should refine the peak profile.

1) Click [Refinement], in refinements click [Profile] now you will see the following screen:



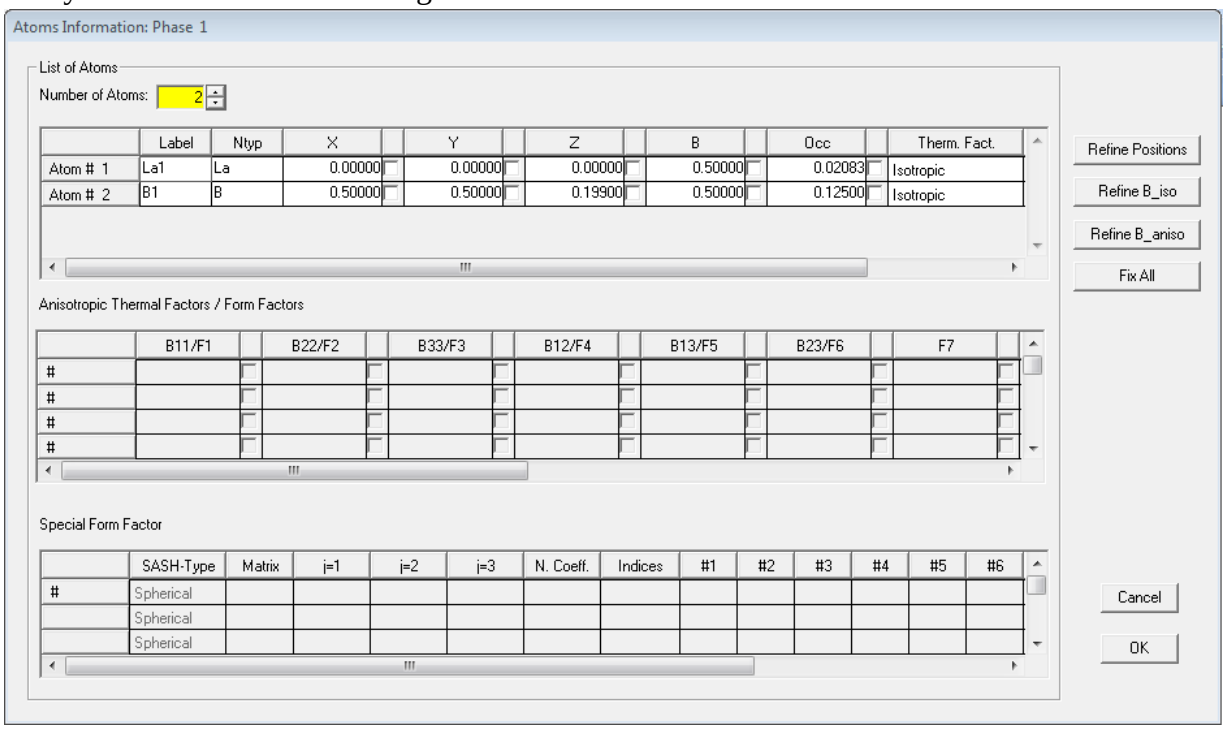
Here you need to tick-off the "Scale" to perform the scale factor refinement.
After this click "OK" and "OK" to come back to the main manu, where you can start the refinement by clicking  .

2) Refinement of unit cell parameters – again go into the profile menu and tick off the unit cell parameters:



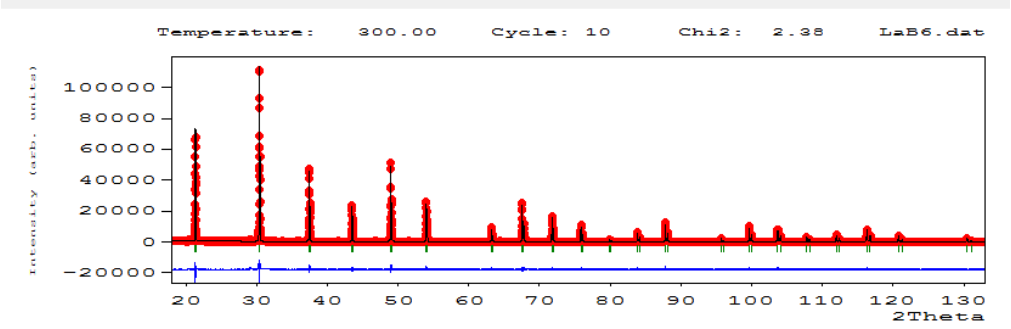
Again click  to refine the data.

3) The last step is refinement of the peak profile – you therefore have to go back into the "profile" and this time you need to tick-off, W and X. with these parameters refined you should have a pretty good fit of the data.
4) If we now wish to improve the fit further we have to refine the atomic coordinates and the thermal vibration. This is done by entering Refinement and clicking on Atoms now you should see the following window:



Here you click on "Refine Positions" and "Refine B_iso". After this you go back and click the refine bottom.

```
   => ---------> Pattern#       1
   => Phase:       1
   =>    Bragg R-factor:   3.269
   =>    RF-factor     :   2.374
=> Normal end, final calculations and writing...
```

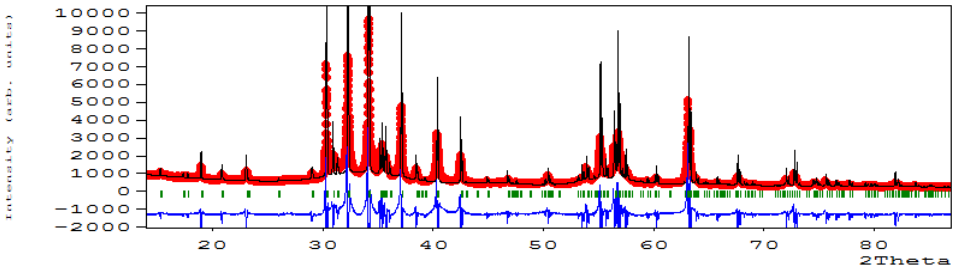


Example of $SrFe_{12}O_{19}$

Chose the correct library and select `SrFe12O19.pcr`

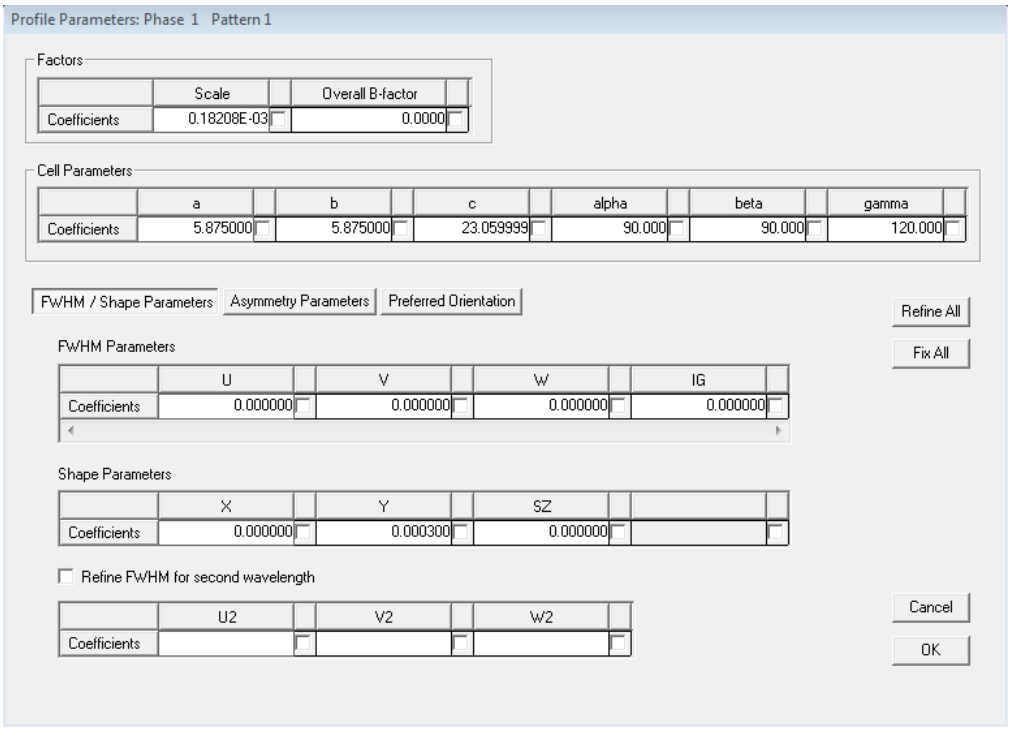
Currently there are no parameters being refined so clicking is almost the same as doing a model refinement. Please click the icon to see the calculation.

The following screen appears:

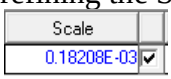


Clearly the parameters peaks are too sharp – actually they are intended to be very close to instrumental broadening. It is also clear that the intensities are too high – so there is a problem with the scale factor. By zooming it can also be seen that the unit cell is not completely correct. We need to address these problems one at the time: 1) refinement of the scale factor, 2) refinement of zero point and unit cell parameters, 3) initial refinement of the peak width.

1) Scale factor: Click [Refinement], in refinements click [Profile] now you will see the following screen:

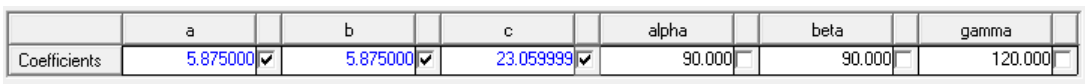


This screen actually holds a number of interesting parameters, but let us first starts refining the Scale factor. Therefore click:



After this click OK and OK – to come back to the main menu, where you can click  to start the refinements.

2) Refine unit cell parameters – these are also fund in the menu refinements, profile – click:



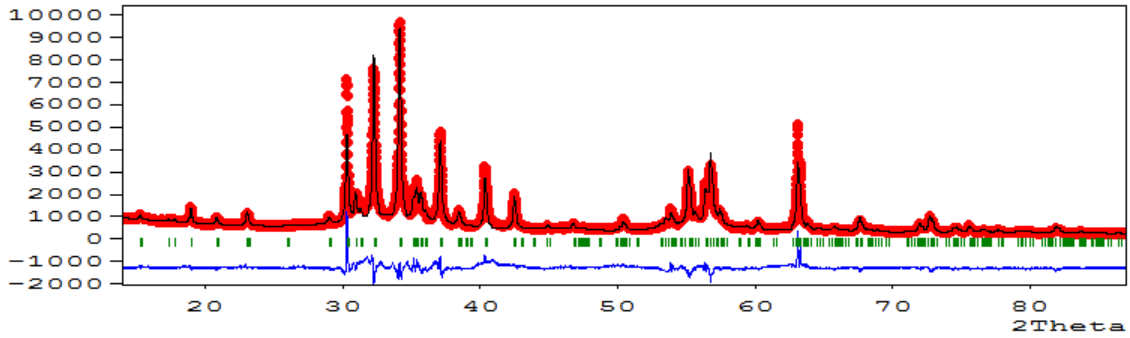
We also need to refine the zero point, which is found under "Refinements", "Instrument".

Go back to the main menu and click on refine  .

3) To really improve the fit we need to include the peak broadening

	X	Y	SZ	
Coefficients	0.000000 ☐	0.000 ☑	0.000000 ☐	☐

Again, refine the data. The fit should now improve and look like the data below – however the is a problem especially for the (110) and (220) reflections.



4) To improve the fit, we need to include an additional parameter that describes the morphology of the sample – i.e. the fact that the sample is larger along some crystallographic direction.

Therefore, go into "refinement", here you have to click Micro-Structure

In the microstructure menu you must click size – and chose: Platelets Vector Size

Strain | Size

Model: Platelets Vector Size ,

Here we wish the c-axis to be different from the a/b-axis, this is done by write the vector specifying the c-direction, which is (001)

	V1	V2	V3
Coefficients	0.00000	0.00000	1.00000

Now we have to go into "profile" and tick off the parameter "SZ"

Run the refinement!

All peak should now be relatively well described – you can now take a look at the refined sizes – this information is found in the *.mic file – open the *.mic file with a text editor and look for the (110) and (008) reflection, the size is given in Å:

```
!
!    s=1/d     HGo      HGi      HLo      HLi      betaG     betaL     beta     betaVo  App-size Max-strain   h   k   l     twtet     HpVo      eta
     0.1733   0.0440   0.0440   0.1923   0.0074   0.0000    3.2619   3.2619   3.5067    306.66   0.0000    0   0   4   15.3429   0.2024   0.9627
     0.1963   0.0440   0.0440   0.0799   0.0084   0.0000    1.2585   1.2585   1.6402    795.17   0.0000    1   0   0   17.3908   0.1002   0.8420
     0.2010   0.0440   0.0440   0.1047   0.0086   0.0000    1.6903   1.6903   2.0336    591.91   0.0000    1   0   1   17.8128   0.1215   0.8941
     0.2145   0.0440   0.0440   0.1270   0.0092   0.0000    2.0676   2.0676   2.3930    483.86   0.0000    1   0   2   19.0246   0.1414   0.9226
     0.2354   0.0440   0.0440   0.1452   0.0101   0.0000    2.3647   2.3647   2.6875    423.06   0.0000    1   0   3   20.8942   0.1581   0.9384
     0.2599   0.0440   0.0440   0.1983   0.0112   0.0000    3.2619   3.2619   3.5674    306.66   0.0000    0   0   6   23.1018   0.2080   0.9647
     0.2618   0.0440   0.0440   0.1596   0.0113   0.0000    2.5845   2.5845   2.9151    387.06   0.0000    1   0   4   23.2708   0.1714   0.9478
     0.2923   0.0440   0.0440   0.1709   0.0127   0.0000    2.7432   2.7432   3.0880    364.67   0.0000    1   0   5   26.0253   0.1820   0.9538
     0.3257   0.0440   0.0440   0.1801   0.0142   0.0000    2.8574   2.8574   3.2208    350.09   0.0000    1   0   6   29.0616   0.1907   0.9579
     0.3399   0.0440   0.0440   0.0881   0.0149   0.0000    1.2585   1.2585   1.7274    795.17   0.0000    1   1   0   30.3601   0.1072   0.8626
     0.3466   0.0440   0.0440   0.2054   0.0152   0.0000    3.2619   3.2619   3.6272    306.66   0.0000    0   0   8   30.9698   0.2148   0.9669
     0.3508   0.0440   0.0440   0.1177   0.0154   0.0000    1.7533   1.7533   2.1875    570.64   0.0000    1   1   2   31.3555   0.1330   0.9122
     0.3612   0.0440   0.0440   0.1879   0.0159   0.0000    2.9404   2.9404   3.3251    340.20   0.0000    1   0   7   32.3131   0.1981   0.9611
```

The program has also created a file, which allow us to look at the structure at rotate the structure in space.

Use the Fullprof studio from the Fullprof toolbar.