OAK RIDGE National Laboratory

Using CW data and GSASII to solve magnetic structures (HB-2A data)

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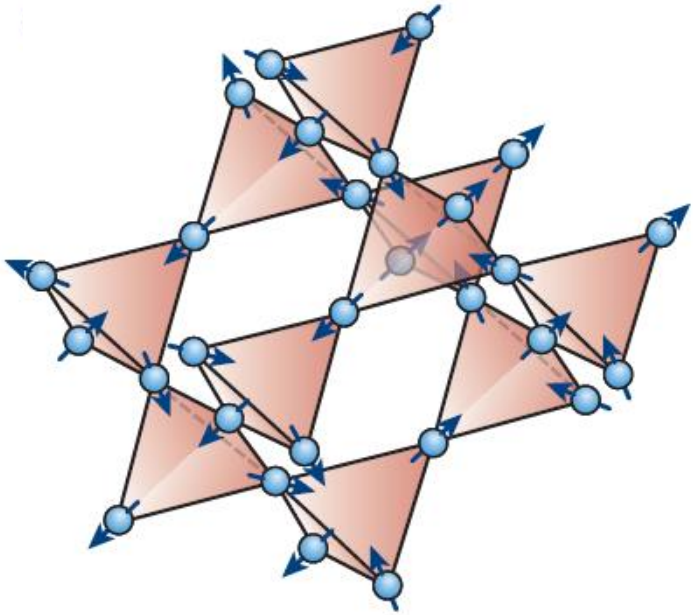
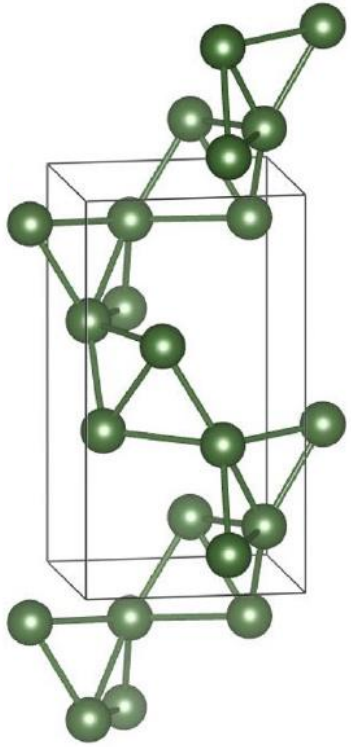
U.S. DEPARTMENT OF ENERGY

Starting point – study frustration in pyrogermanates

- We want to study systems with geometric frustration
- We'll take the pyrochlore as a starting point and study a related stoichiometry but with a tetragonal structure
- The Pyrogermanates have an interesting spiral triangular *RE* sub-lattice

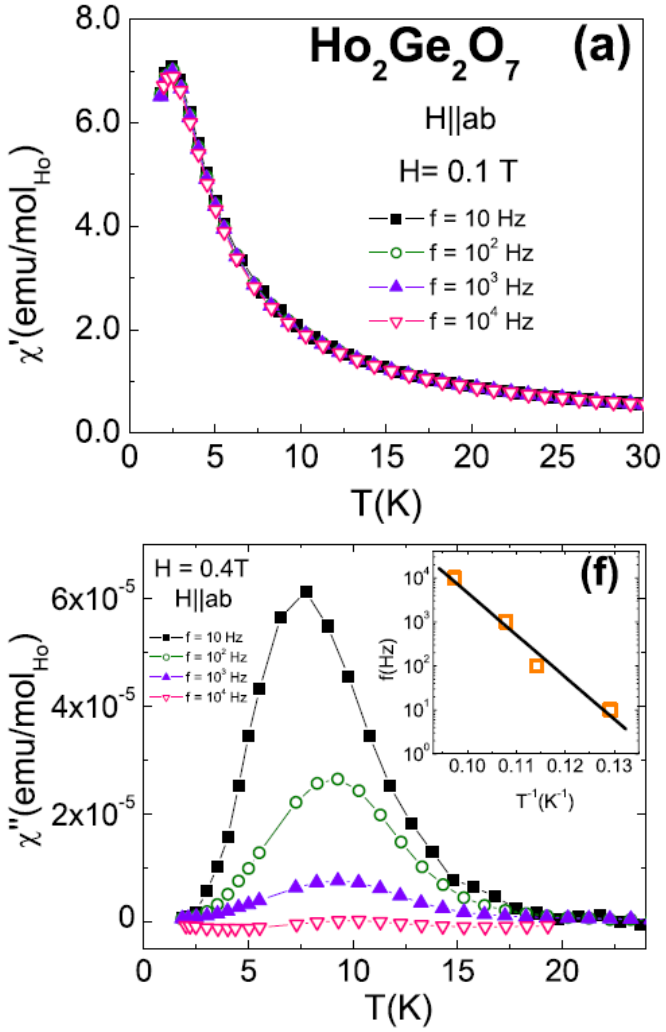


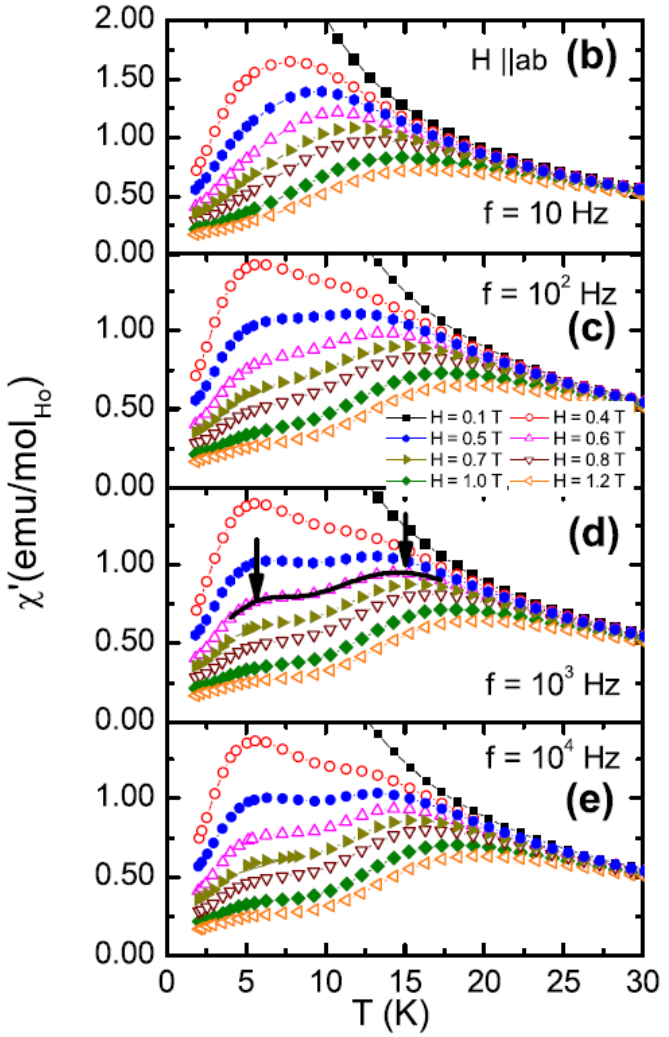
Pyrochlore Lattice:
$RE_2M_2O_7$
Corner sharing *RE* tetrahedra – geometric frustration

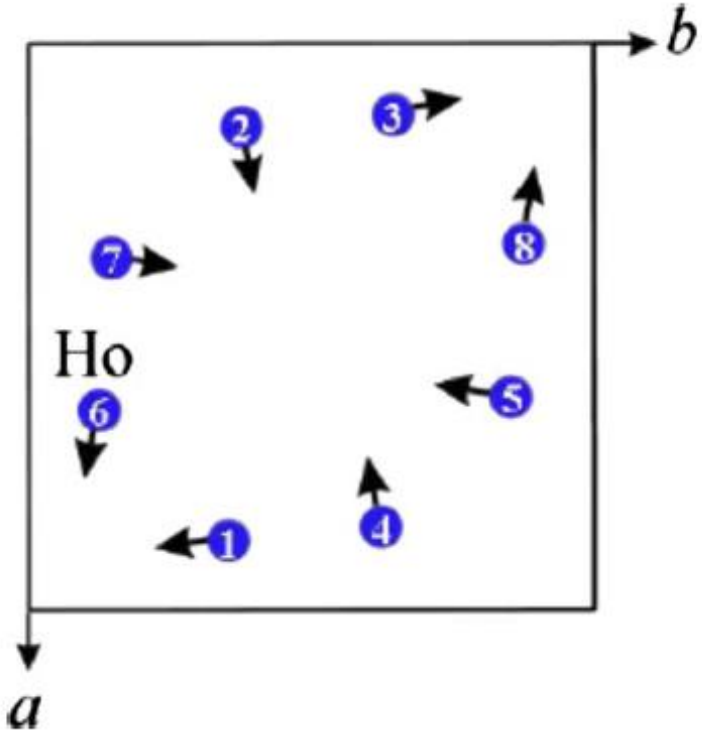


Pryogermanate Lattice:
$RE_2M_2O_7$
$P4_12_12$
Corner/edge sharing *RE* triangle spiral

Evidence of frustration in Ho and Dy pyrogermanates



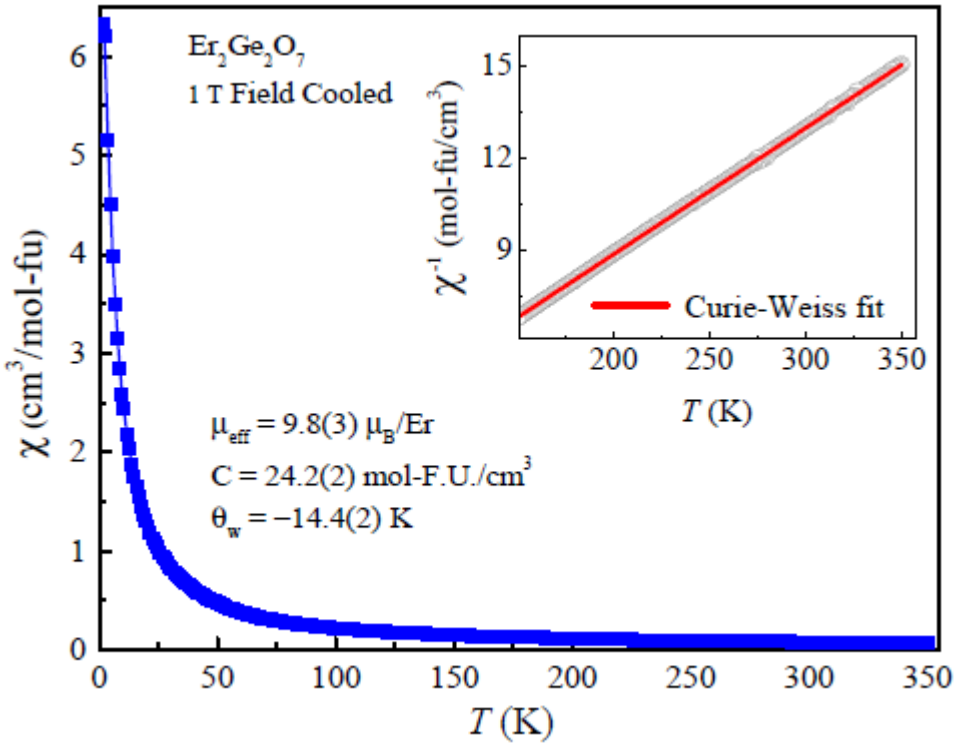




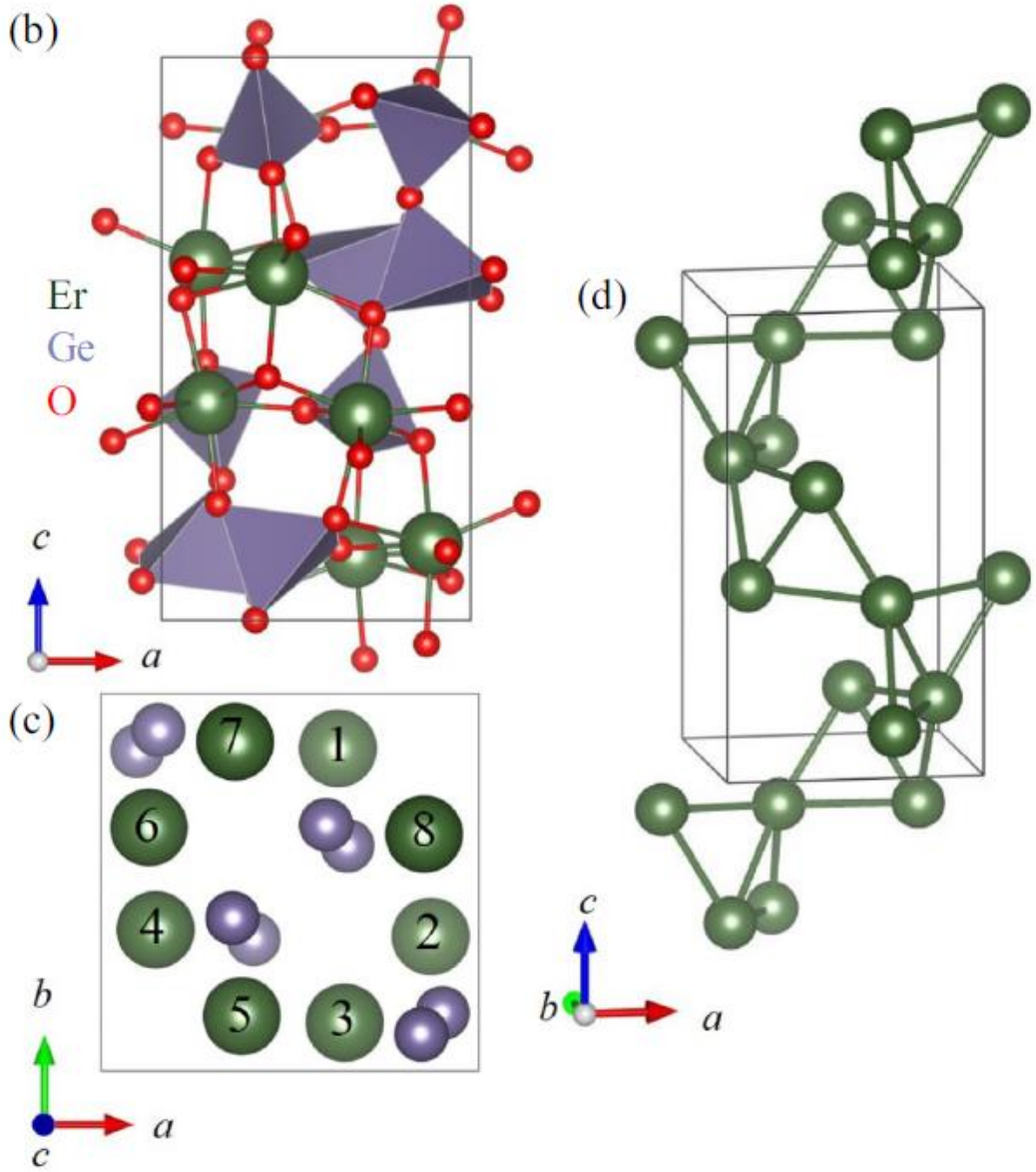
Similar frustration and spin-ice dynamics in $Dy_2Ge_2O_7$ and $Ho_2Ge_2O_7$

E. Morosan, et al,. PRB 2008
x. Ke, et al., PRB 2008

We'll look for similar behavior in $Er_2Ge_2O_7$

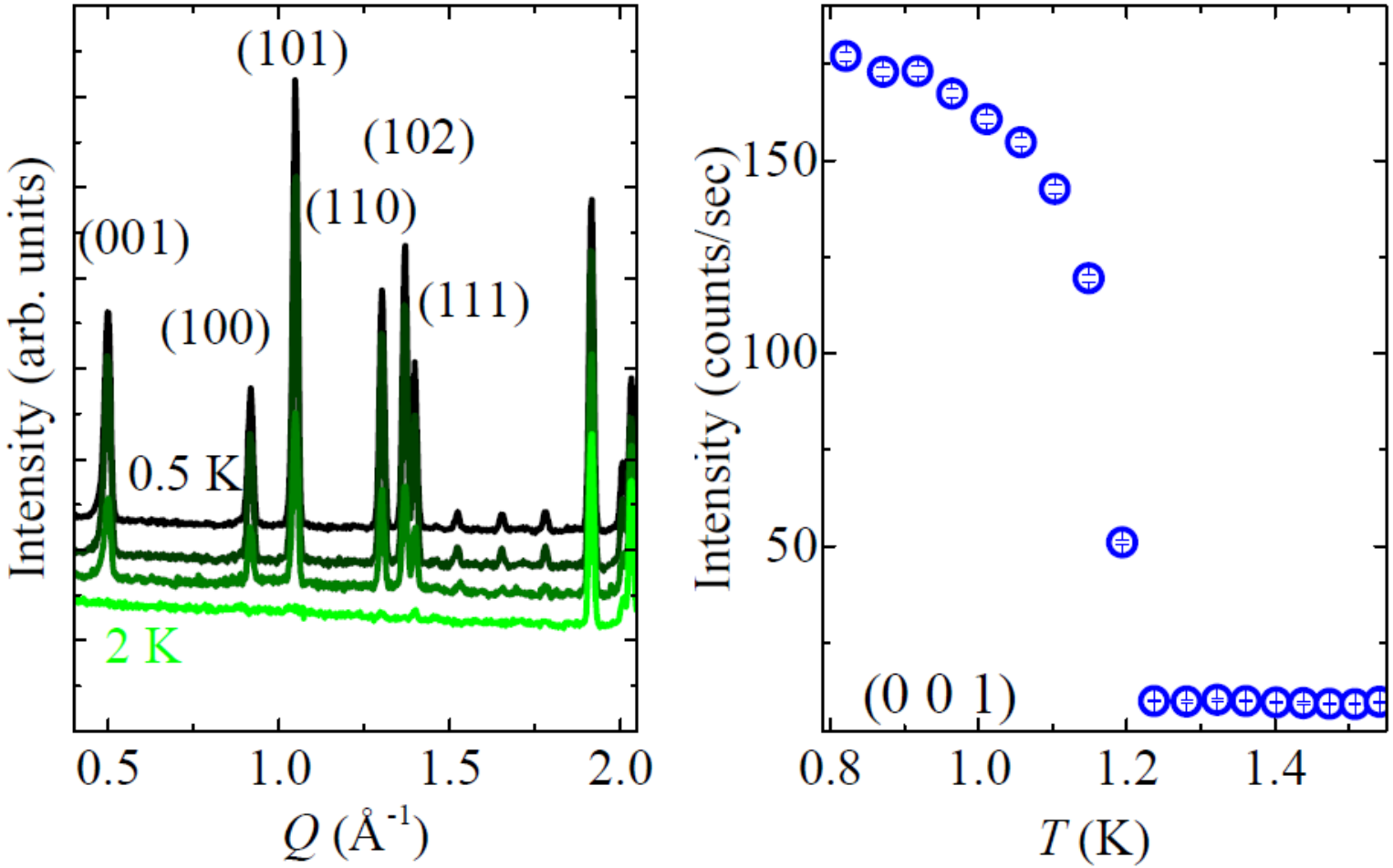


No evidence of magnetic order above 2 K, but we expect it from the Dy and Ho compounds



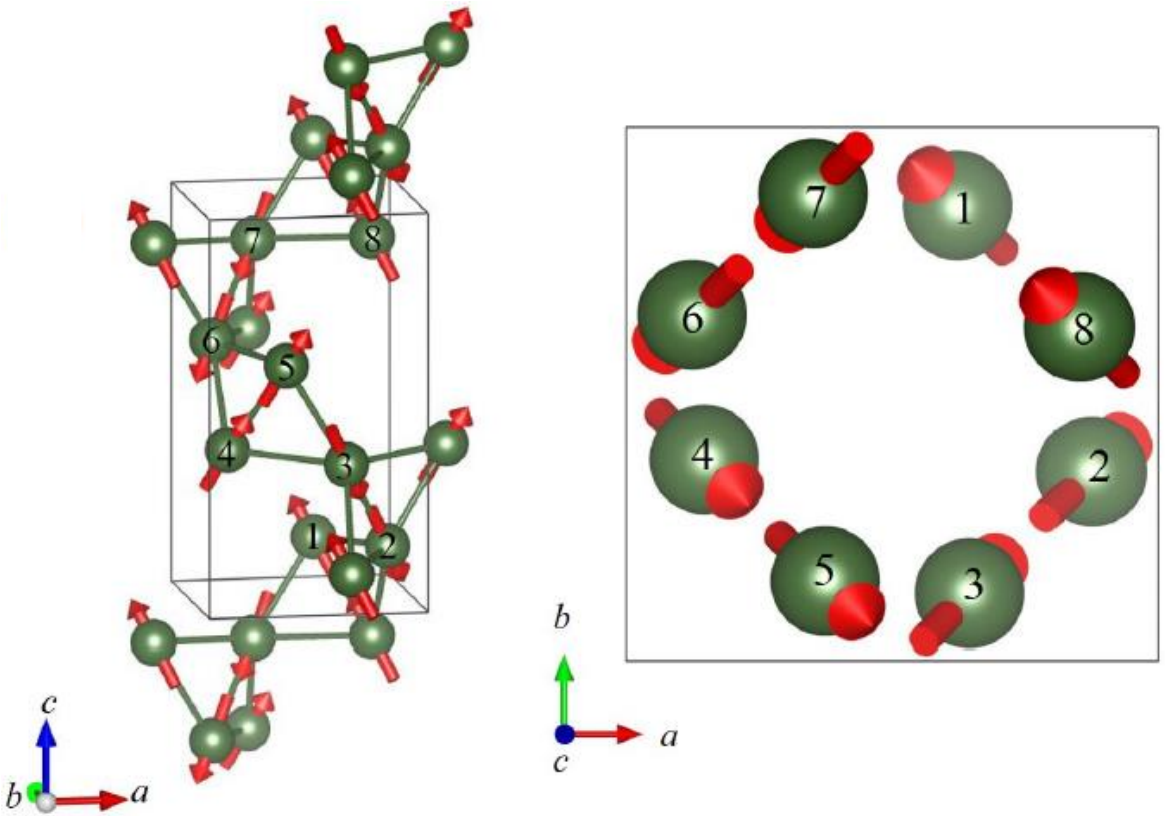
PRM 3, 014405 (2019)

The neutron results we'll look at

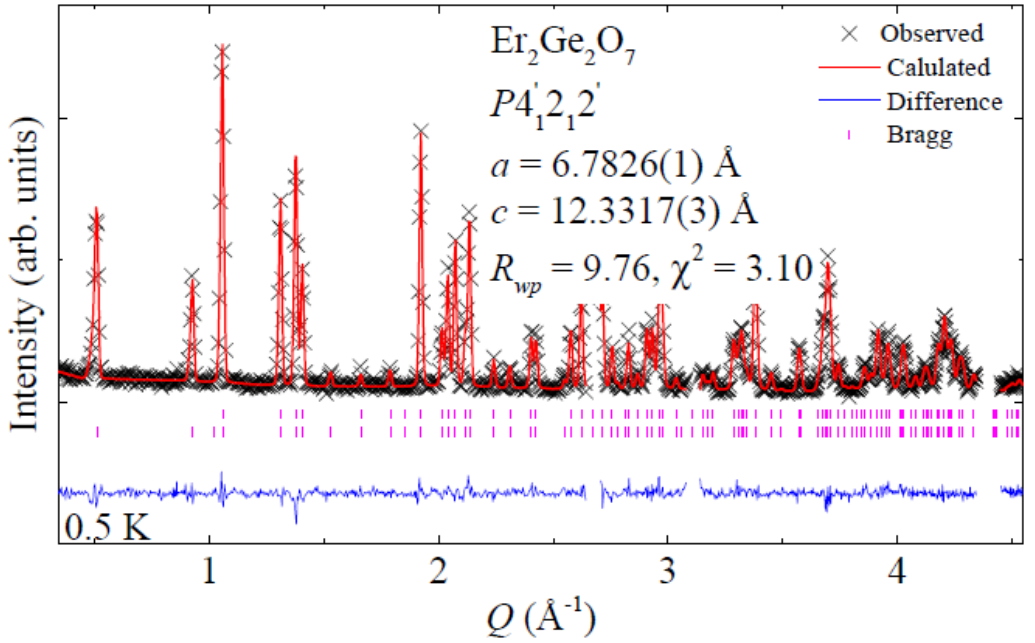


PRM 3, 014405 (2019)

The solved structure

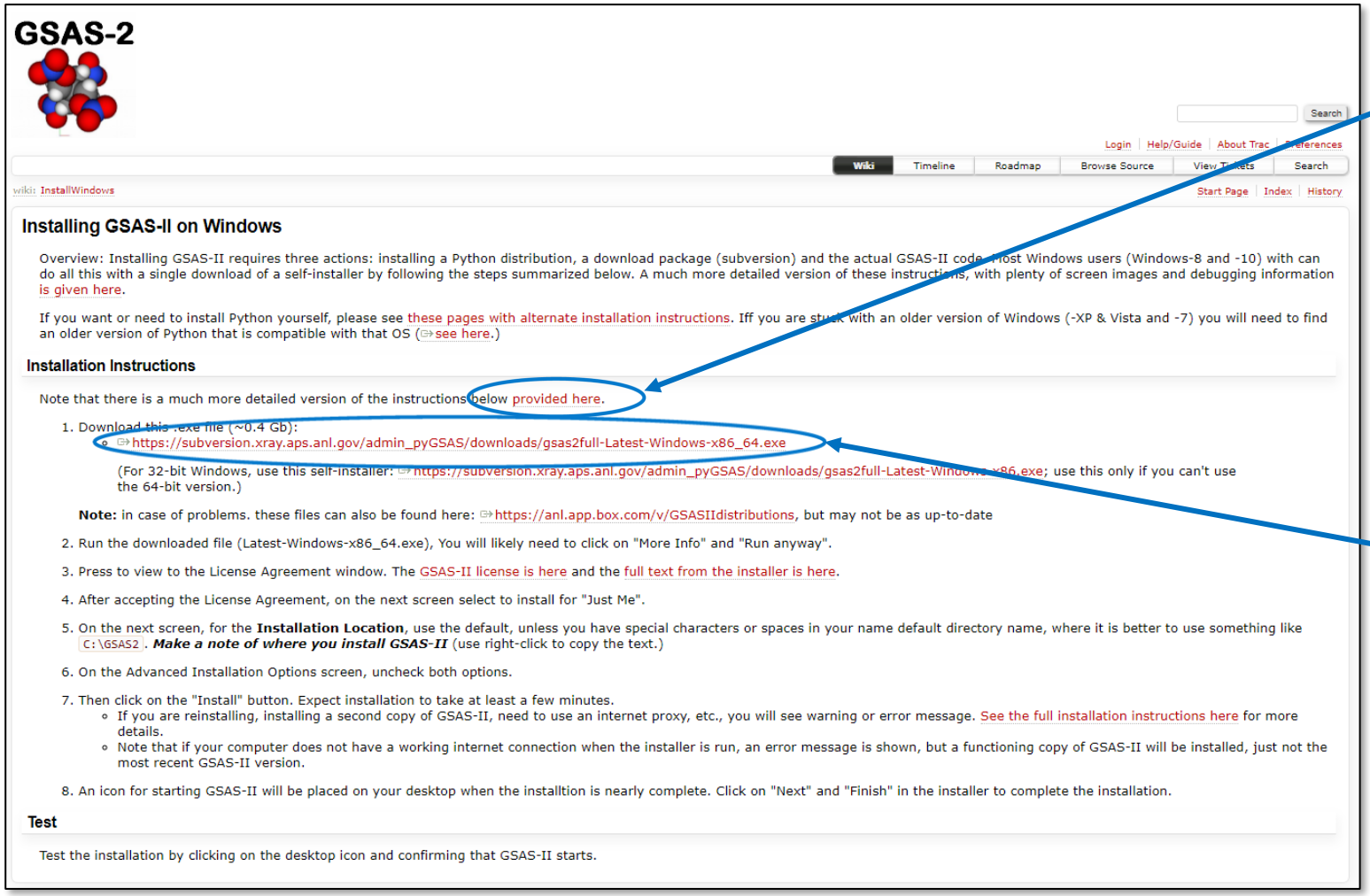


Γ	Magnetic space group	ψ	R_{wp}	χ^2
Γ_1 $(mGM1)$	$P4_12_12$	3	48.6	76.8
Γ_2 $(mGM2)$	$P4_1'2_12'$	3	9.76	3.10
Γ_3 $(mGM3)$	$P4_12_1'2'$	3	49.8	80.7
Γ_4 $(mGM4)$	$P4_12_1'2$	3	52.3	88.9
Γ_5 $(mGM5)$	$P2_1'2_1'2$	6	39.6	51.0
	$C22'2_1'$	6	40.4	53.3
	$P2_1'$	12		



PRM 3, 014405 (2019)

Installing GSAS-II



GSAS-2

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Installing GSAS-II on Windows

Overview: Installing GSAS-II requires three actions: installing a Python distribution, a download package (subversion) and the actual GSAS-II code. Most Windows users (Windows-8 and -10) with can do all this with a single download of a self-installer by following the steps summarized below. A much more detailed version of these instructions, with plenty of screen images and debugging information is given here.

If you want or need to install Python yourself, please see these pages with alternate installation instructions. Iff you are stuck with an older version of Windows (-XP & Vista and -7) you will need to find an older version of Python that is compatible with that OS (see here.)

Installation Instructions

Note that there is a much more detailed version of the instructions below provided here.

1. Download this .exe file (~0.4 Gb):
   - https://subversion.xray.aps.anl.gov/admin_pyGSAS/downloads/gsas2full-Latest-Windows-x86_64.exe

     (For 32-bit Windows, use this self-installer: https://subversion.xray.aps.anl.gov/admin_pyGSAS/downloads/gsas2full-Latest-Windows-x86.exe; use this only if you can't use the 64-bit version.)

   Note: in case of problems. these files can also be found here: https://anl.app.box.com/v/GSASIIdistributions, but may not be as up-to-date
2. Run the downloaded file (Latest-Windows-x86_64.exe), You will likely need to click on "More Info" and "Run anyway".
3. Press to view to the License Agreement window. The GSAS-II license is here and the full text from the installer is here.
4. After accepting the License Agreement, on the next screen select to install for "Just Me".
5. On the next screen, for the **Installation Location**, use the default, unless you have special characters or spaces in your name default directory name, where it is better to use something like `C:\GSAS2`. ***Make a note of where you install GSAS-II*** (use right-click to copy the text.)
6. On the Advanced Installation Options screen, uncheck both options.
7. Then click on the "Install" button. Expect installation to take at least a few minutes.
   - If you are reinstalling, installing a second copy of GSAS-II, need to use an internet proxy, etc., you will see warning or error message. See the full installation instructions here for more details.
   - Note that if your computer does not have a working internet connection when the installer is run, an error message is shown, but a functioning copy of GSAS-II will be installed, just not the most recent GSAS-II version.
8. An icon for starting GSAS-II will be placed on your desktop when the installtion is nearly complete. Click on "Next" and "Finish" in the installer to complete the installation.

Test

Test the installation by clicking on the desktop icon and confirming that GSAS-II starts.

Detailed installation instructions

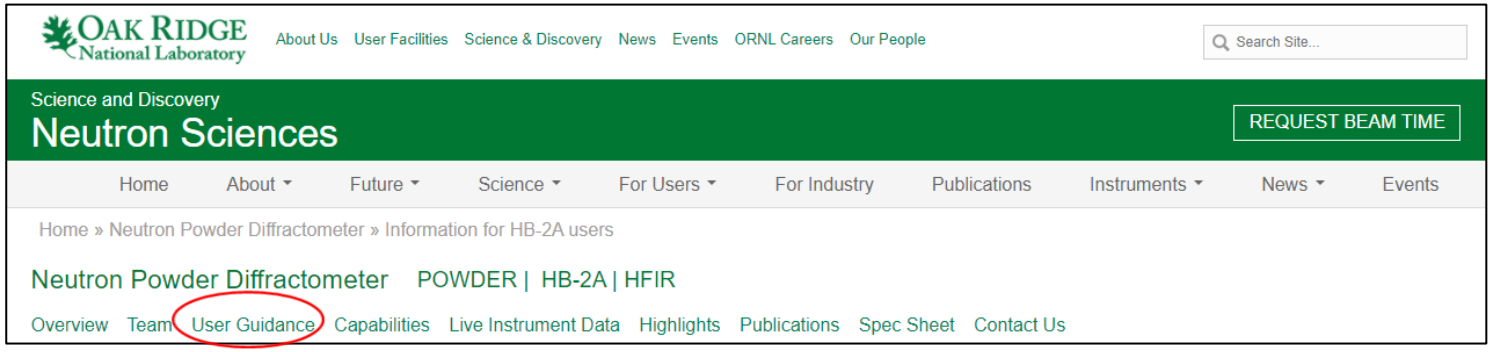
Executable

https://subversion.xray.aps.anl.gov/trac/pyGSAS/wiki/InstallWindows

At: https://subversion.xray.aps.anl.gov/trac/pyGSAS instructions for other installs (including via Anaconda) can be found

Before starting your refinement:

- Be sure to have
  - Data file
    - From HB-2A: *file_name*.gss
    - From P3G: *file_name*.gsa
  - Instrument parameter file
    - From HB-2A: *file_name_inst_settings*.instprm
    - From P3G: *file_name_cycle_inst_settings*.instprm
  - Crystallographic information file (.cif)



HB-2A

Calibration Standards

- Si_cycle451 (FullProf and GSAS)
- Lab6_cycle451 (FullProf)
- NAC (Na2Al2Ca3F14) (FullProf)

POWGEN

Data Reduction and Analysis by Run Cycle

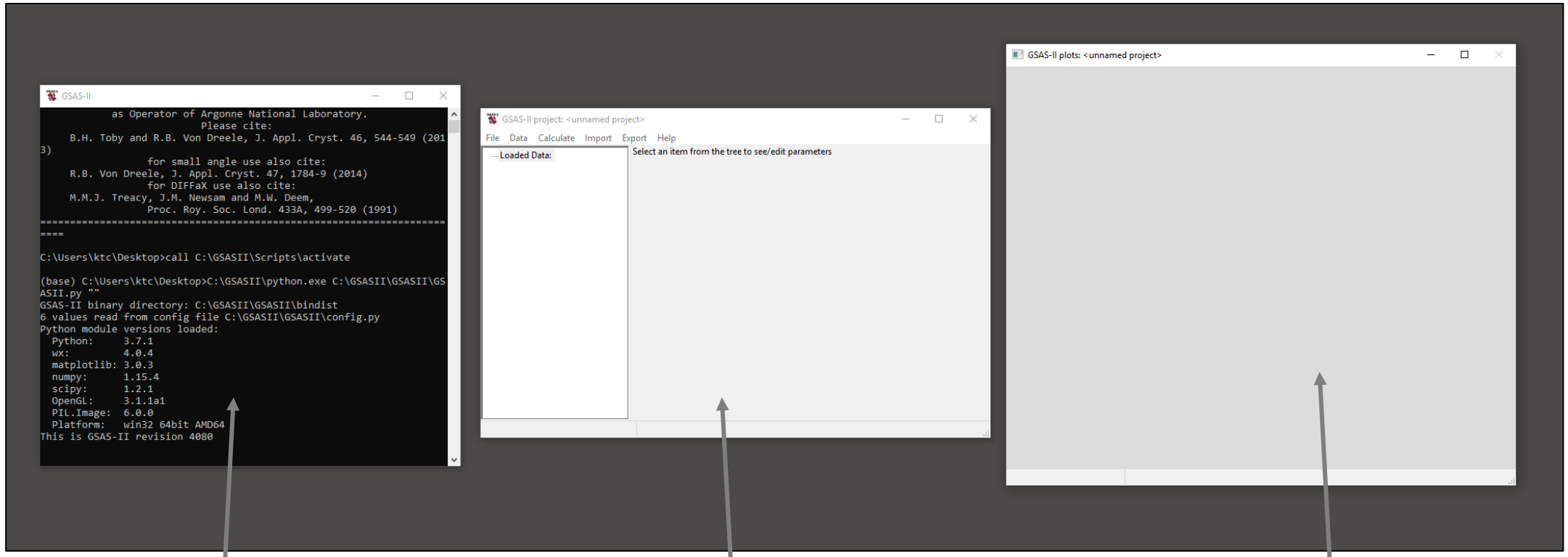
Parameter files for GSAS, GSAS-II, Fullprof and Topas can be downloaded from the table below. Be sure to pick the correct cycle. Also these files are stored in the **/SNS/PG3/run_cycle_11A_CAL** folder in the analysis computer where cycle is year_1 or year_2 based on cycle A or B.

Data will be automatically reduced into the following formats:

- Extension .gsa for GSAS and GSAS-II: Files are numbered with banks which correspond to the wavelength used. Please use the corresponding bank # from the parameter files for refinements.
- Extension .dat for fullprof: The data file names themselves tell you which bank they are and the same bank IRF file should be used.
- Extension .xye for topas: Please refer to the Topas example below.

Cycle	GSAS	GSAS-II	Fullprof	Topas	PDF	Manuals
2020A (May-)		GSASII_2020A-may_HR.zip	Fullprof_2020A-may_HR.zip		PDF_2020A-may.zip	
2020-A (Jan-Feb)		GSASII_2020A.zip	Fullprof_2020A_HR.zip	TOPAS_2020A.zip	PDF_2020A_.zip	Data Reduction Data Acquisition
2019-B	GSAS.prm.zip	GSASII_2019B_HR.zip	Fullprof_2019B_HR.zip	Topas_2019B_HR.zip Topas_2019B_HI.zip	POWGEN_PDF_2019B	Data Reduction Data Acquisition
2019-A	GSAS.prm	GSAS-II-HighRes.instprm GSAS-II-HighInt.instprm	Fullprof_HighRes.irf Fullprof_HighInt.irf	Topas_Highres.inp Topas_HighInt.inp	Topas_and PDFGui_fit	Data Reduction Data Acquisition
2018-B	GSAS.prm	GSAS-II-HighRes.instprm GSAS-II-HighInt.instprm	Fullprof_HighRes.irf Fullprof_HighInt.irf	Topas_Highres.inp Topas_HighInt.inp	Topas_fit PDFGui_Fit	Data Reduction Data Acquisition

Starting up GSAS-II

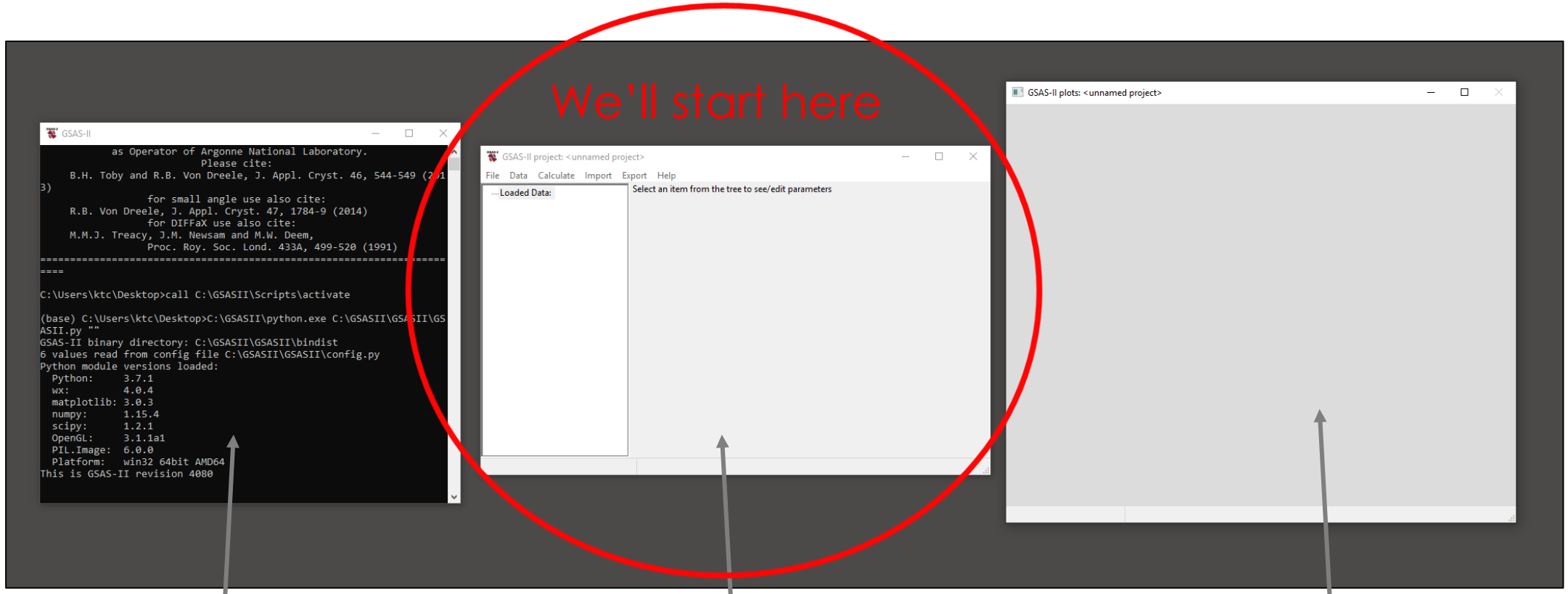


Python window (won't use here)

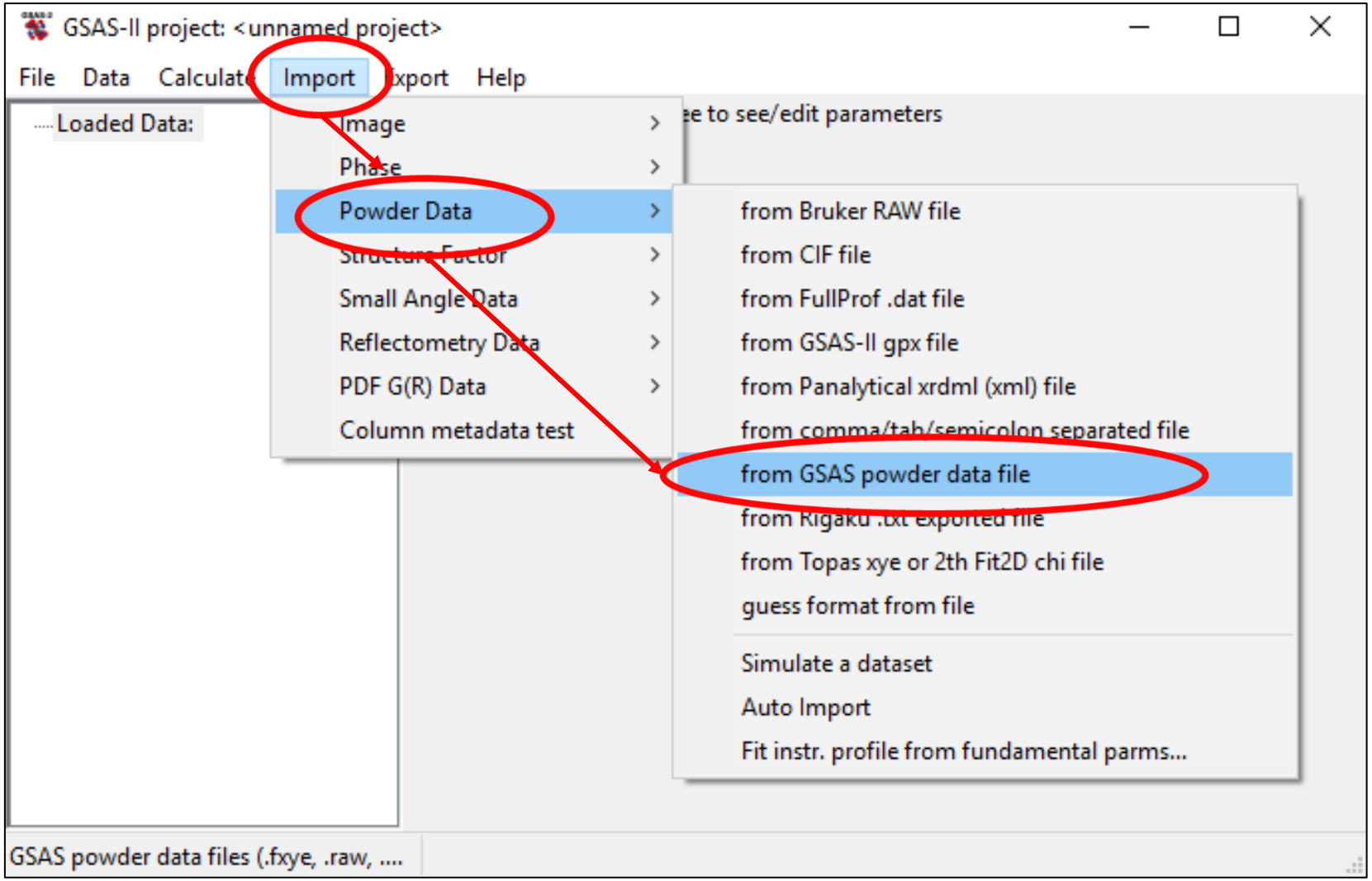
GSAS-II GUI (where we'll setup the data, phase information and run refinements)

Plotting window (automatically plots based on what you do in the GUI)

Starting up GSAS-II

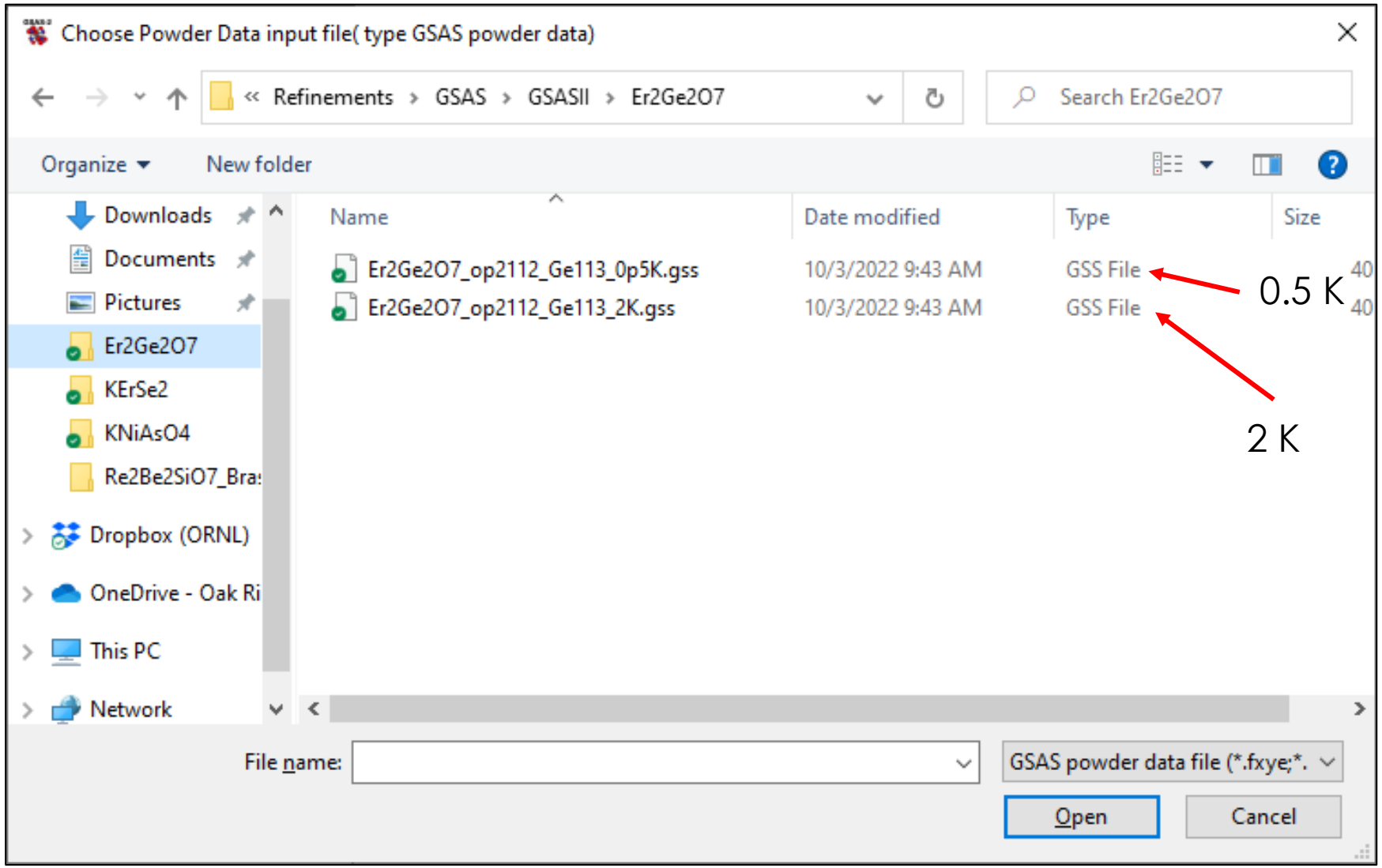


Loading your data



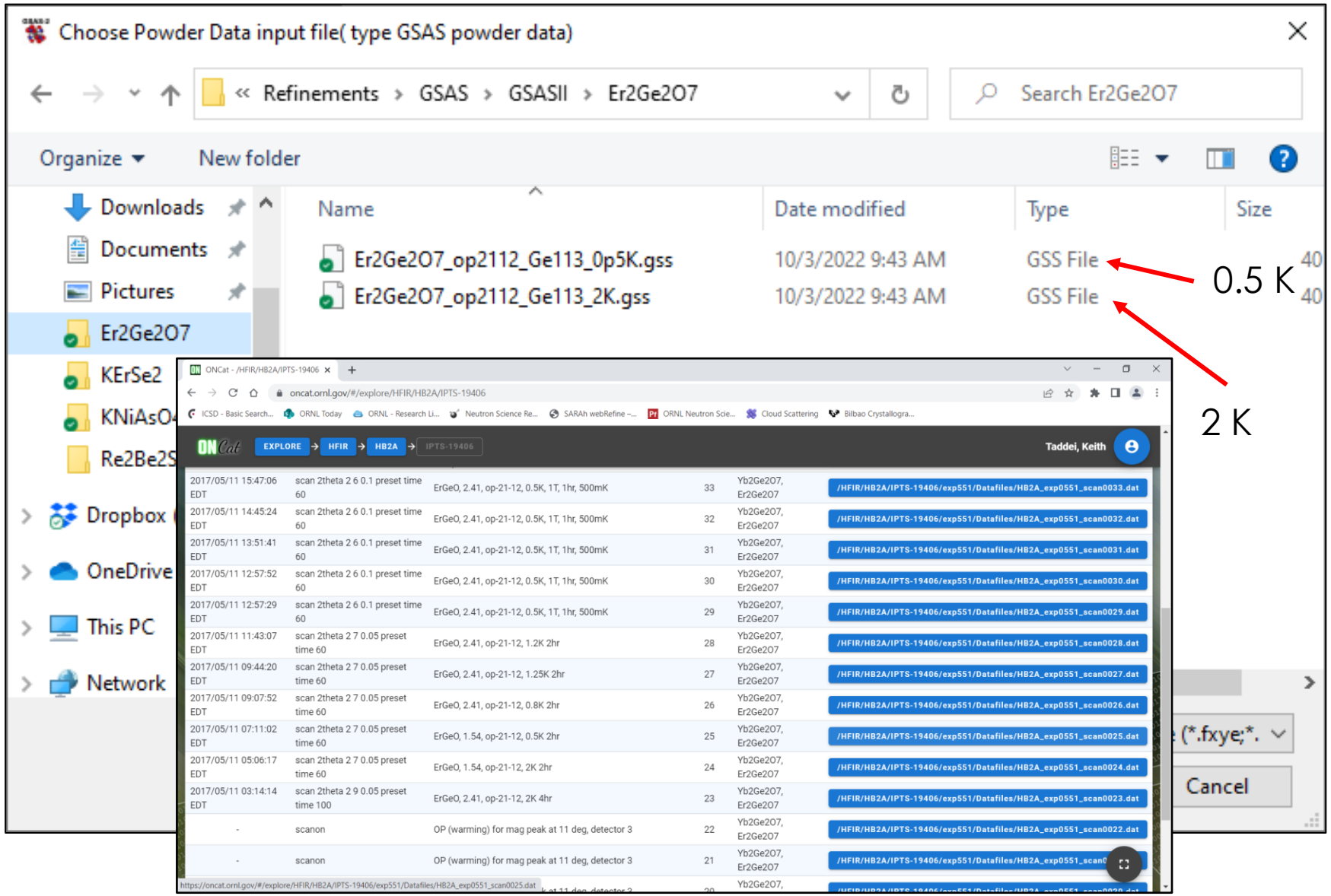
Loading your data

NB: I've renamed these files for convenience. Coming straight from the instrument they'll be named 'HB2A_exp###_scan00##.gss.

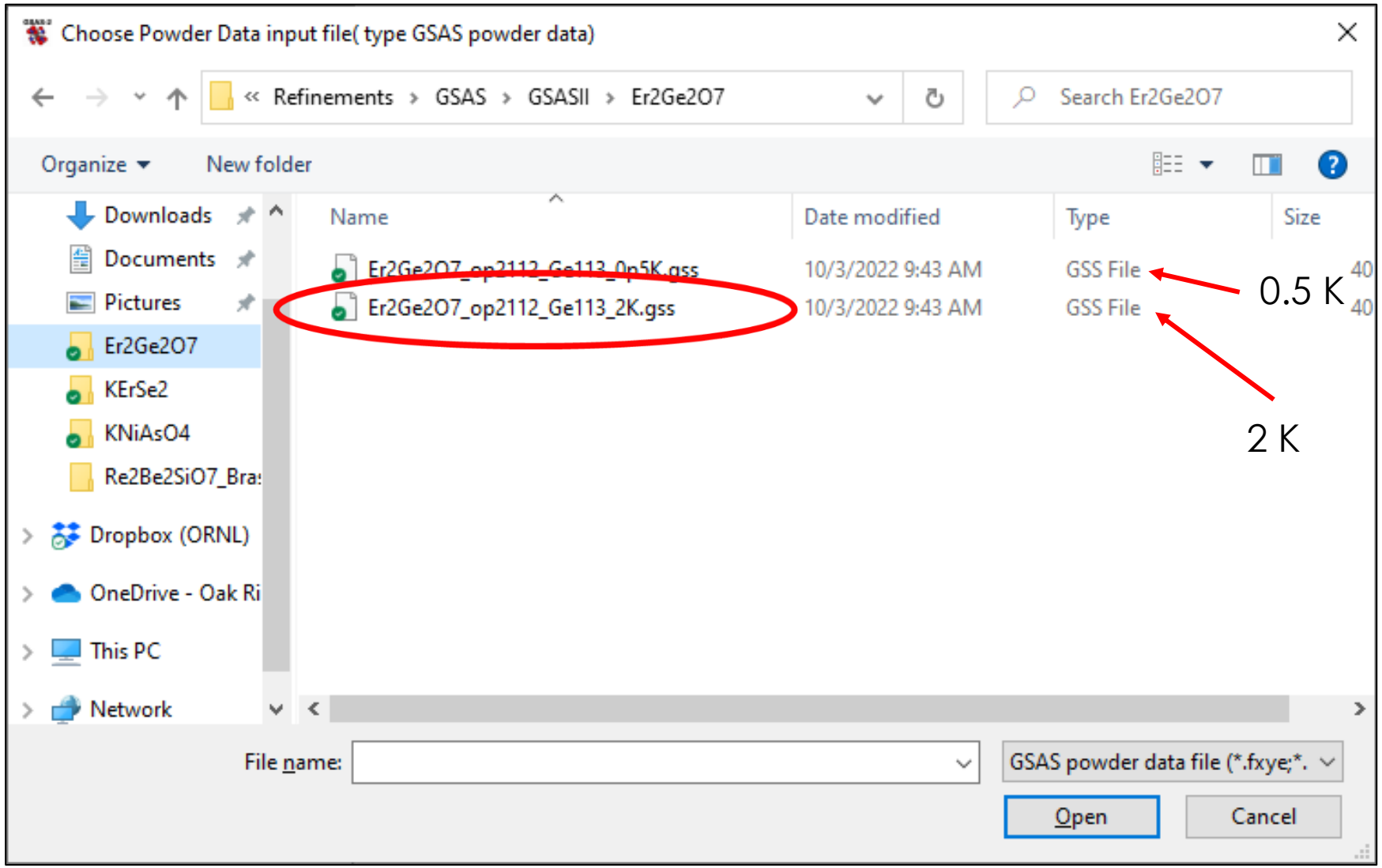


Loading your data

NB: I've renamed these files for convenience. Coming straight from the instrument they'll be named 'HB2A_exp###_scan00##.gss.

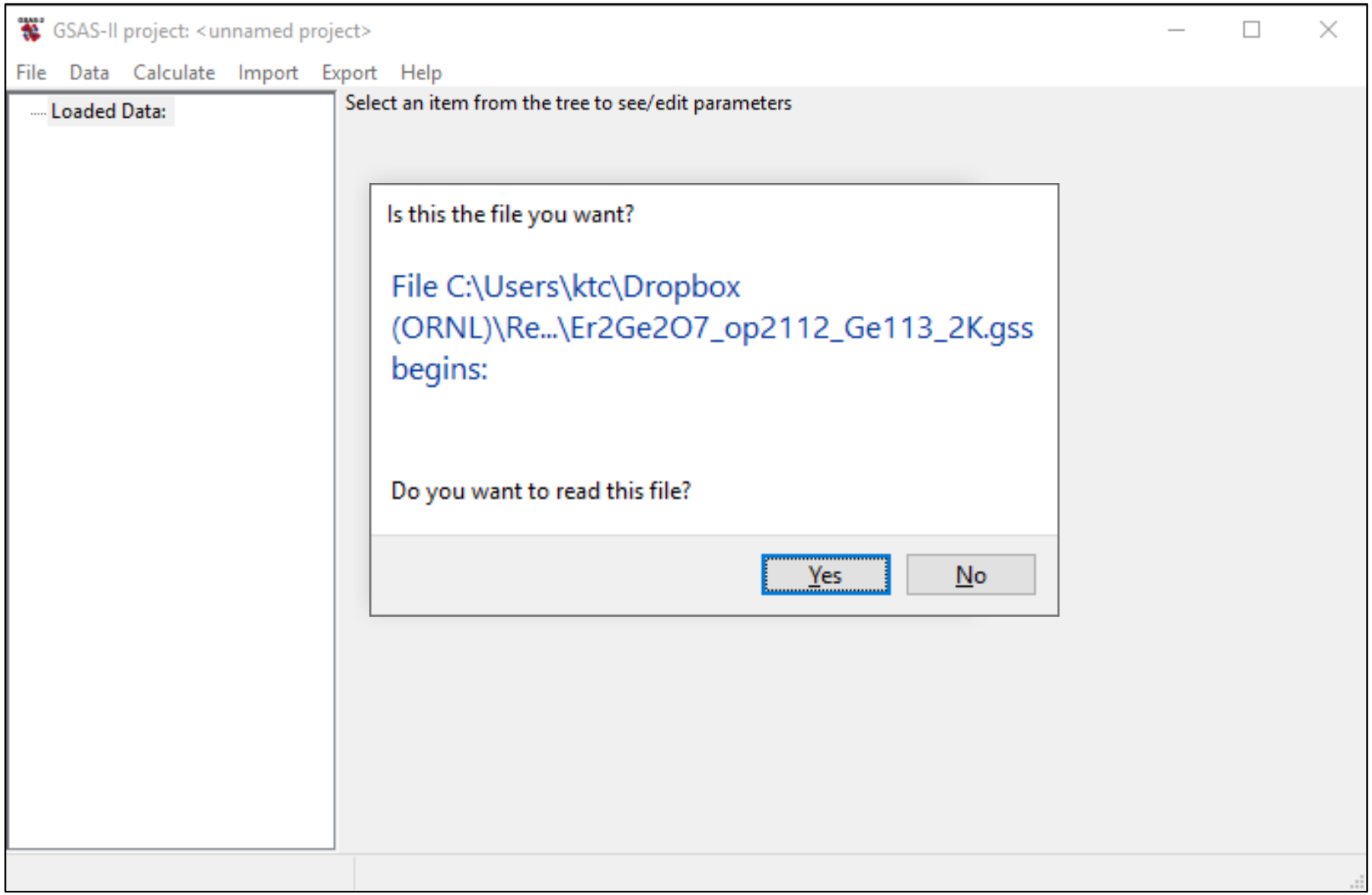


Loading your data



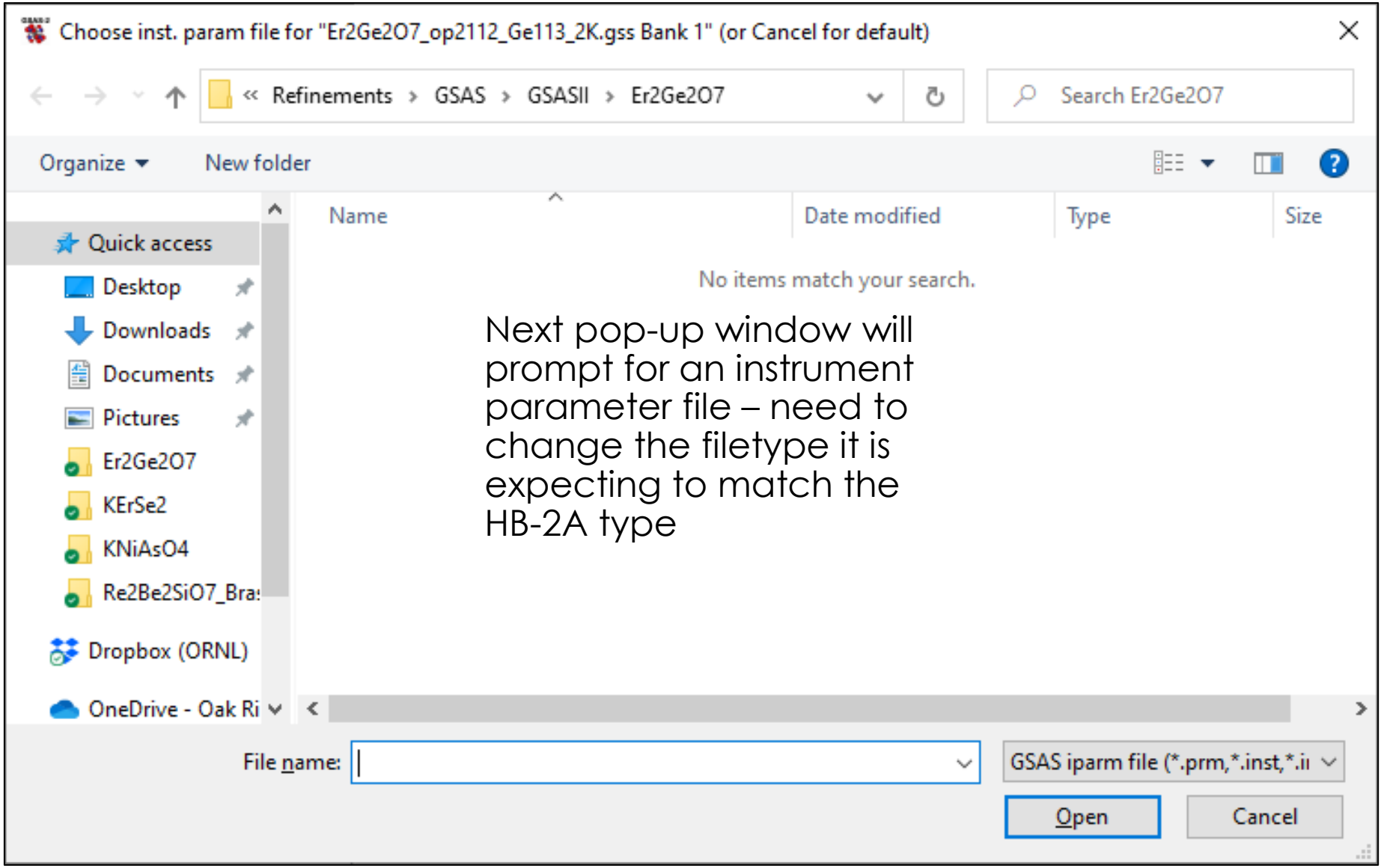
We'll start here

Loading your data



This window should pop-up
Click 'Yes'

Loading your data



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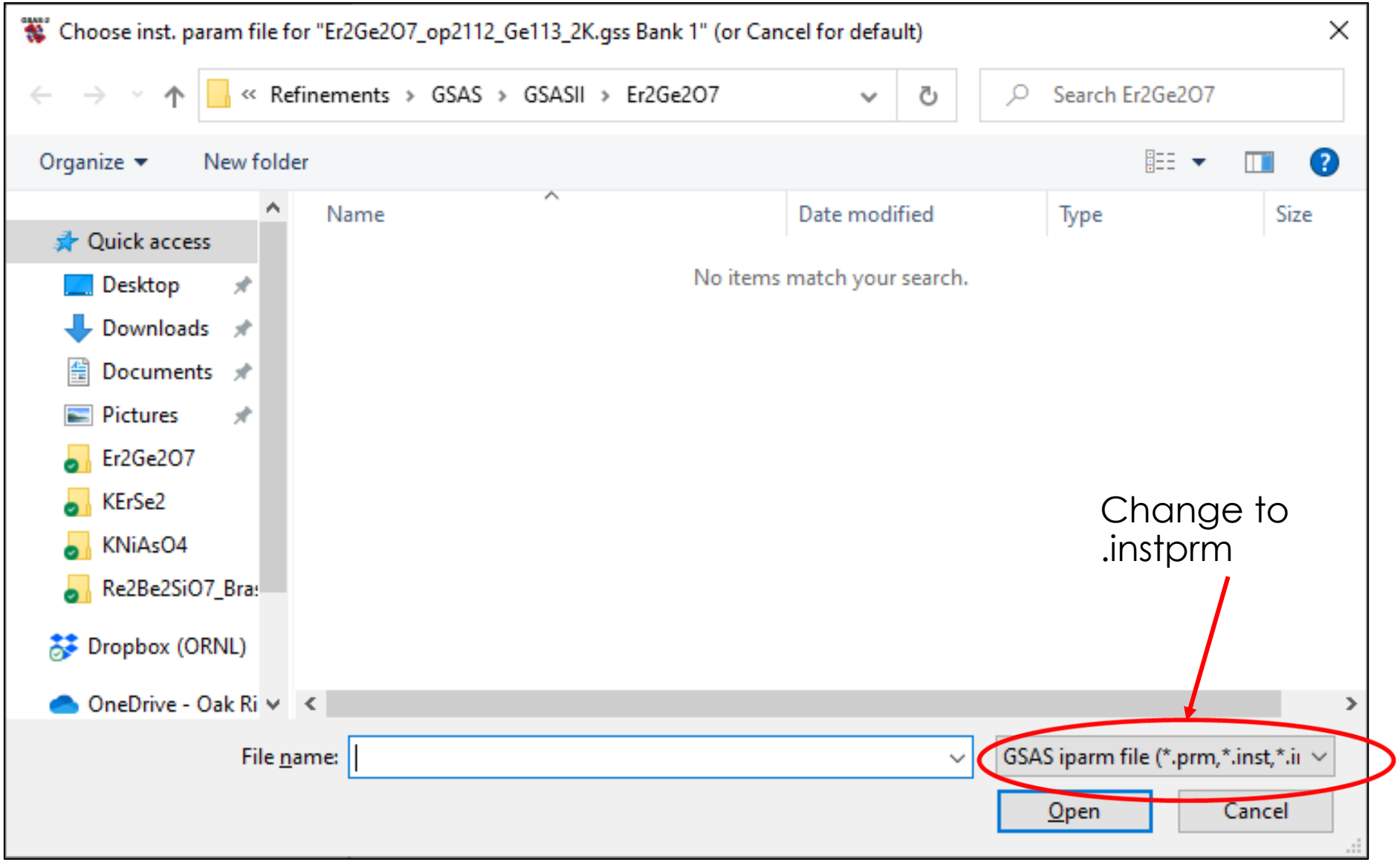

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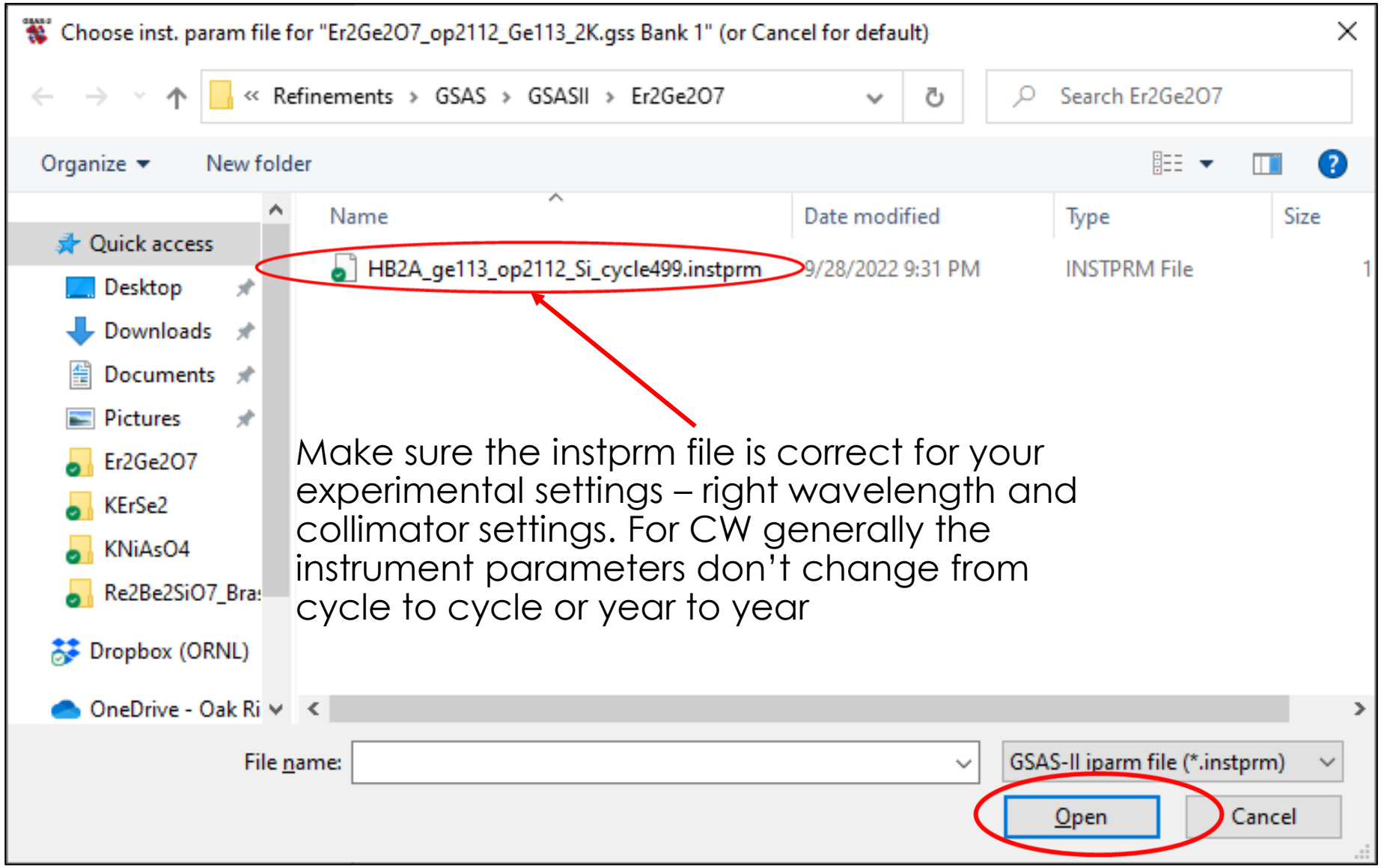
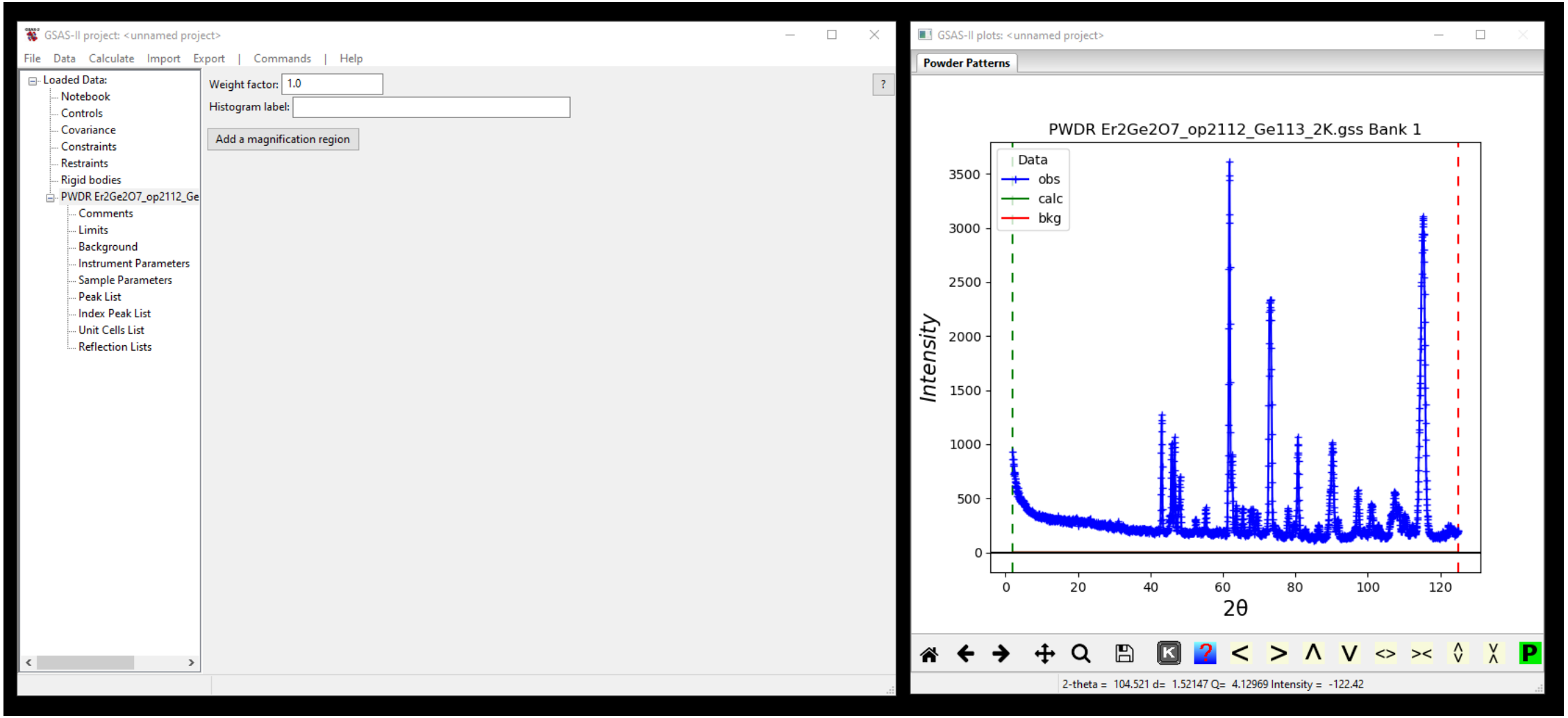
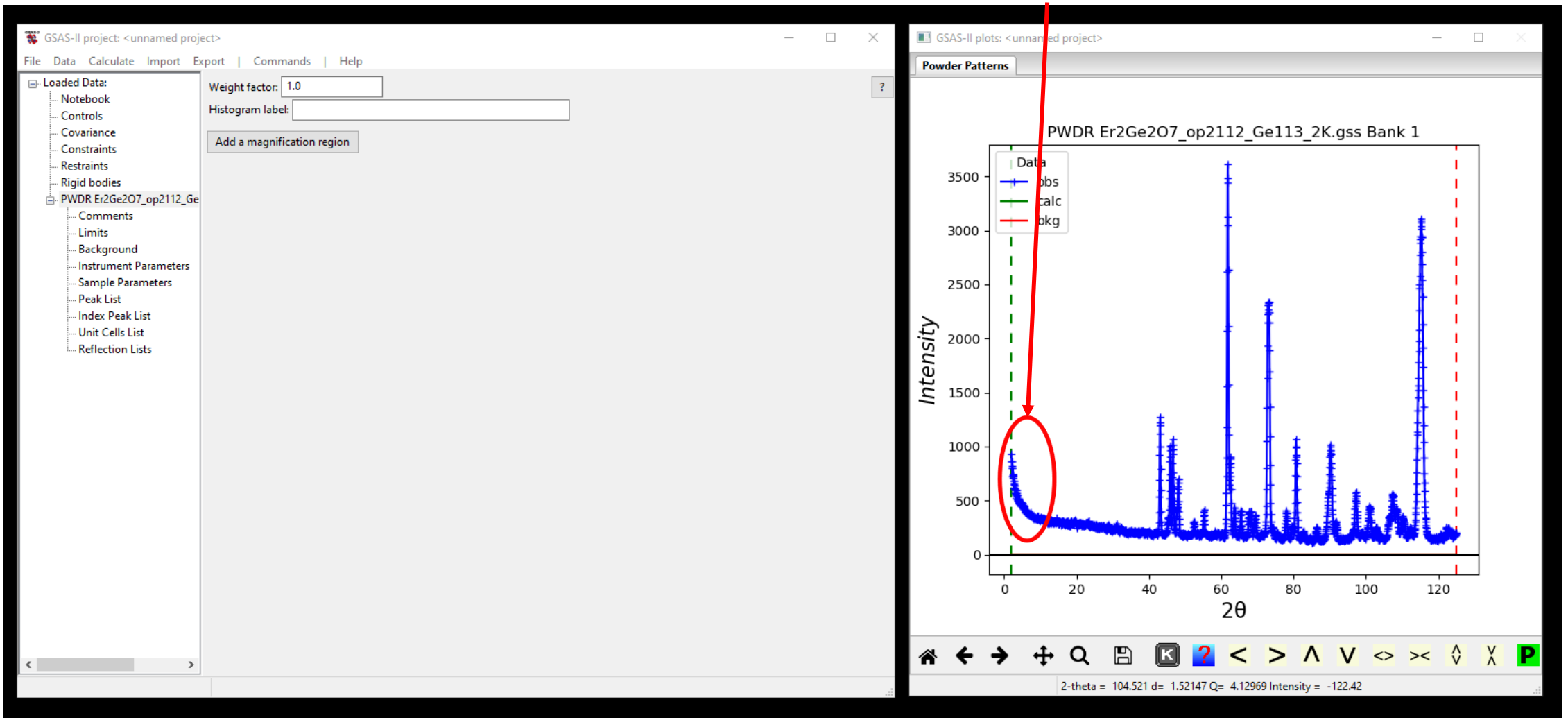


Data loaded


GSAS-II project: <unnamed project>
File Data Calculate Import Export | Commands | Help
Loaded Data:
Notebook
Controls
Covariance
Constraints
Restraints
Rigid bodies
PWDR Er2Ge2O7_op2112_Ge
Comments
Limits
Background
Instrument Parameters
Sample Parameters
Peak List
Index Peak List
Unit Cells List
Reflection Lists
Weight factor: 1.0
Histogram label:
Add a magnification region
GSAS-II plots: <unnamed project>
Powder Patterns
PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1
Data
obs
calc
bkg
Intensity
2θ
2-theta = 104.521 d= 1.52147 Q= 4.12969 Intensity = -122.42

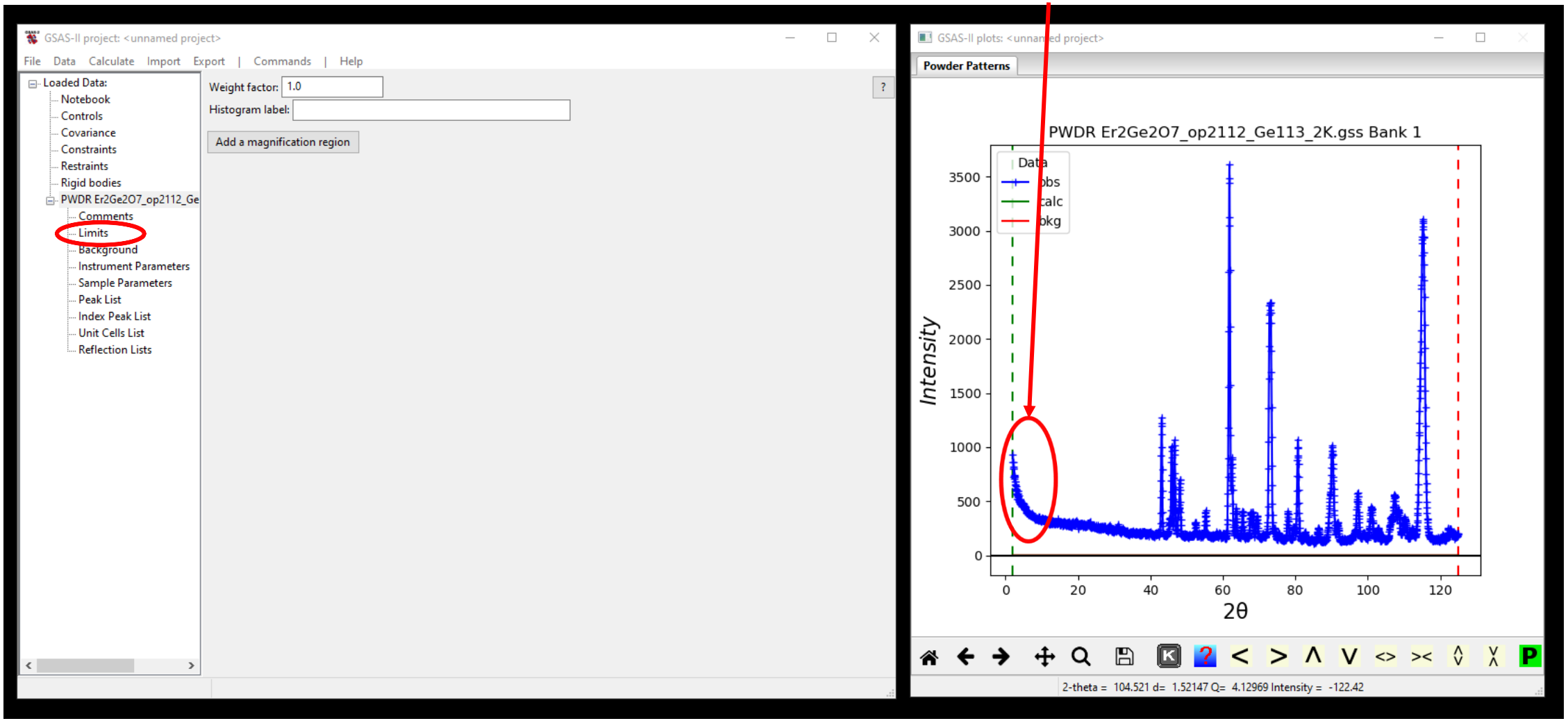
Excluding regions

Need to exclude this region of the data (only do this once sure there will be no magnetic peaks there)



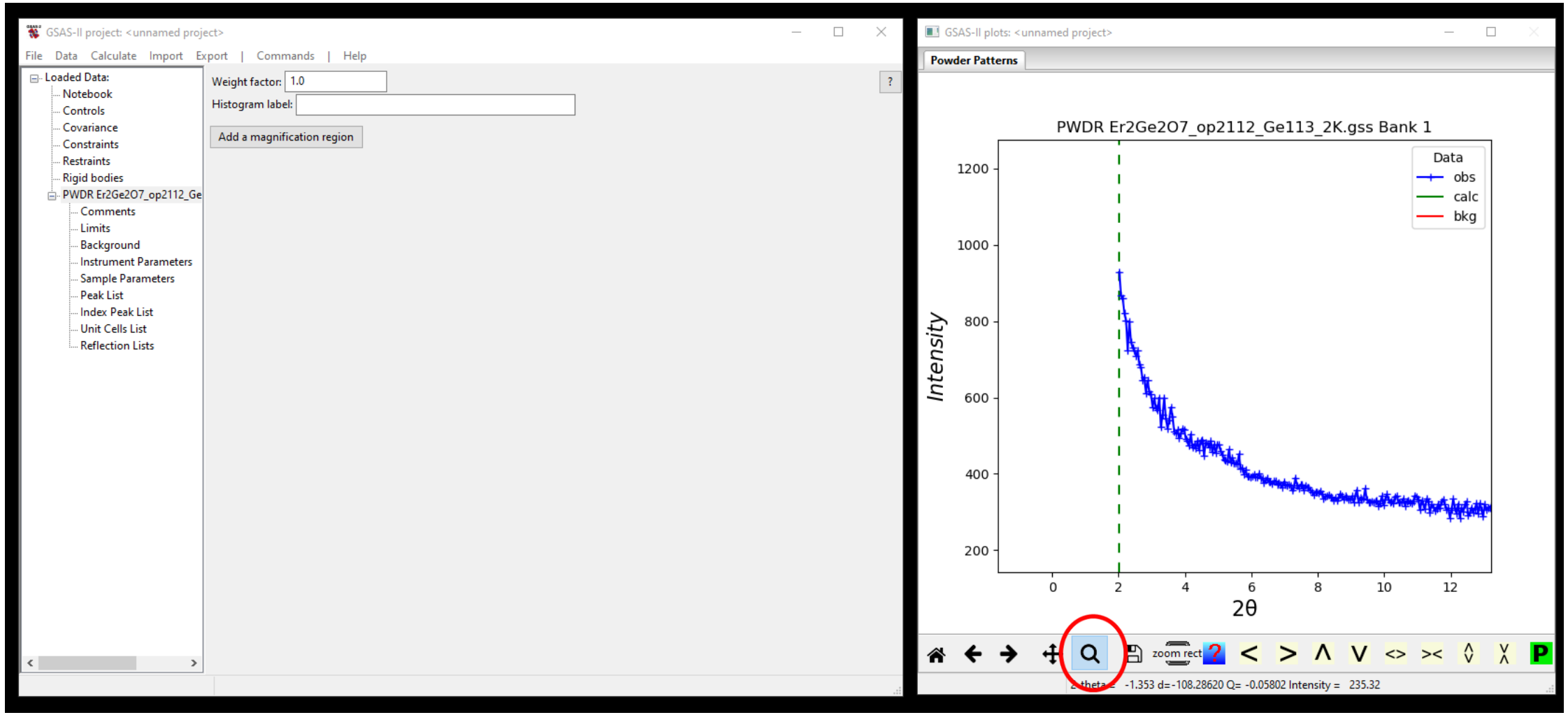
Excluding regions

Need to exclude this regions of the data – went to low angle contaminated by direct beam



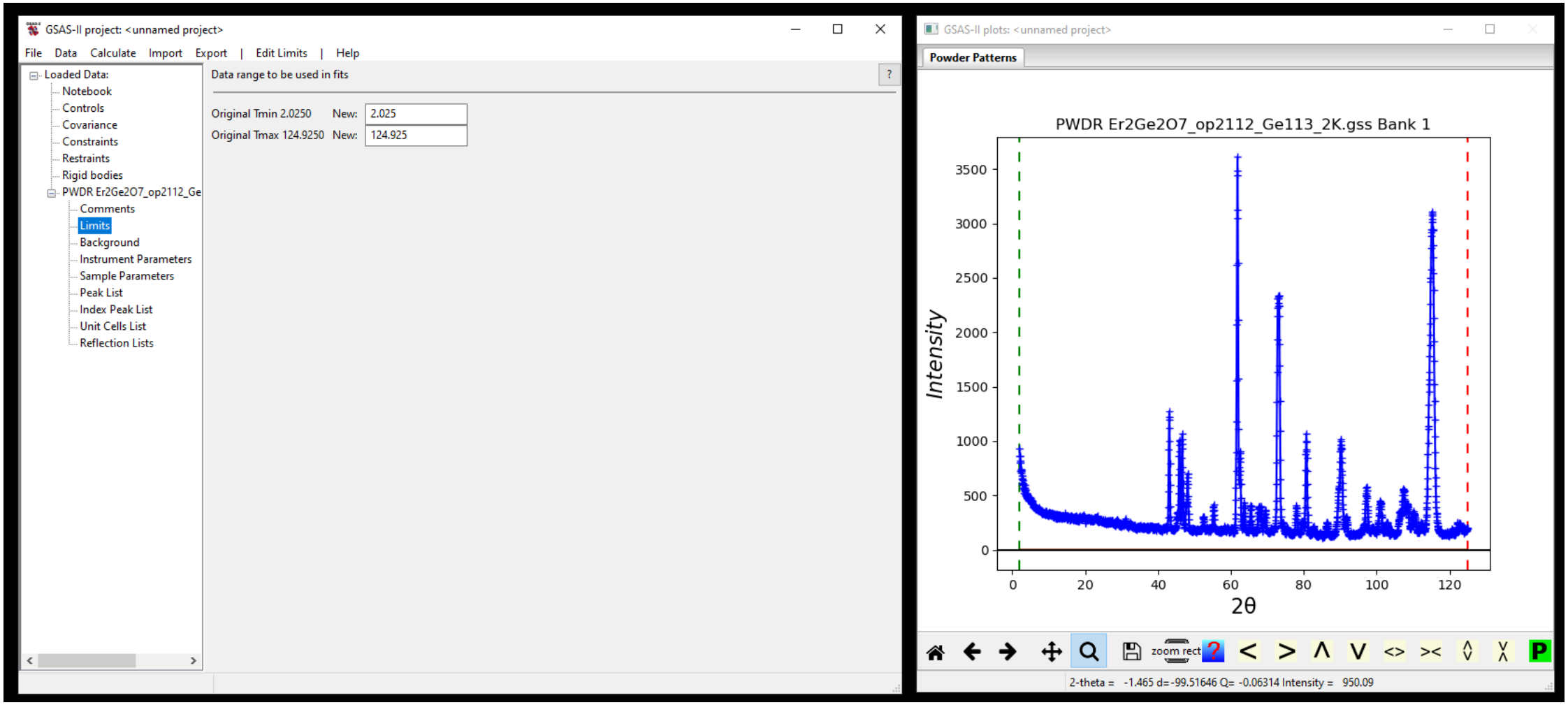
We'll do so using limits

Excluding regions: upper limit



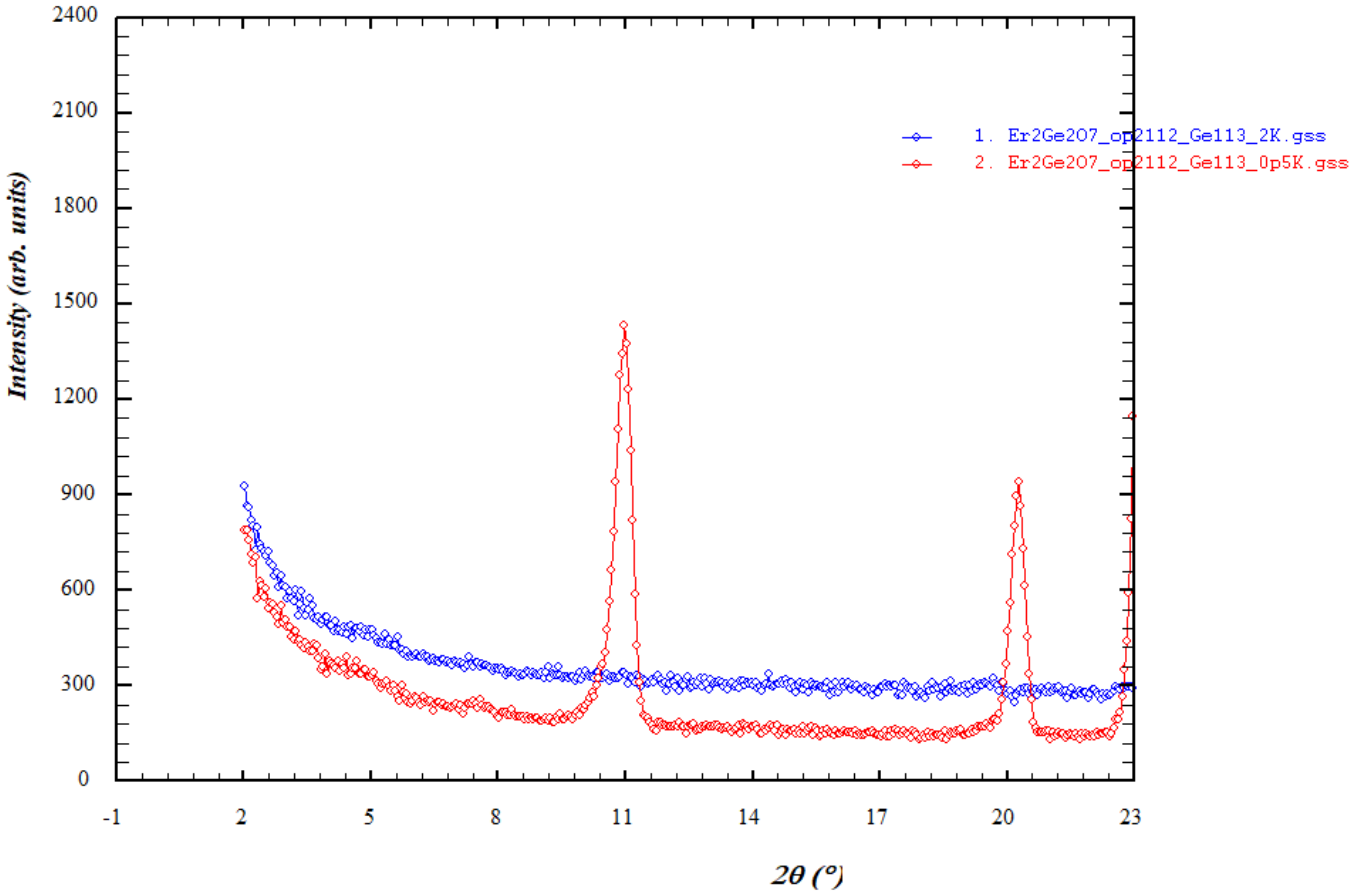
Using the magnifying glass in the plotting window we can zoom in, it has a sharp upturn that is not from our sample but from the direct beam

Excluding regions: upper limit



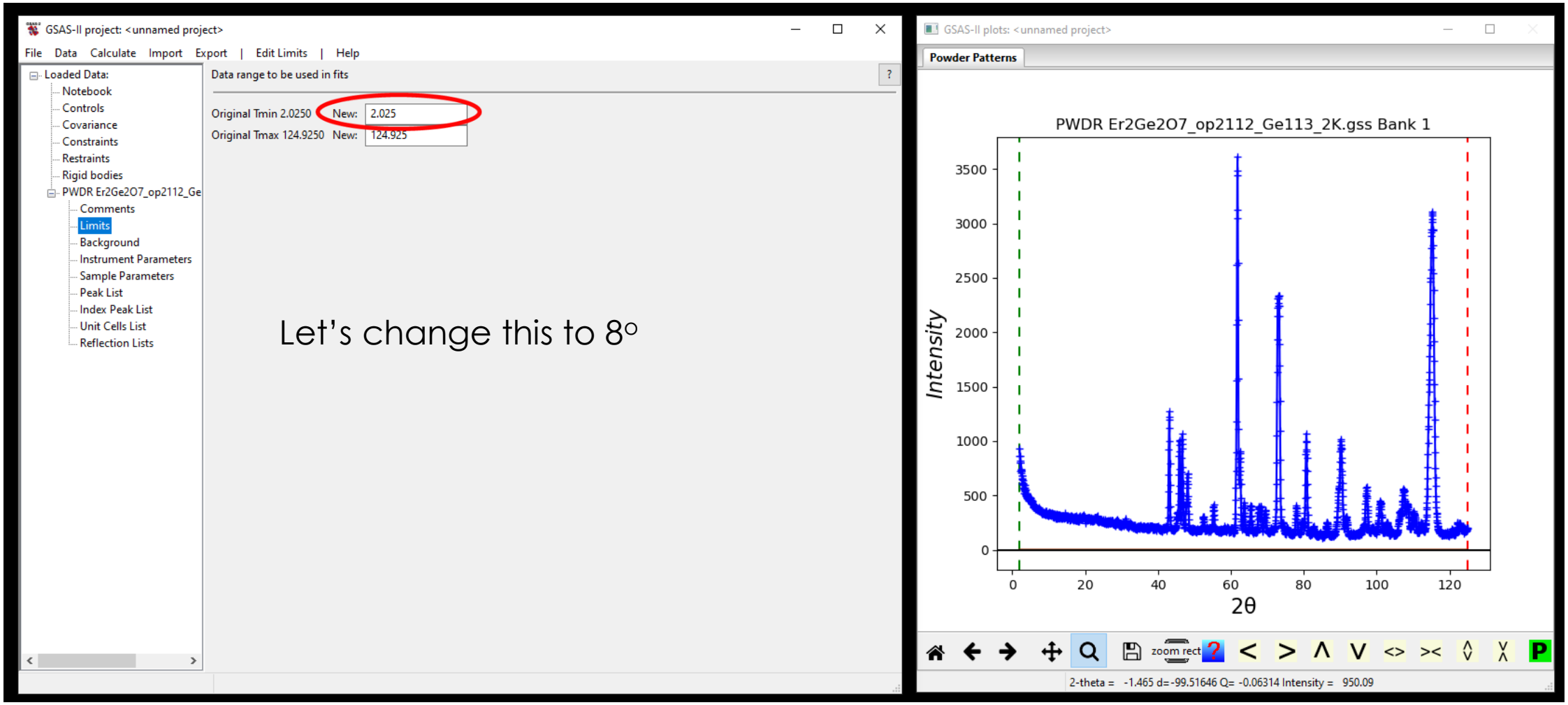
Let's look compare the 20 K and 1.5 K data to see how much we can cut. We don't want to cut so much that we exclude a region which will have a magnetic peak at 1.5 K

Excluding regions: upper limit

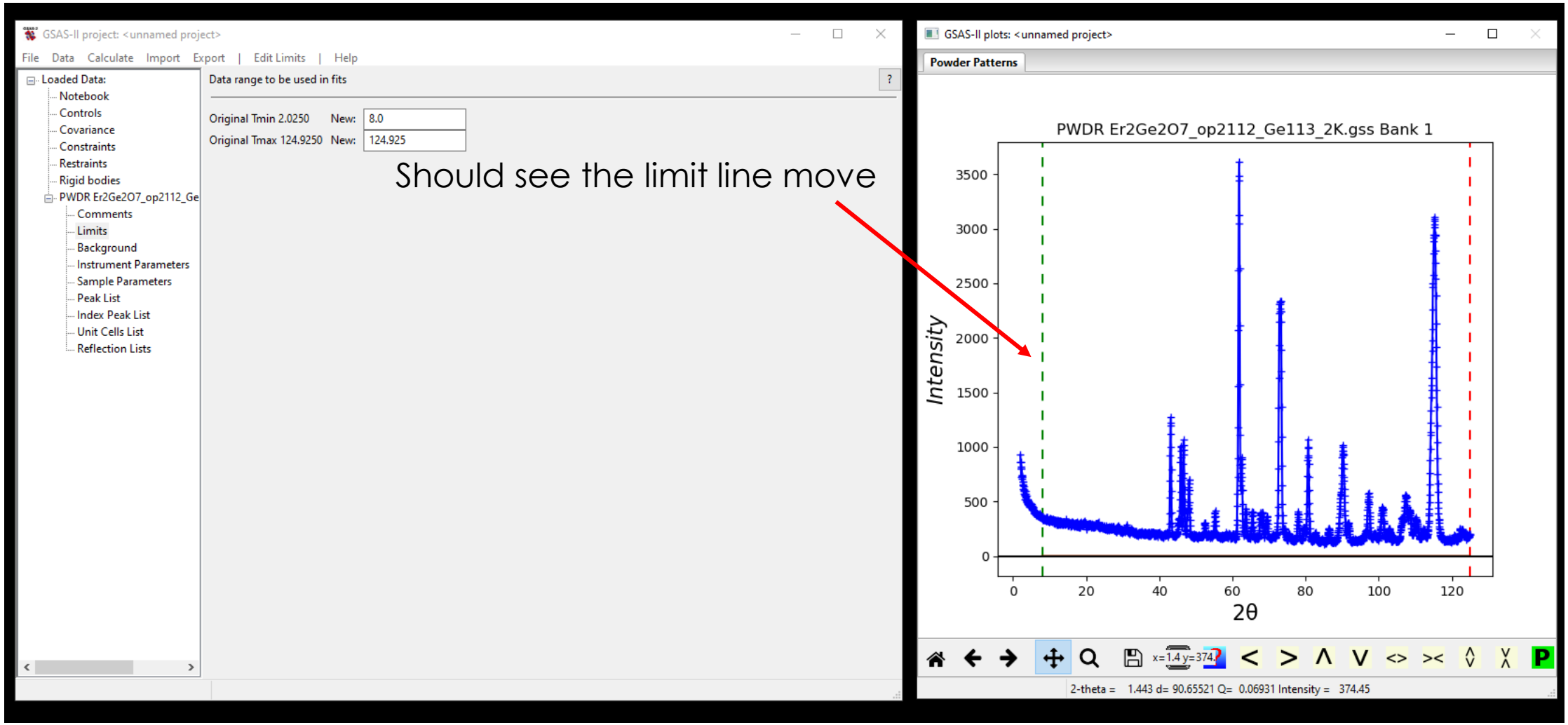


Looks like our first magnetic peak is at ~11° 2θ. Let's cut everything below ~8° 2θ then.

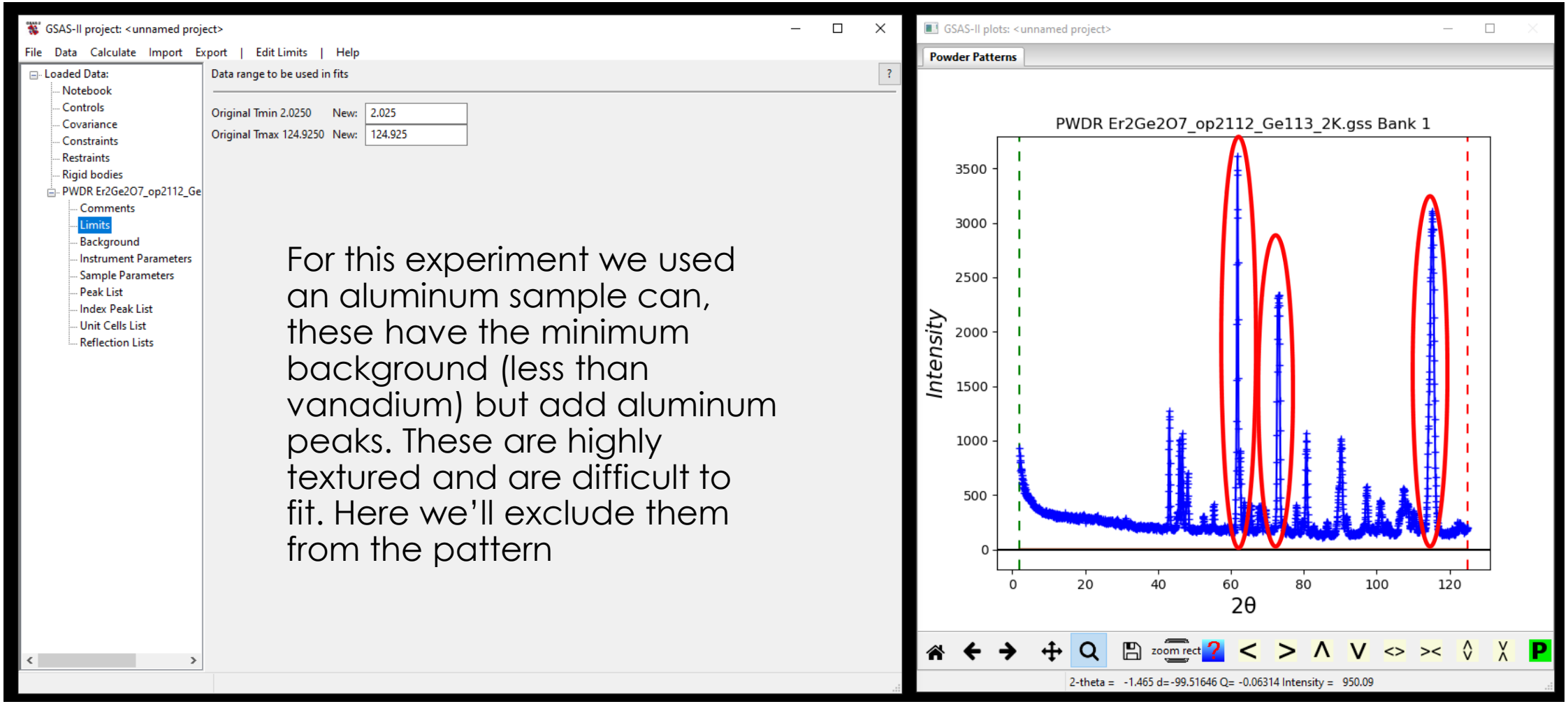
Excluding regions: lower limit



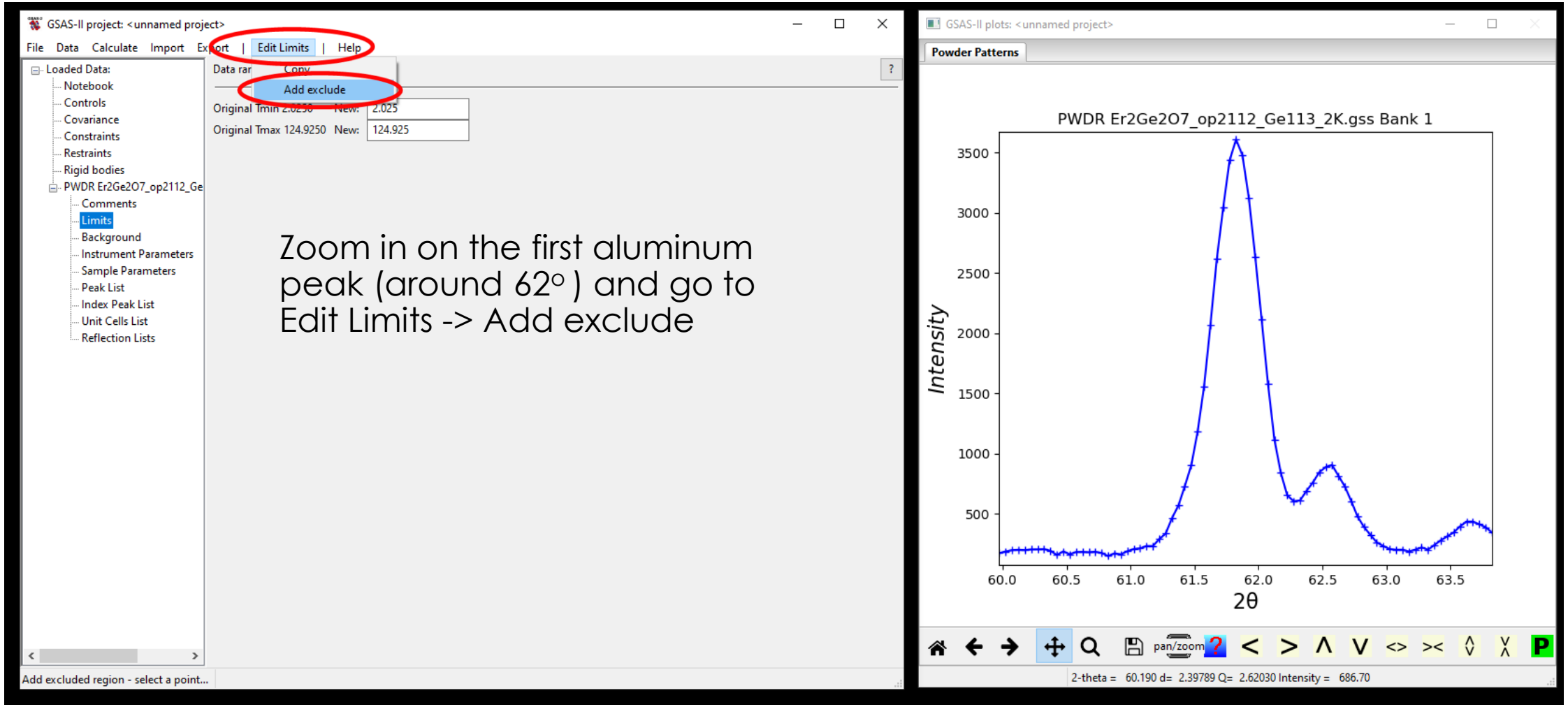
Excluding regions: lower limit



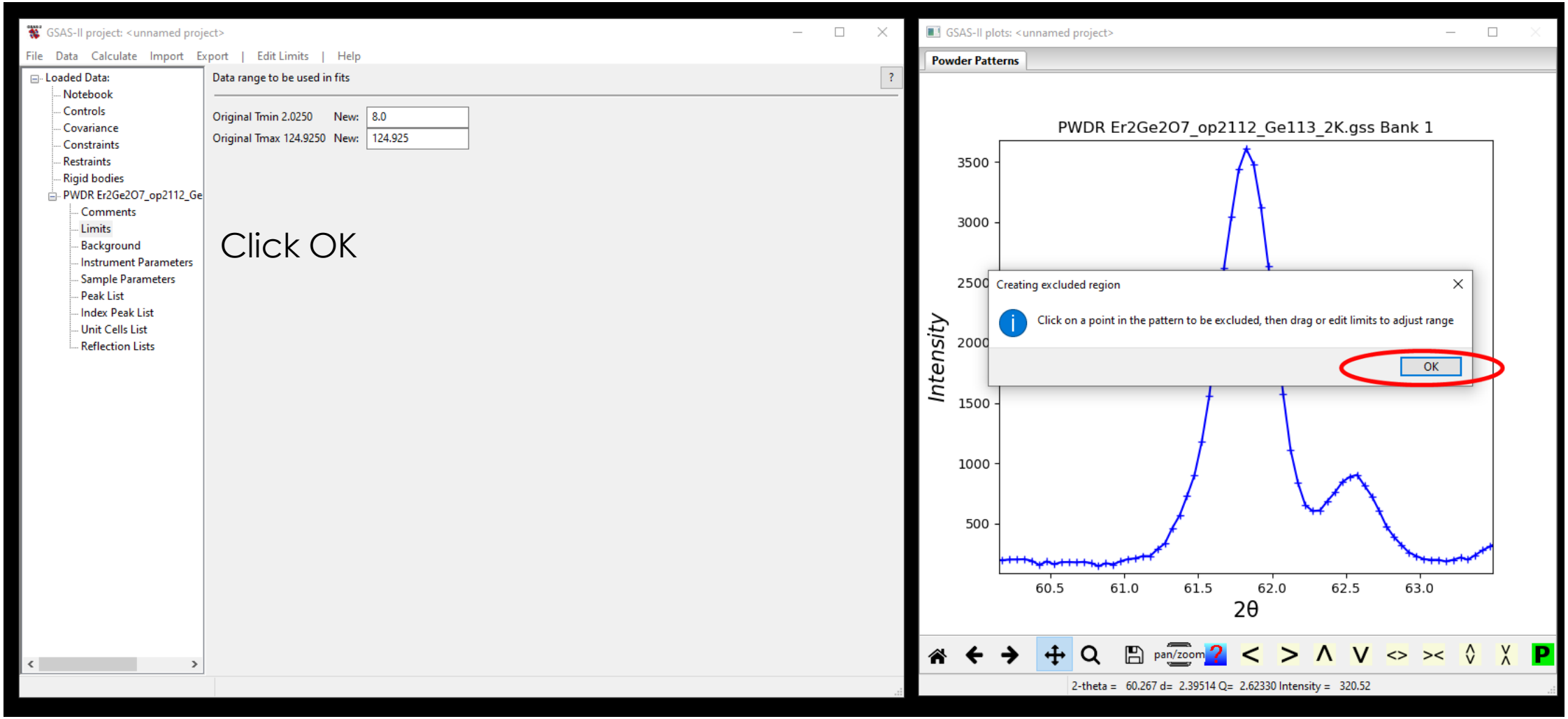
Excluding regions: Aluminum (can) peaks



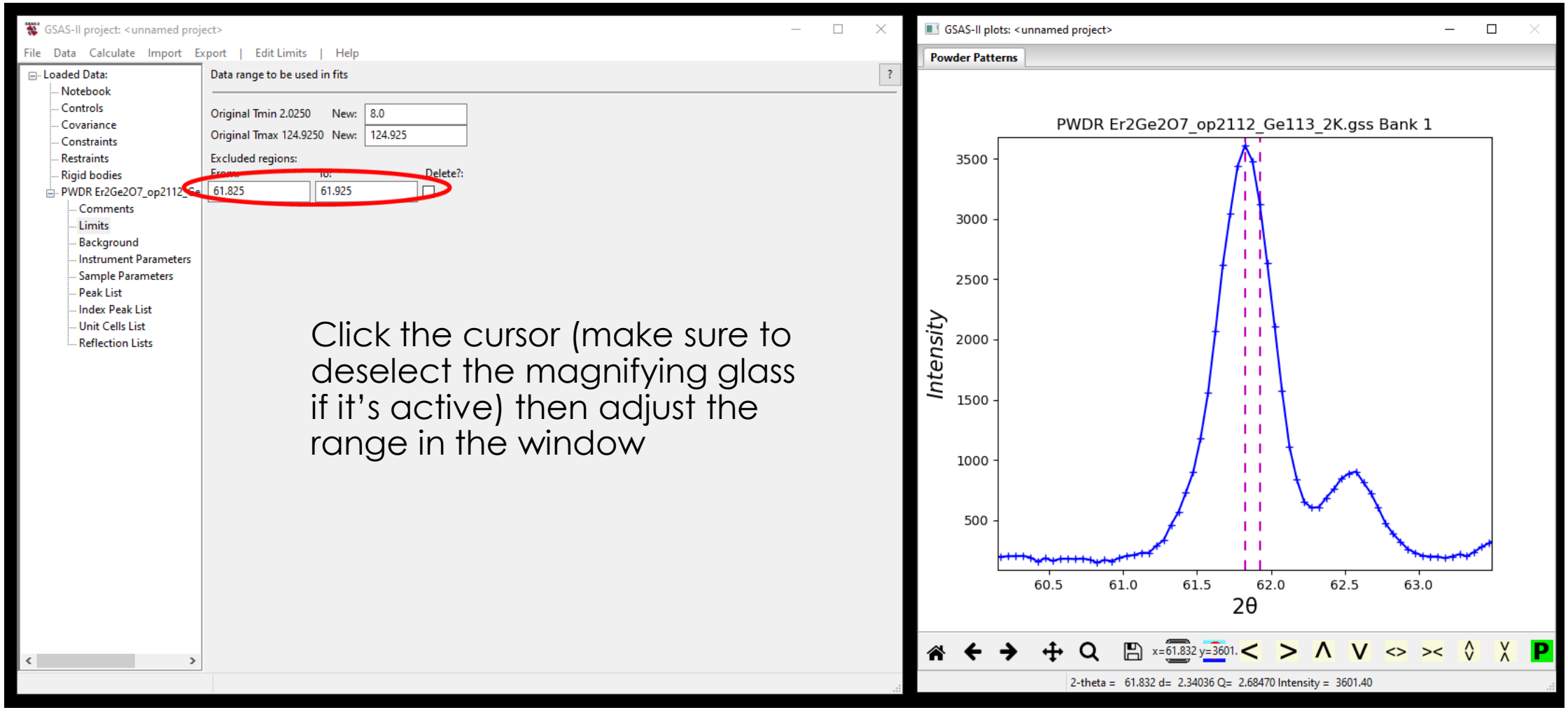
Excluding regions: Aluminum (can) peaks



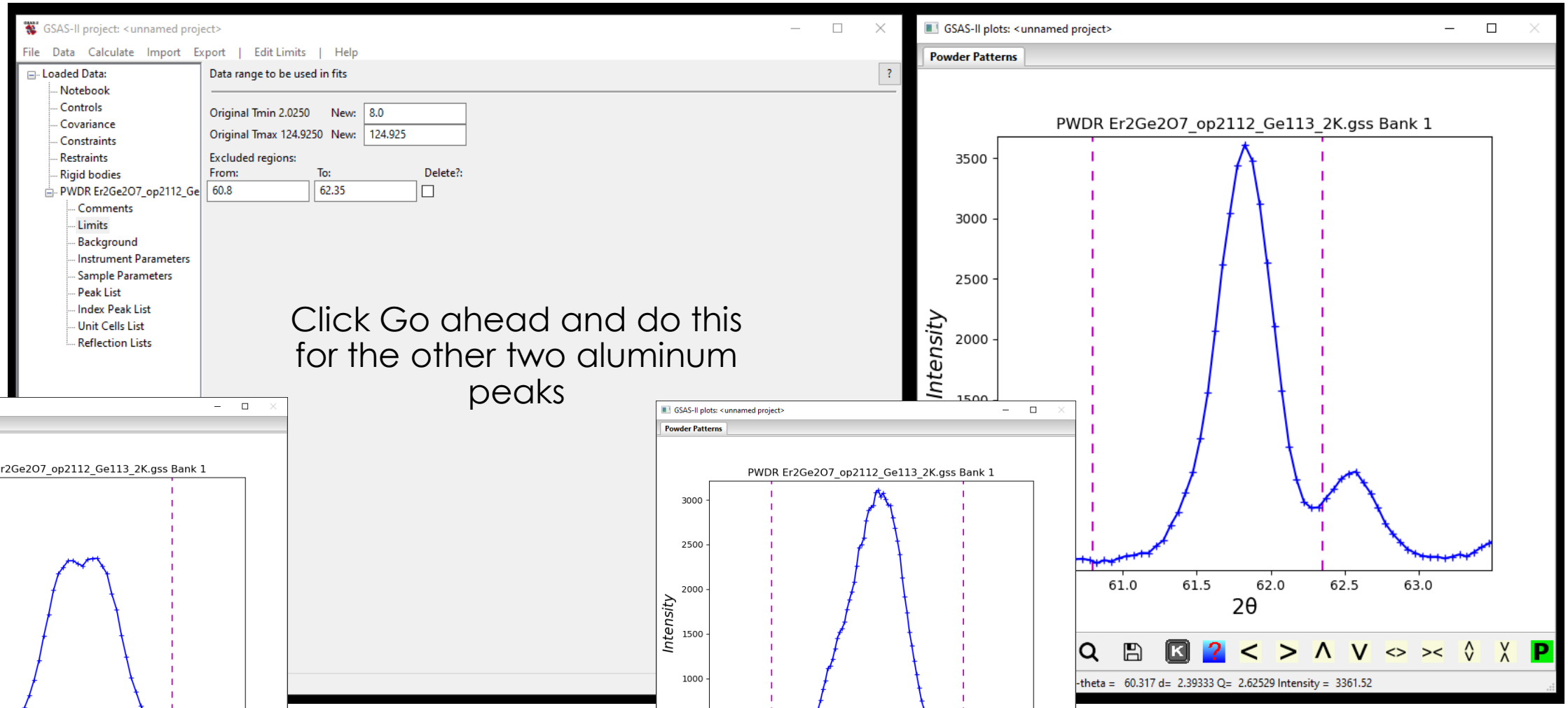
Excluding regions: Aluminum (can) peaks



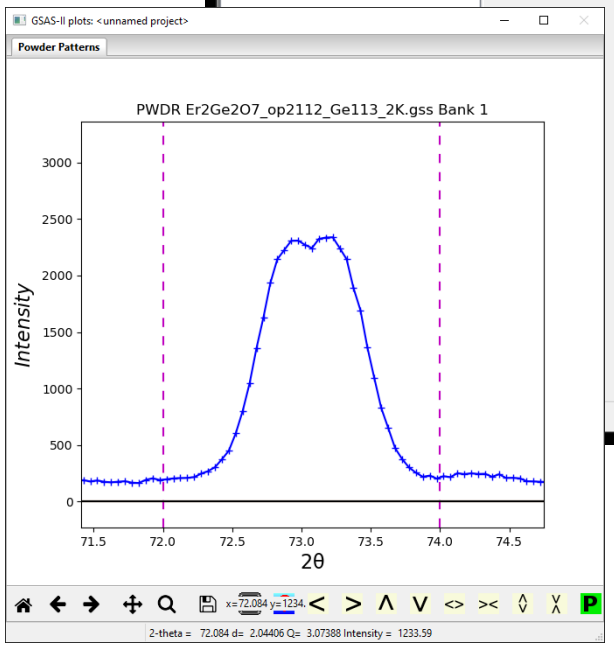
Excluding regions: Aluminum (can) peaks



Excluding regions: Aluminum (can) peaks

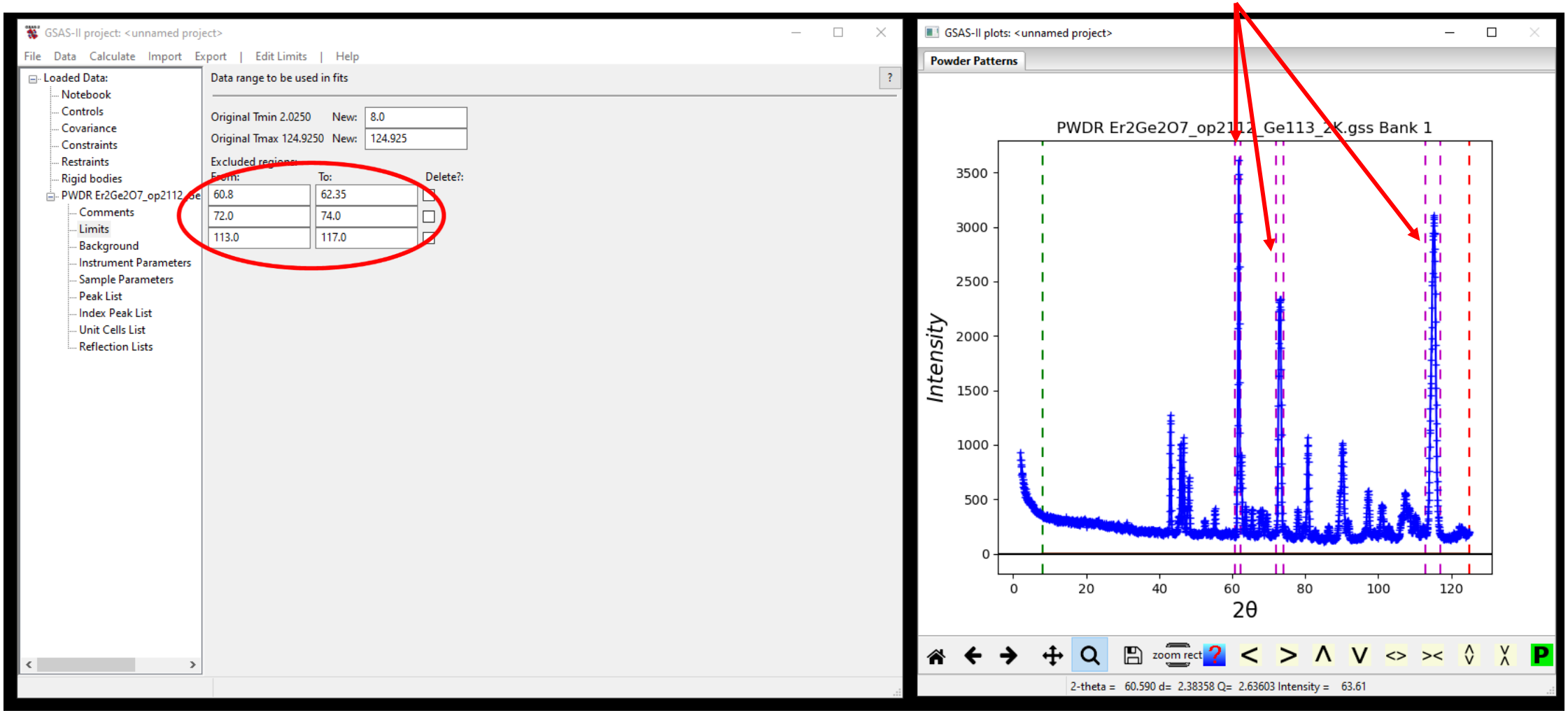


Click Go ahead and do this for the other two aluminum peaks

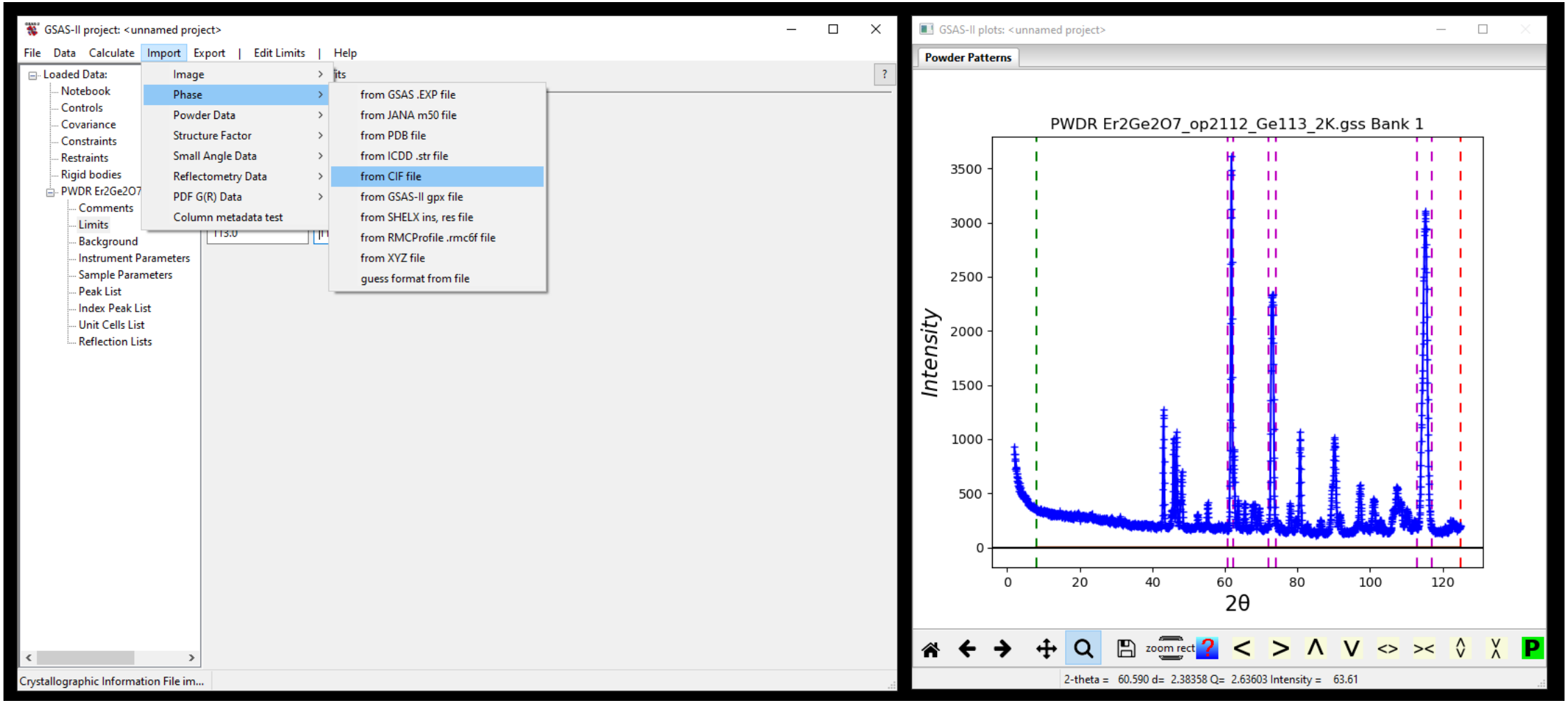


Excluding regions

Now these peaks should have exclusion lines around them

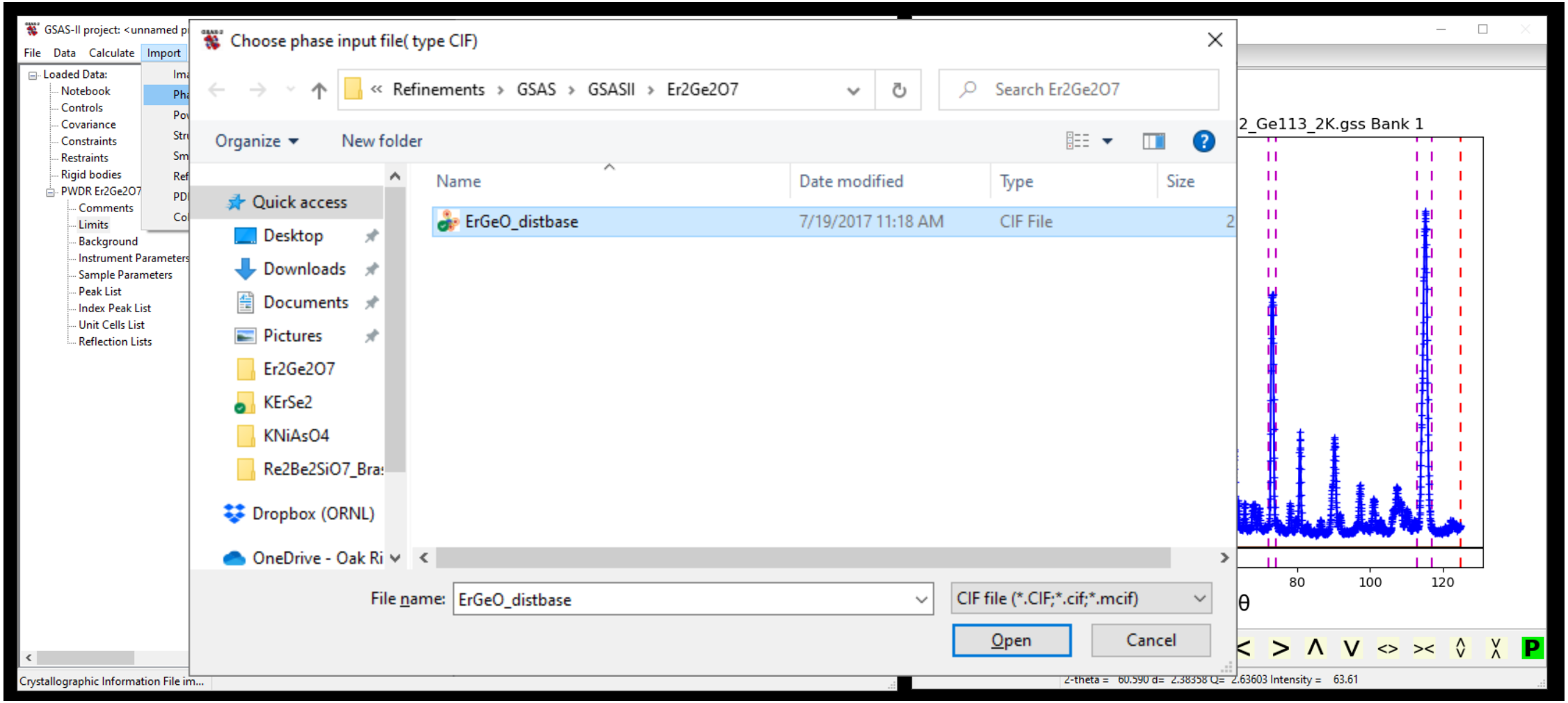


Now we load our phase information (the cif file)



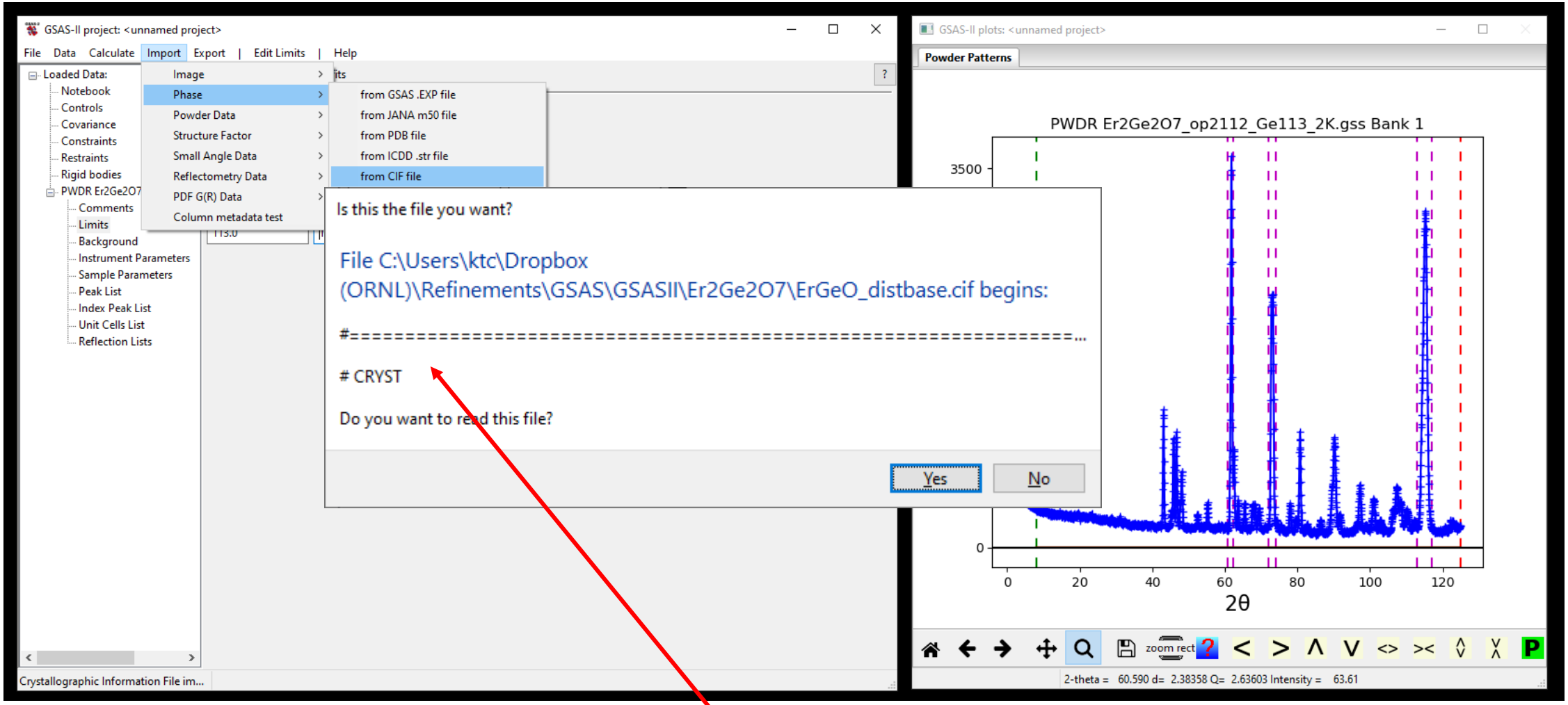
Go to Import –> Phase and click on ‘from CIF file’

Now we load our phase information (the cif file)



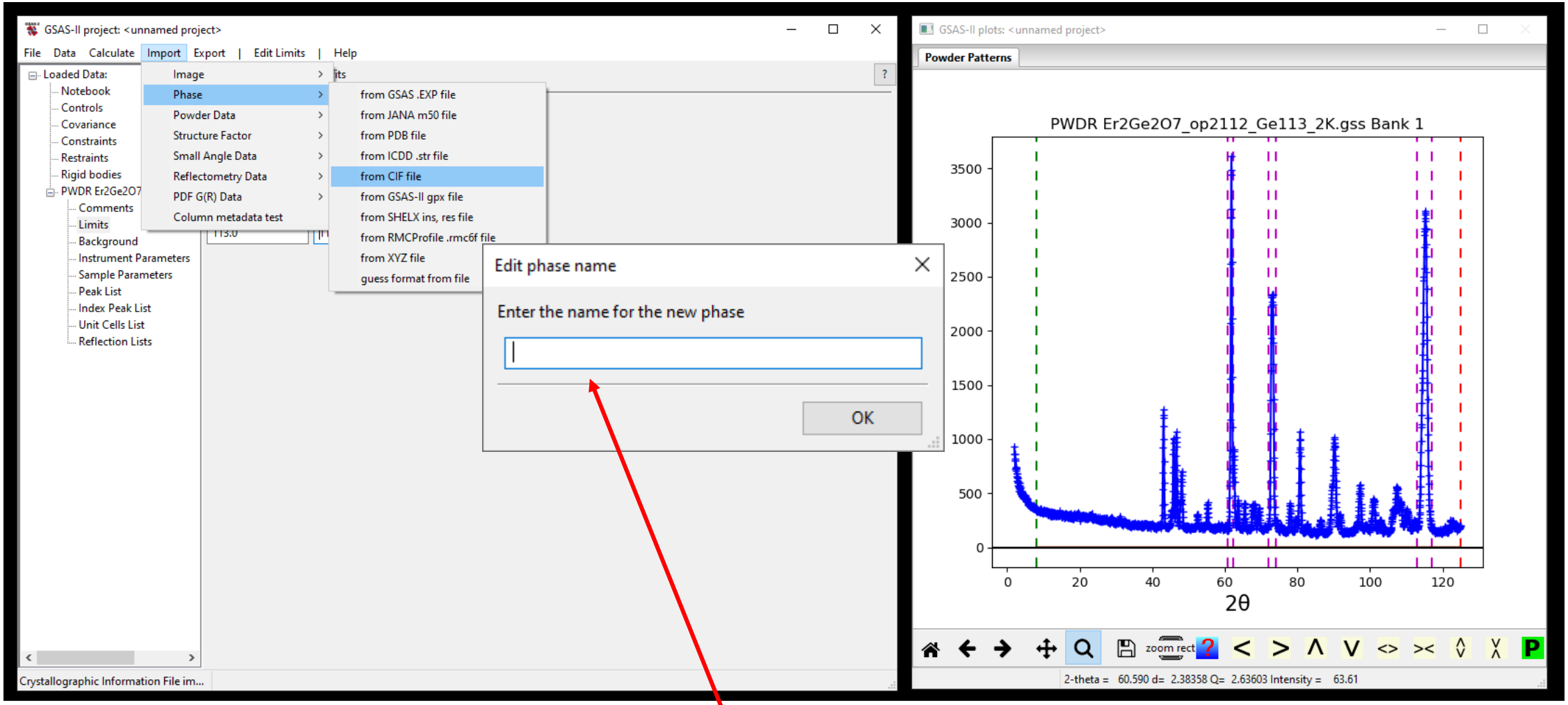
Choose ‘ErGeO_distbase’ (should be the only cif in the folder)

Now we load our phase information (the cif file)



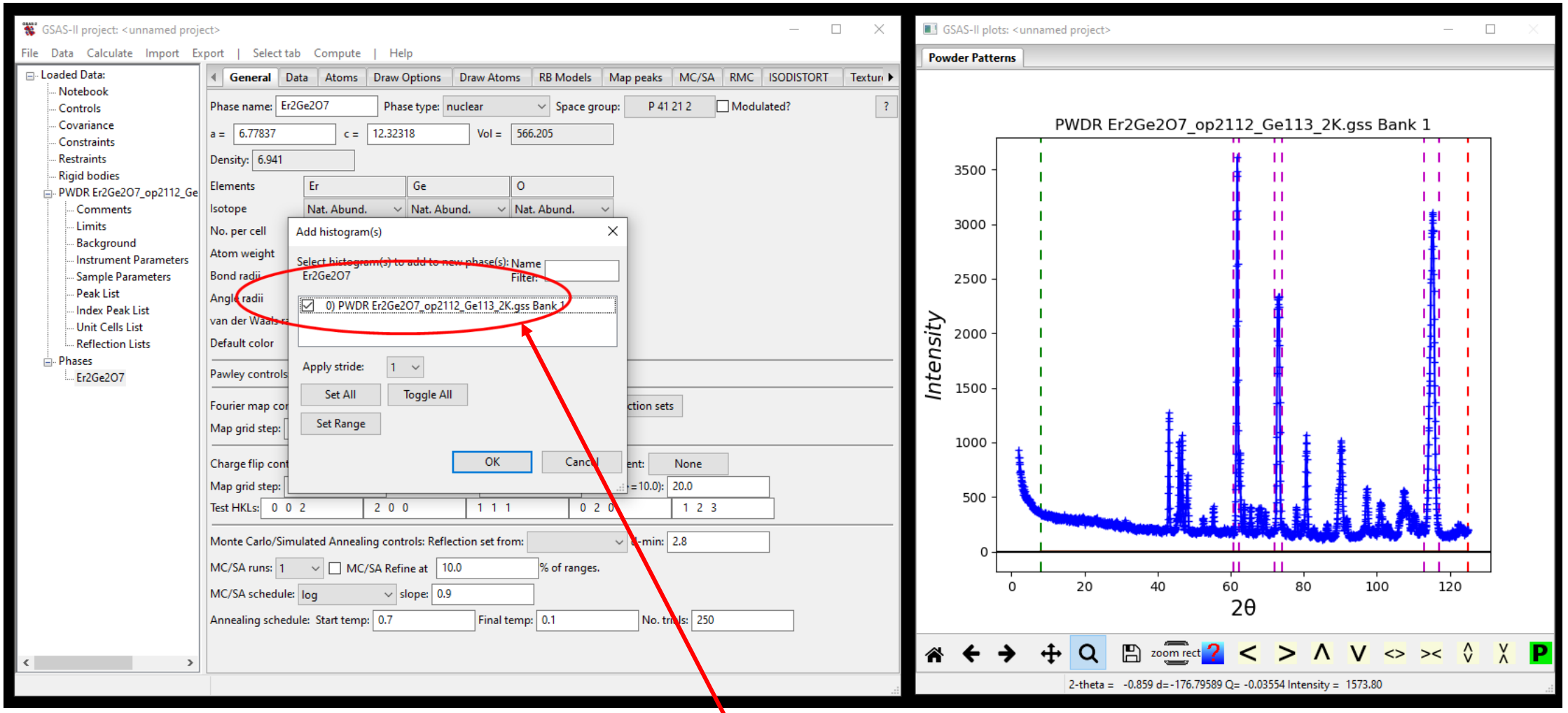
Here it's just pulling the first lines – which is just not too helpful in this case. Click Yes.

Now we load our phase information (the cif file)



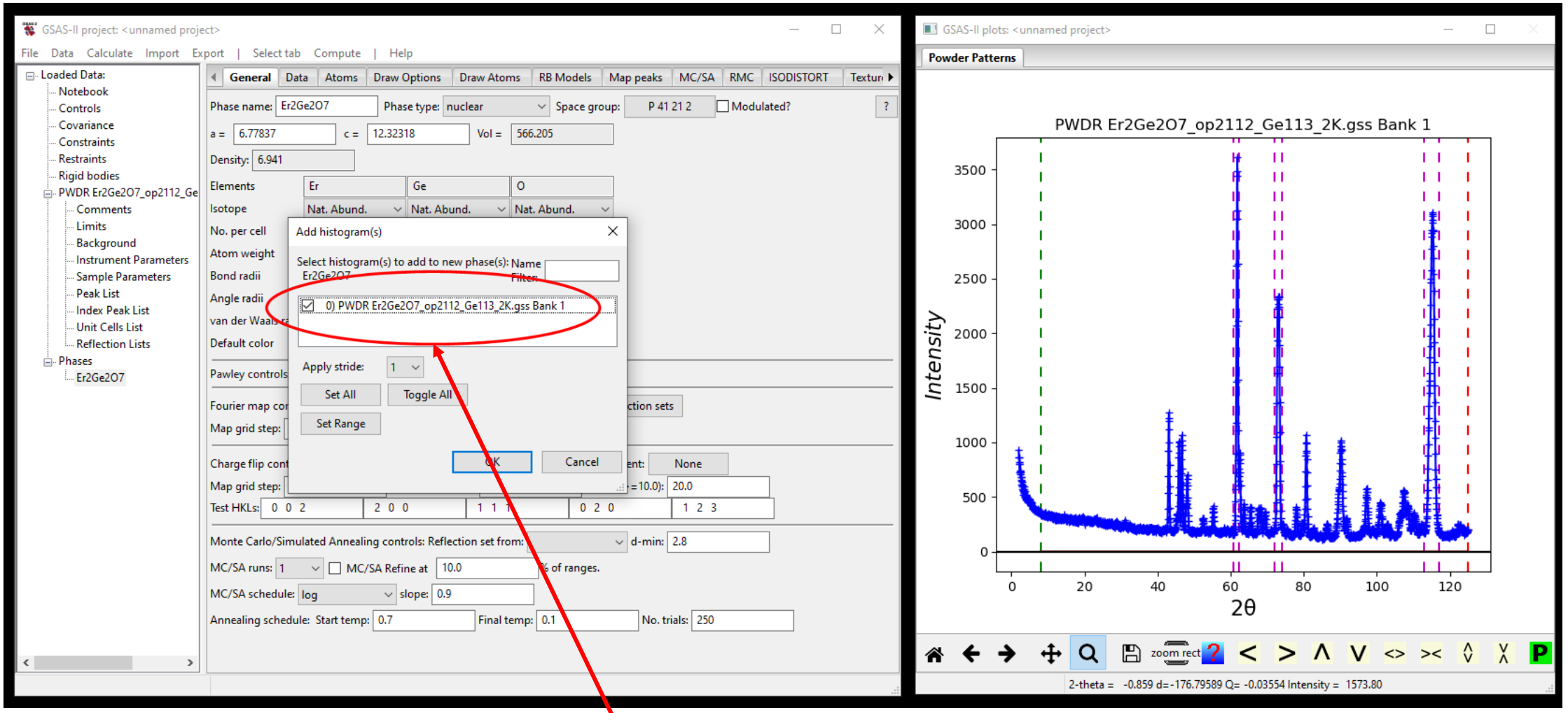
Give it some name which is convenient for you. I'll use 'Er2Ge2O7'

Now we load our phase information (the cif file)



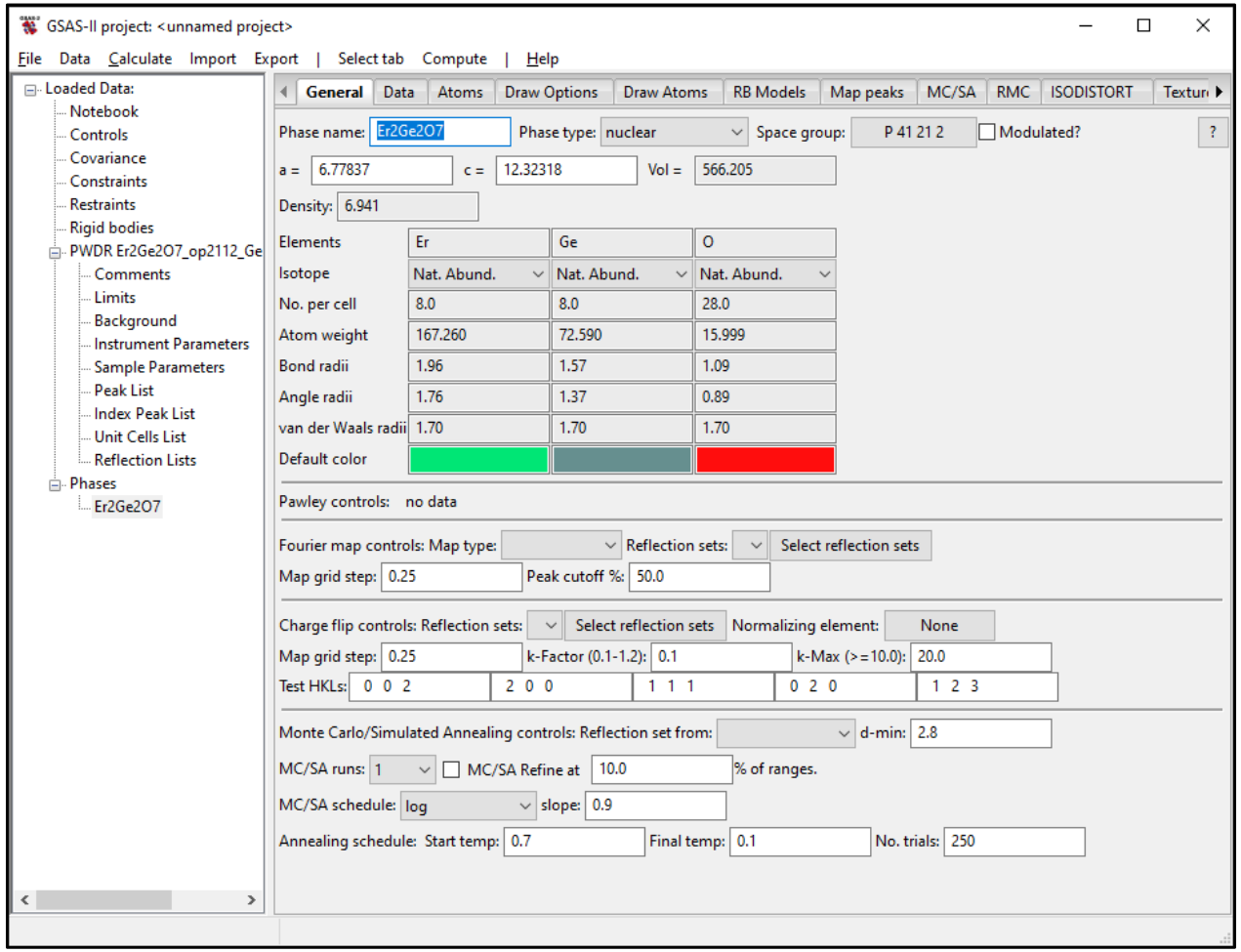
You need to assign the phase to a histogram. For our use this is just an extra button click but could matter if you were using data sets from different measurements. For instance, you wouldn't need to add the magnetic phase to an x-ray histogram in a joint NPD XRD refinement

Now we load our phase information (the cif file)



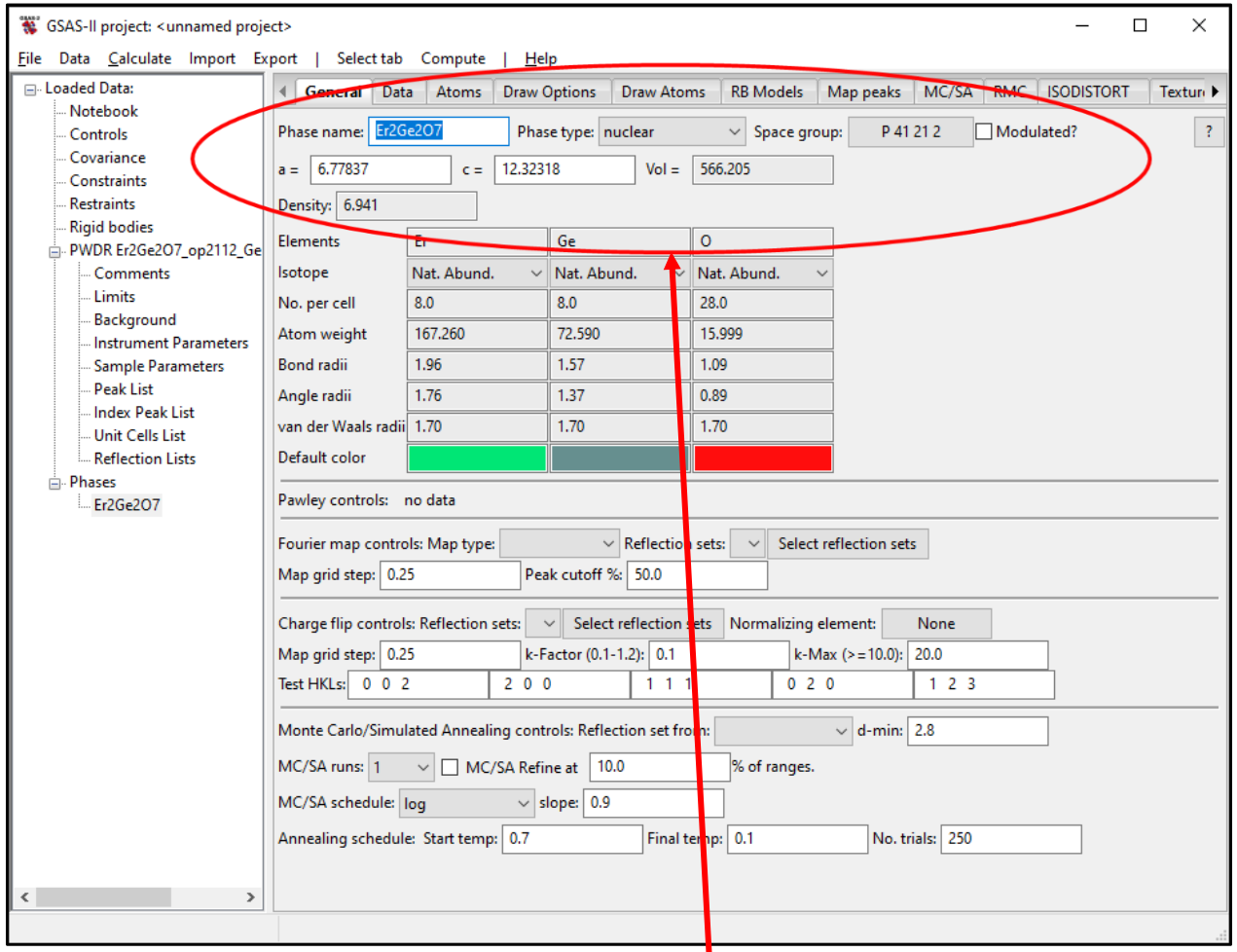
Check the histogram and click 'OK'

Now we're getting somewhere…



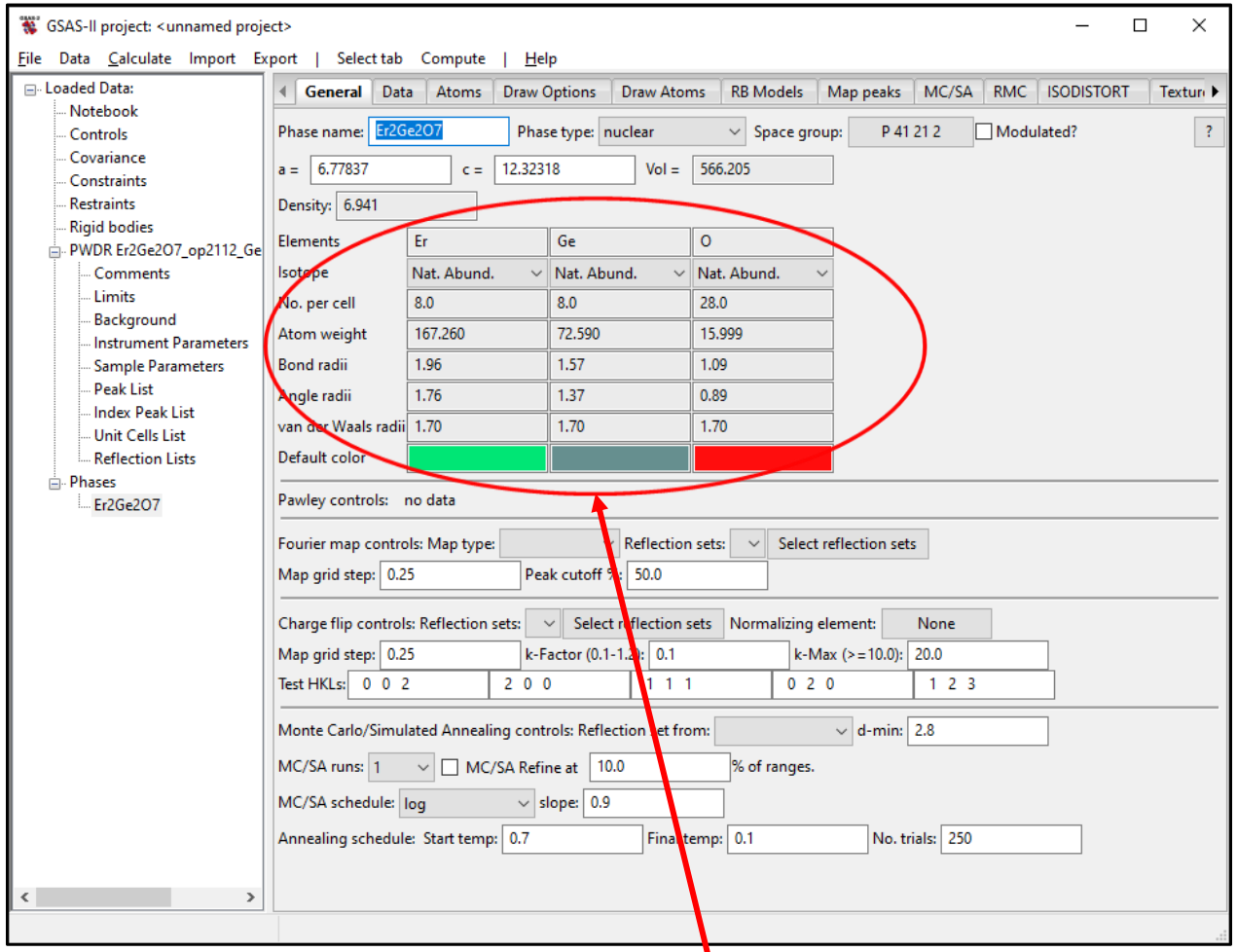
Now there will be a new set of tabs with the phase information and options

Now we're getting somewhere…



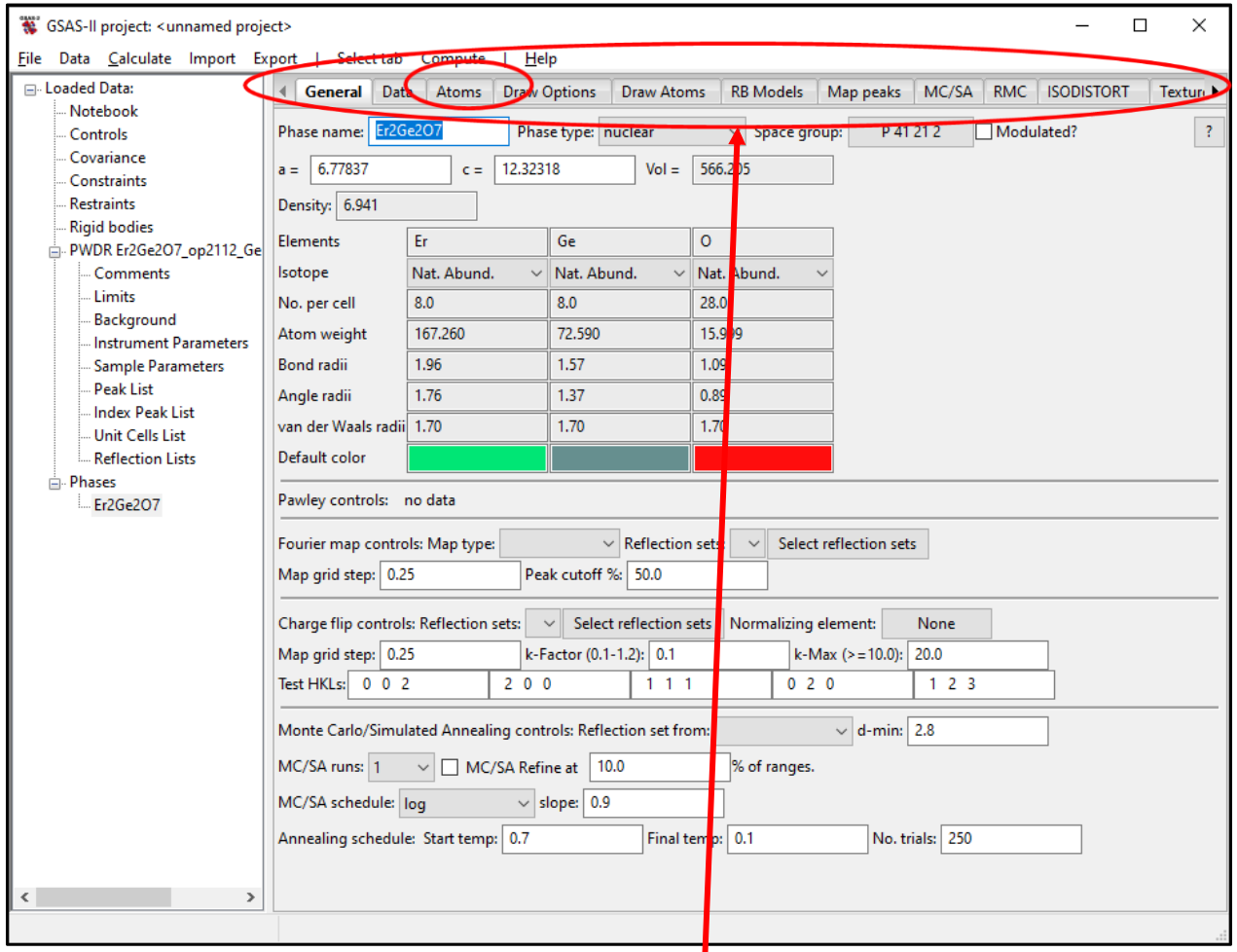
Unit cell information
NB: always check to make sure it read the cif correctly

Now we're getting somewhere…



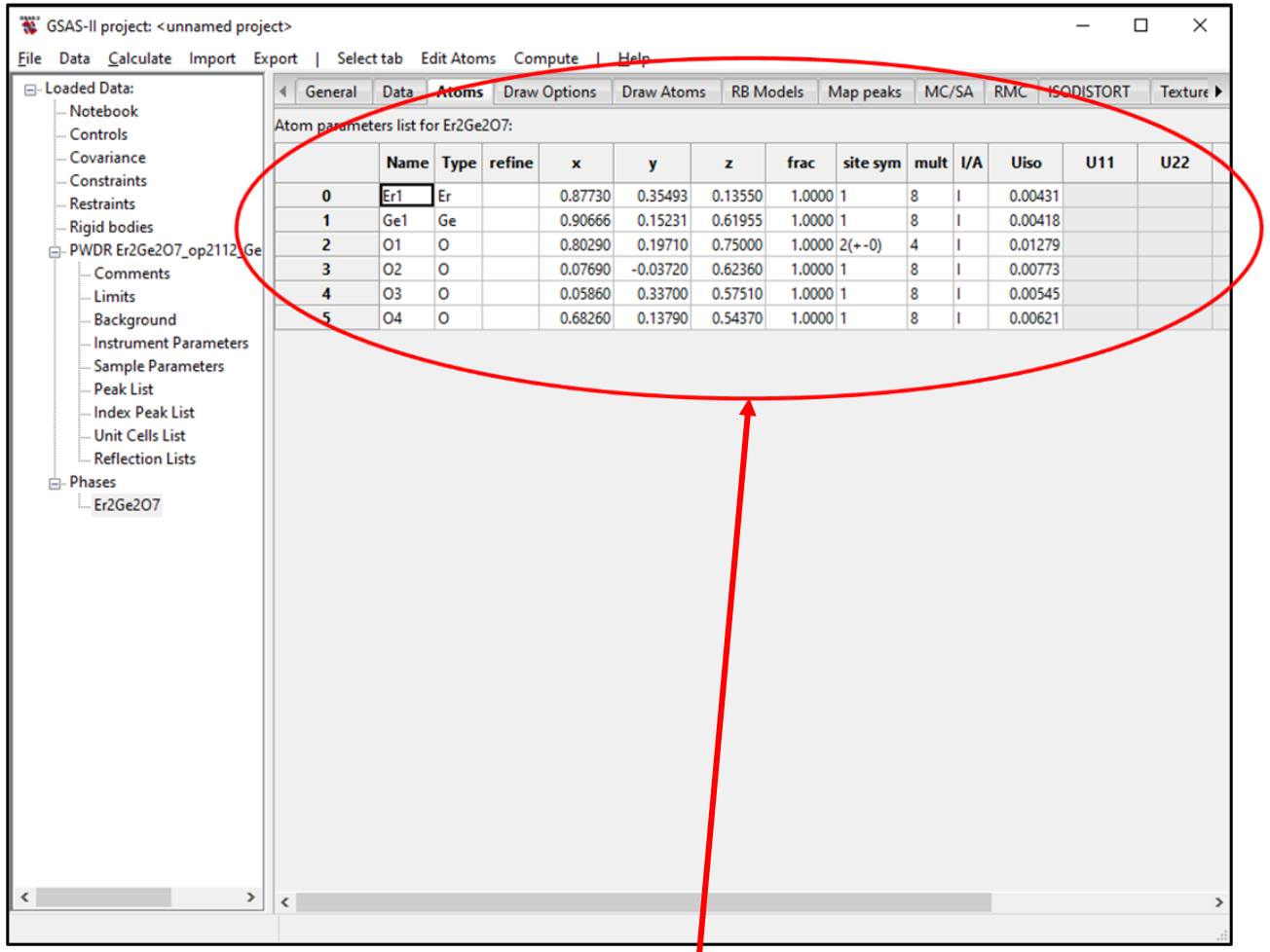
Information on the elements – isotope, stoichiometry, color, etc.

Now we're getting somewhere…



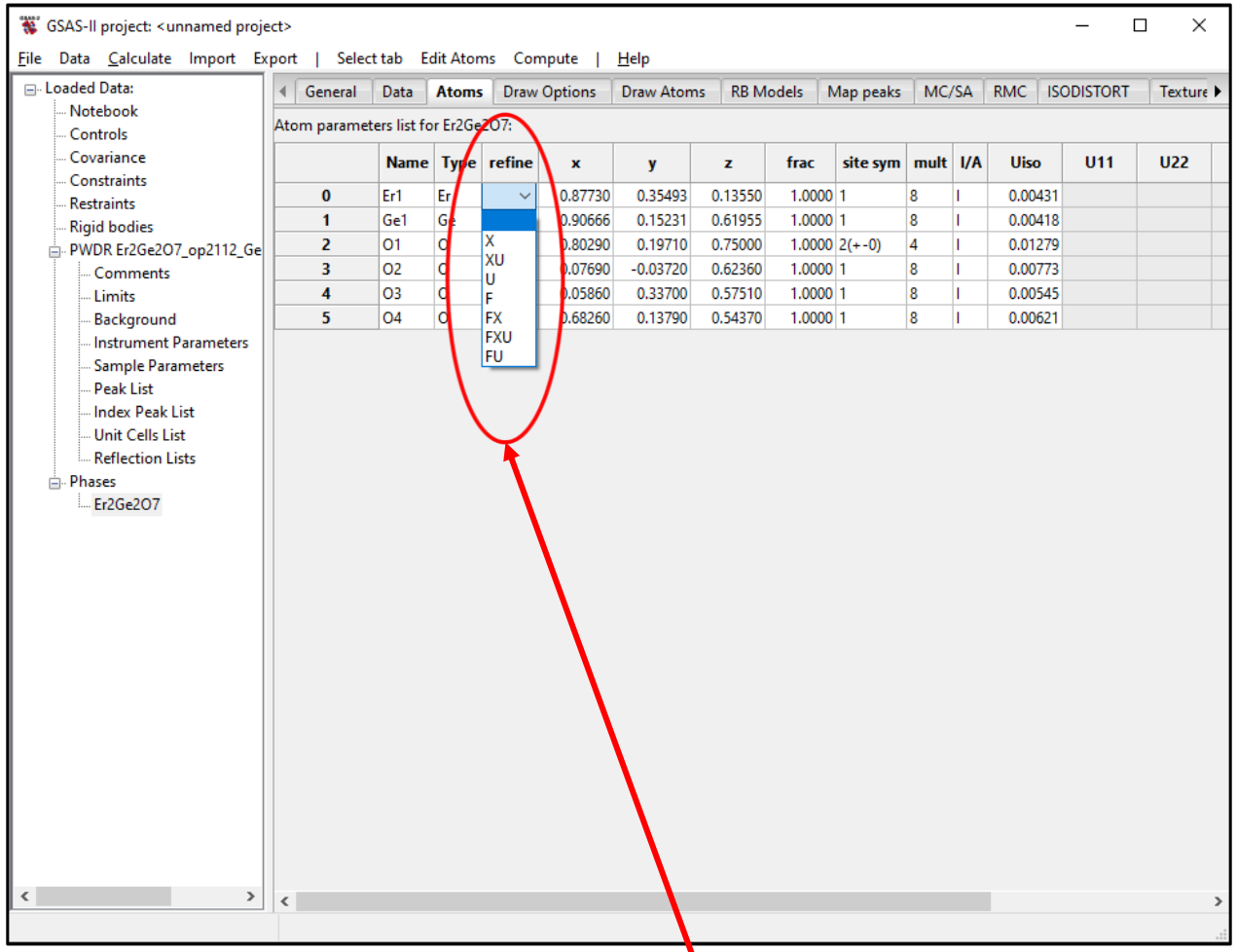
There are also tabs which contain more detailed information about the phase up here for instance, if we click on the atoms tab:

Using the tabs



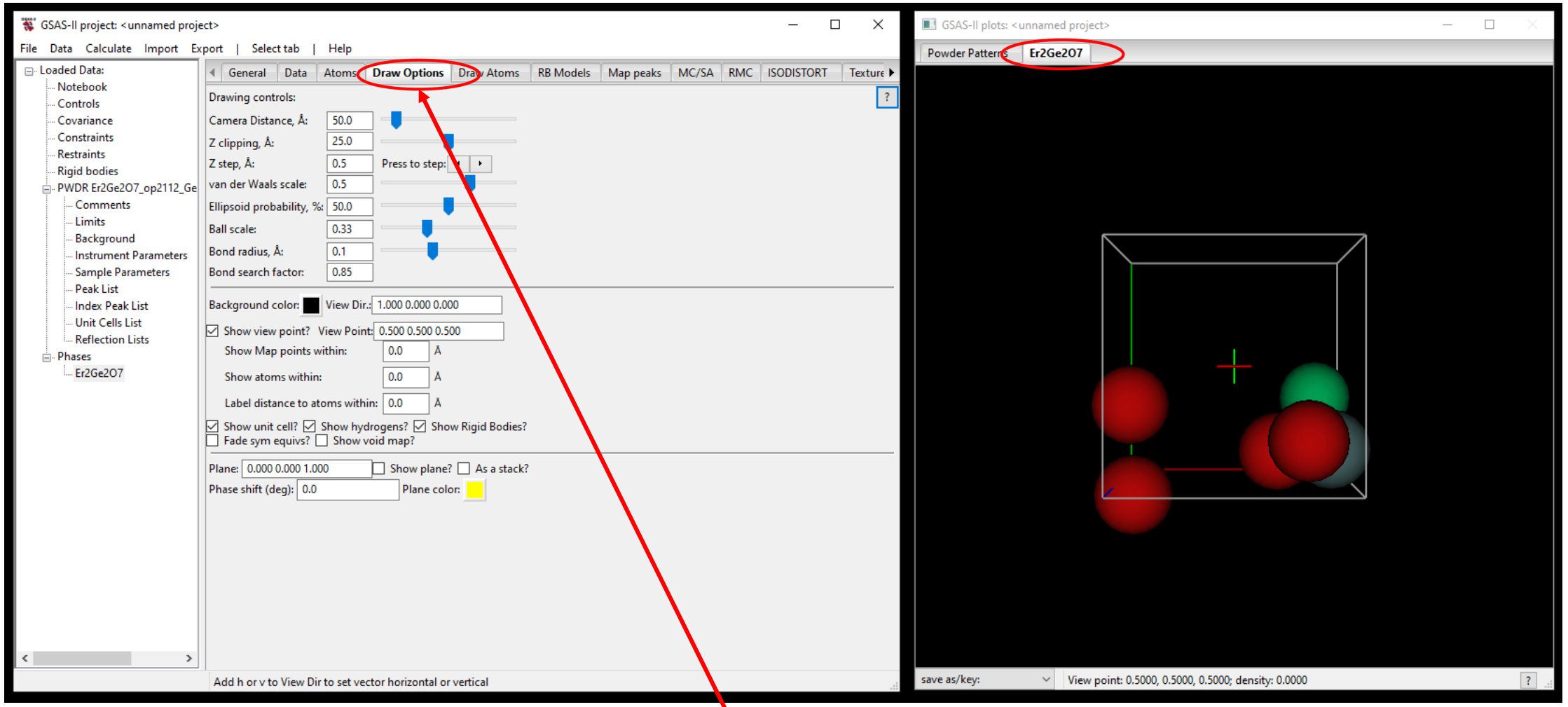
We get a table of the atoms in the phase. Here you can see the atomic positions the site symmetry, multiplicity, displacement parameter(s) and a column with the refinement flag.

Using the tabs



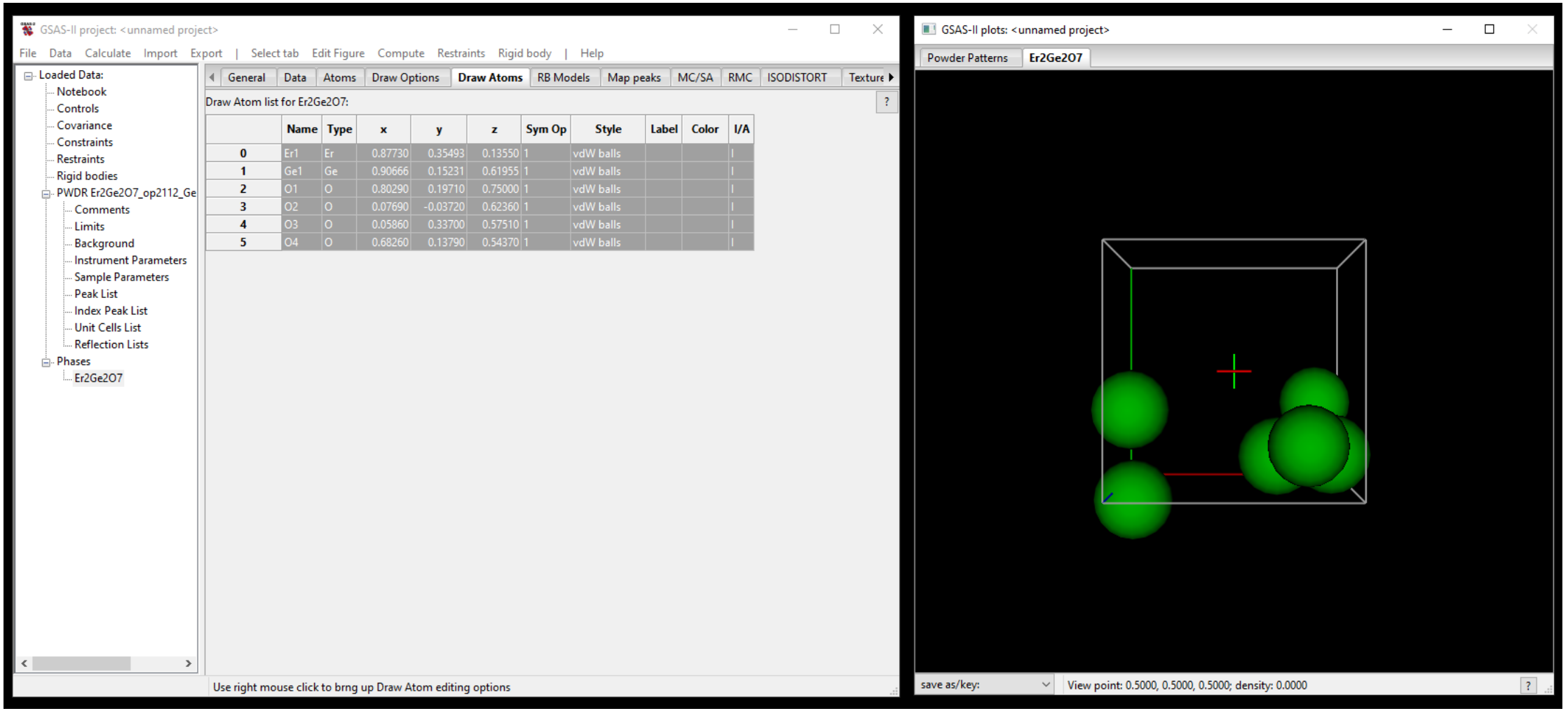
If we click on the refinement box, we get a pull-down list with a selection of refinement flags.

Using the tabs



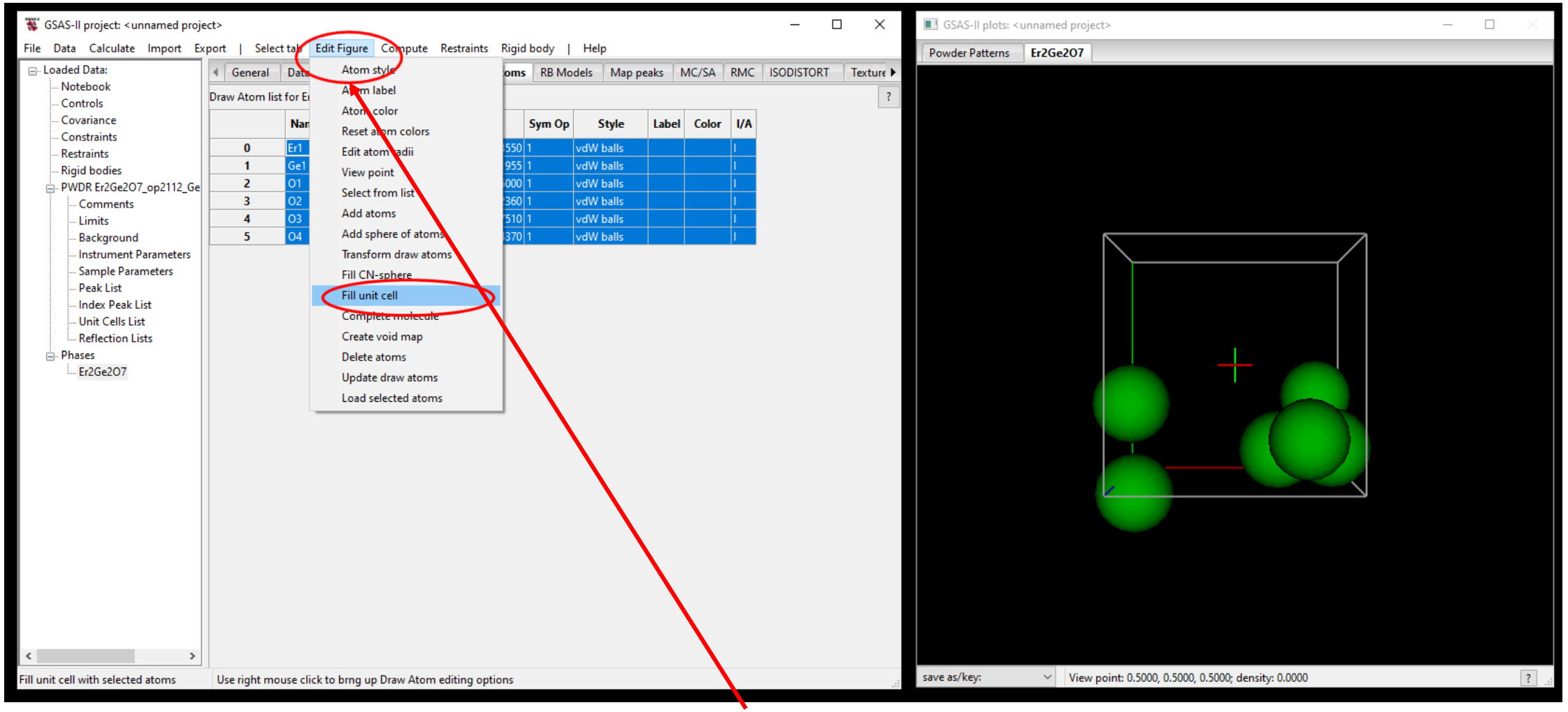
If we click on the 'Draw Atoms' tab, we get a new tab in the plotting window with an (albeit strange) image of the crystal structure

Visualizing the structure



This can be fixed by selecting all the atoms in the draw atoms tab…

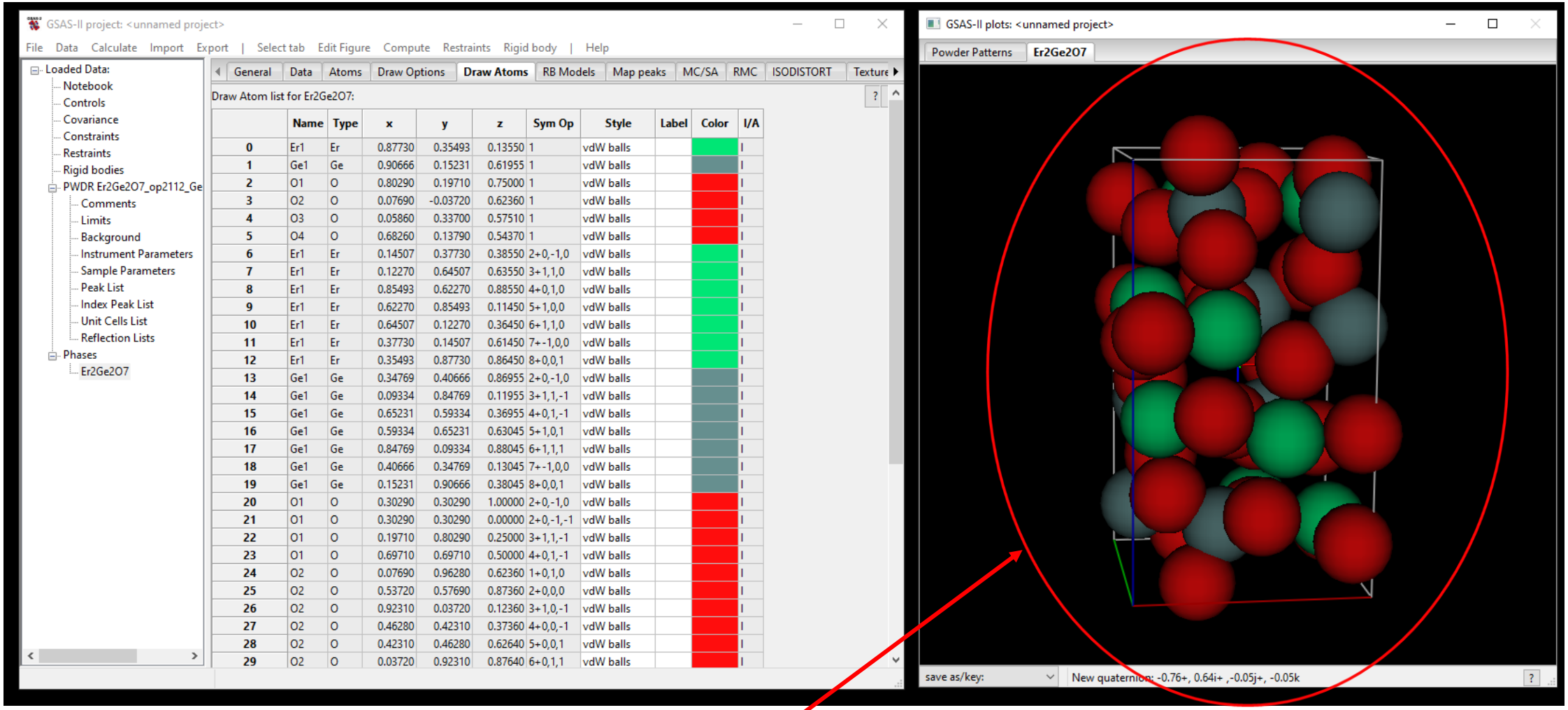
Visualizing the structure



Going to the newly appeared 'Edit Figure' option in the tool bar and selecting 'Fill unit cell'

NB: in GSAS-II, the tool bar is adaptive to the active tab

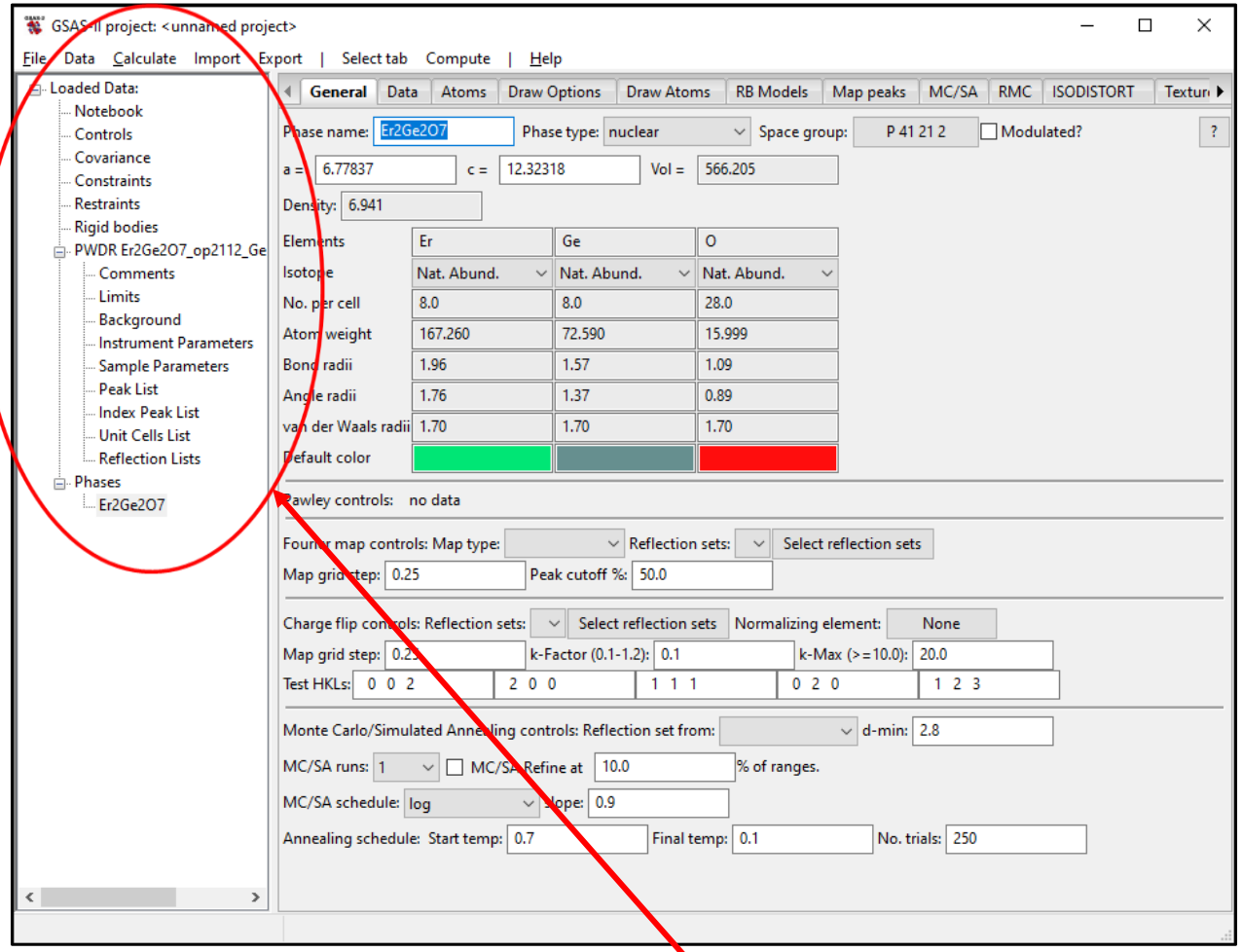
Visualizing the structure



	Name	Type	x	y	z	Sym Op	Style	Label	Color	I/A
0	Er1	Er	0.87730	0.35493	0.13550	1	vdW balls			I
1	Ge1	Ge	0.90666	0.15231	0.61955	1	vdW balls			I
2	O1	O	0.80290	0.19710	0.75000	1	vdW balls			I
3	O2	O	0.07690	-0.03720	0.62360	1	vdW balls			I
4	O3	O	0.05860	0.33700	0.57510	1	vdW balls			I
5	O4	O	0.68260	0.13790	0.54370	1	vdW balls			I
6	Er1	Er	0.14507	0.37730	0.38550	2+0,-1,0	vdW balls			I
7	Er1	Er	0.12270	0.64507	0.63550	3+1,1,0	vdW balls			I
8	Er1	Er	0.85493	0.62270	0.88550	4+0,1,0	vdW balls			I
9	Er1	Er	0.62270	0.85493	0.11450	5+1,0,0	vdW balls			I
10	Er1	Er	0.64507	0.12270	0.36450	6+1,1,0	vdW balls			I
11	Er1	Er	0.37730	0.14507	0.61450	7+-1,0,0	vdW balls			I
12	Er1	Er	0.35493	0.87730	0.86450	8+0,0,1	vdW balls			I
13	Ge1	Ge	0.34769	0.40666	0.86955	2+0,-1,0	vdW balls			I
14	Ge1	Ge	0.09334	0.84769	0.11955	3+1,1,-1	vdW balls			I
15	Ge1	Ge	0.65231	0.59334	0.36955	4+0,1,-1	vdW balls			I
16	Ge1	Ge	0.59334	0.65231	0.63045	5+1,0,1	vdW balls			I
17	Ge1	Ge	0.84769	0.09334	0.88045	6+1,1,1	vdW balls			I
18	Ge1	Ge	0.40666	0.34769	0.13045	7+-1,0,0	vdW balls			I
19	Ge1	Ge	0.15231	0.90666	0.38045	8+0,0,1	vdW balls			I
20	O1	O	0.30290	0.30290	1.00000	2+0,-1,0	vdW balls			I
21	O1	O	0.30290	0.30290	0.00000	2+0,-1,-1	vdW balls			I
22	O1	O	0.19710	0.80290	0.25000	3+1,1,-1	vdW balls			I
23	O1	O	0.69710	0.69710	0.50000	4+0,1,-1	vdW balls			I
24	O2	O	0.07690	0.96280	0.62360	1+0,1,0	vdW balls			I
25	O2	O	0.53720	0.57690	0.87360	2+0,0,0	vdW balls			I
26	O2	O	0.92310	0.03720	0.12360	3+1,0,-1	vdW balls			I
27	O2	O	0.46280	0.42310	0.37360	4+0,0,-1	vdW balls			I
28	O2	O	0.42310	0.46280	0.62640	5+0,0,1	vdW balls			I
29	O2	O	0.03720	0.92310	0.87640	6+0,1,1	vdW balls			I

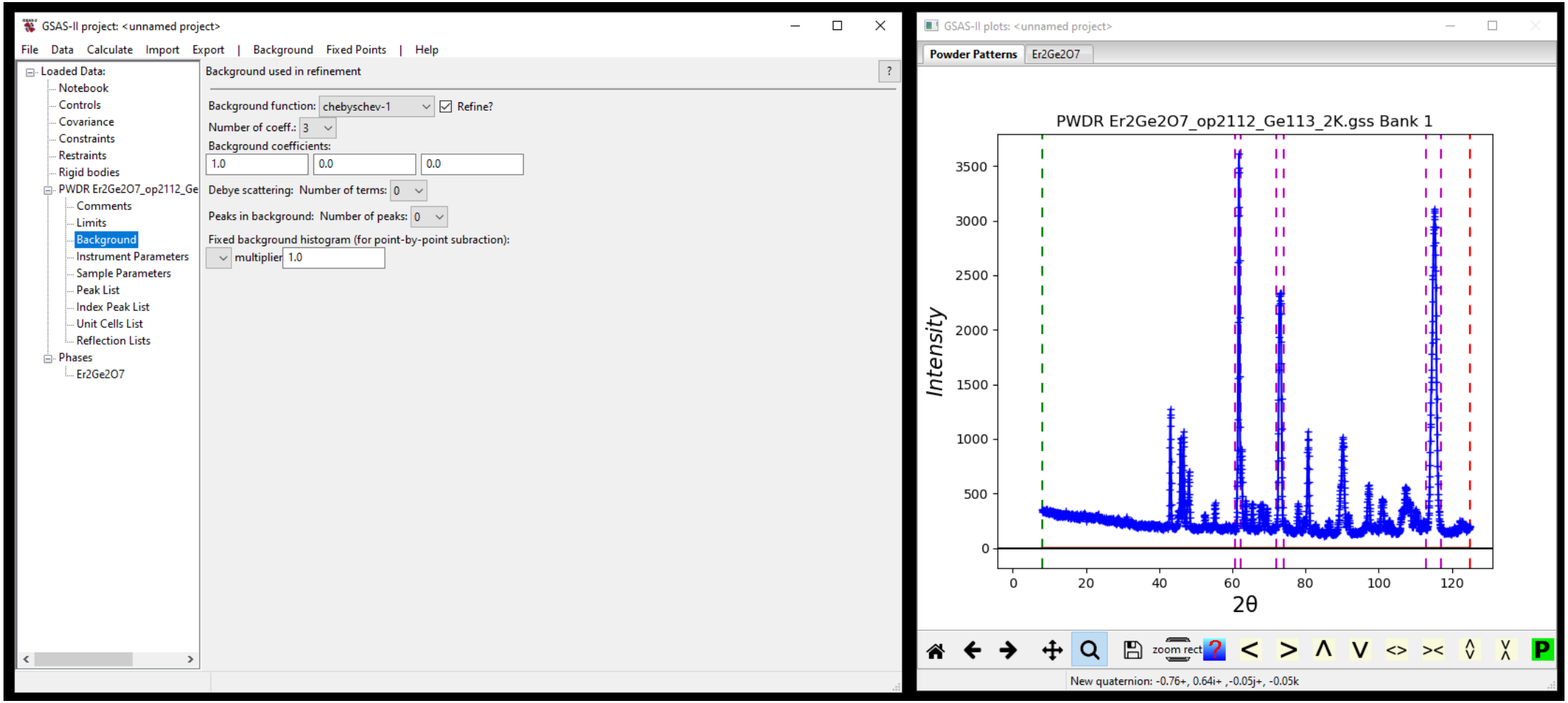
Now all the atoms in the unit cell show
(I've rotated this by clicking and dragging in the window)

Just a general comment on GSAS-II philosophy



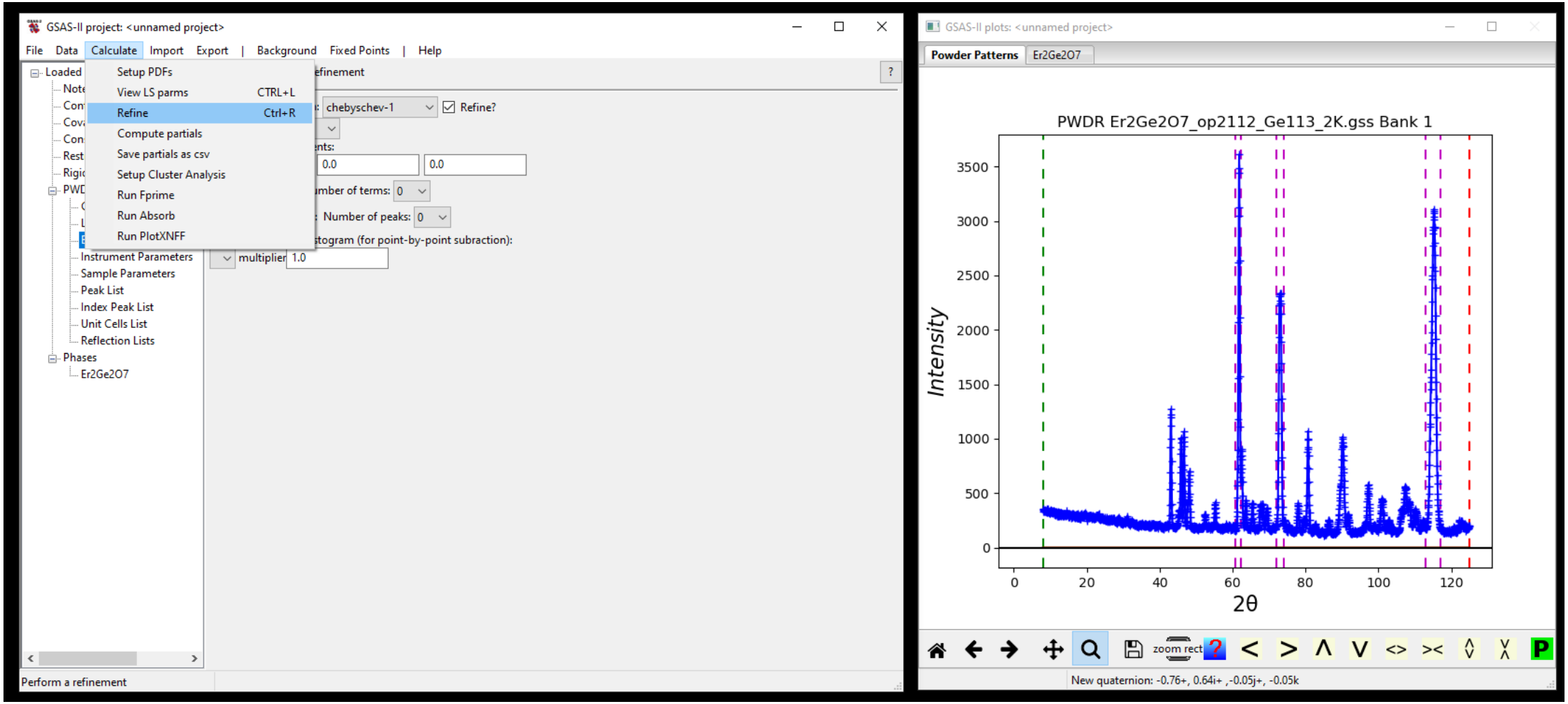
Unlike GSAS or FullProf, GSAS-II has a tree structure. You can see there's now a branching node for the historgram and another for the phases as well as some higher-level nodes with global parameters, constraints, refinement statistics, etc

Let's start refining



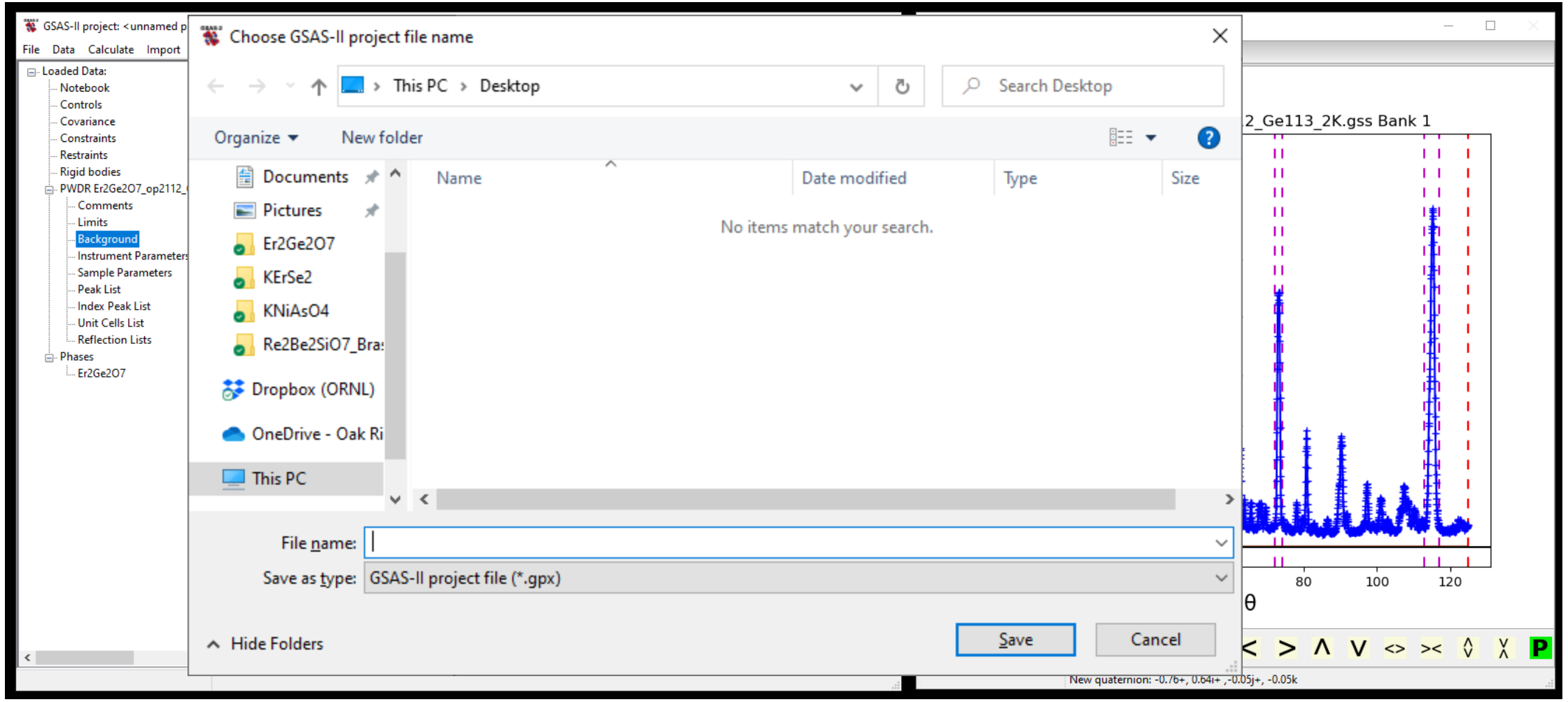
If you check through the tree and associated tabs you can see what is refining (e.g. three background terms, a scale factor). The structural parameters are not refining. Let's try a refinement and see what happens.

Let's start refining



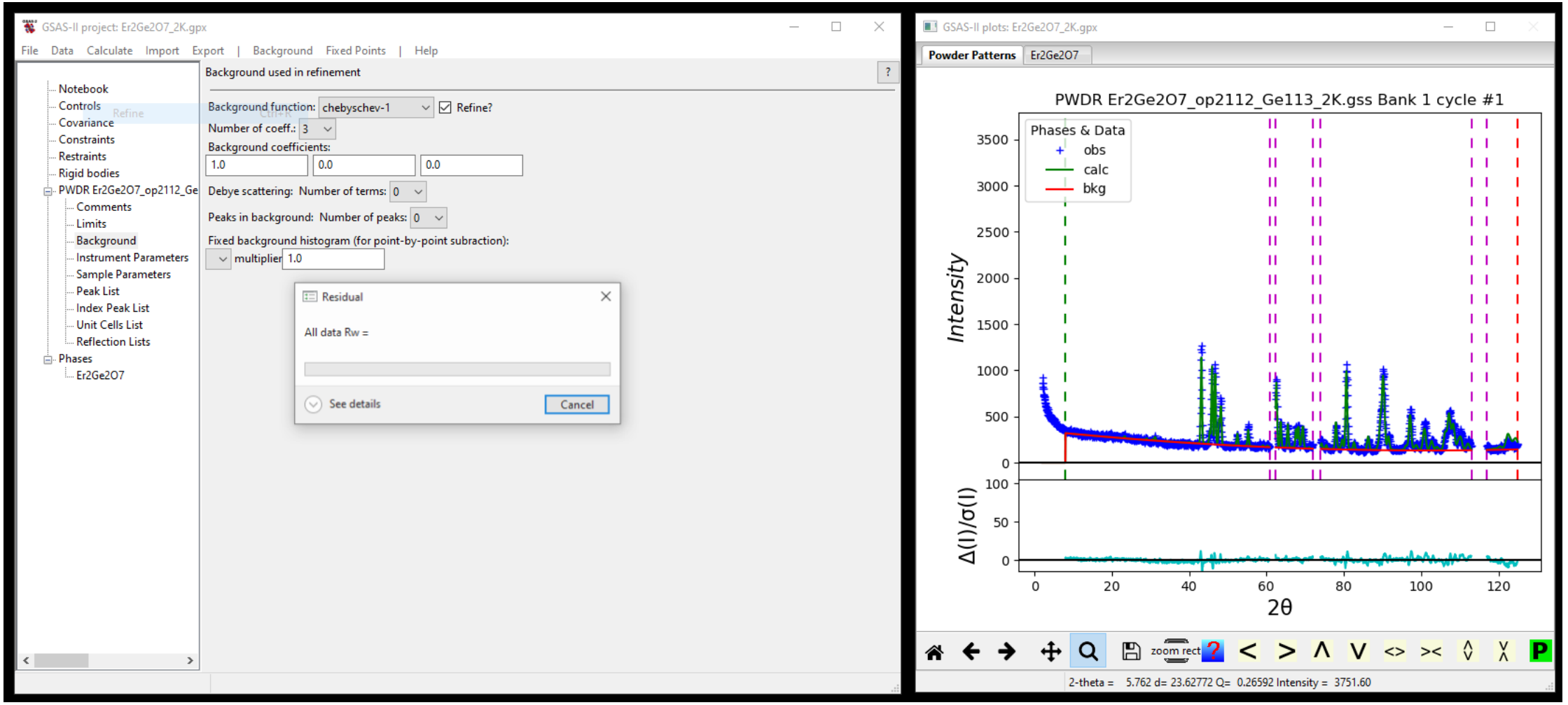
Go to Calculate -> Refine

Let's start refining



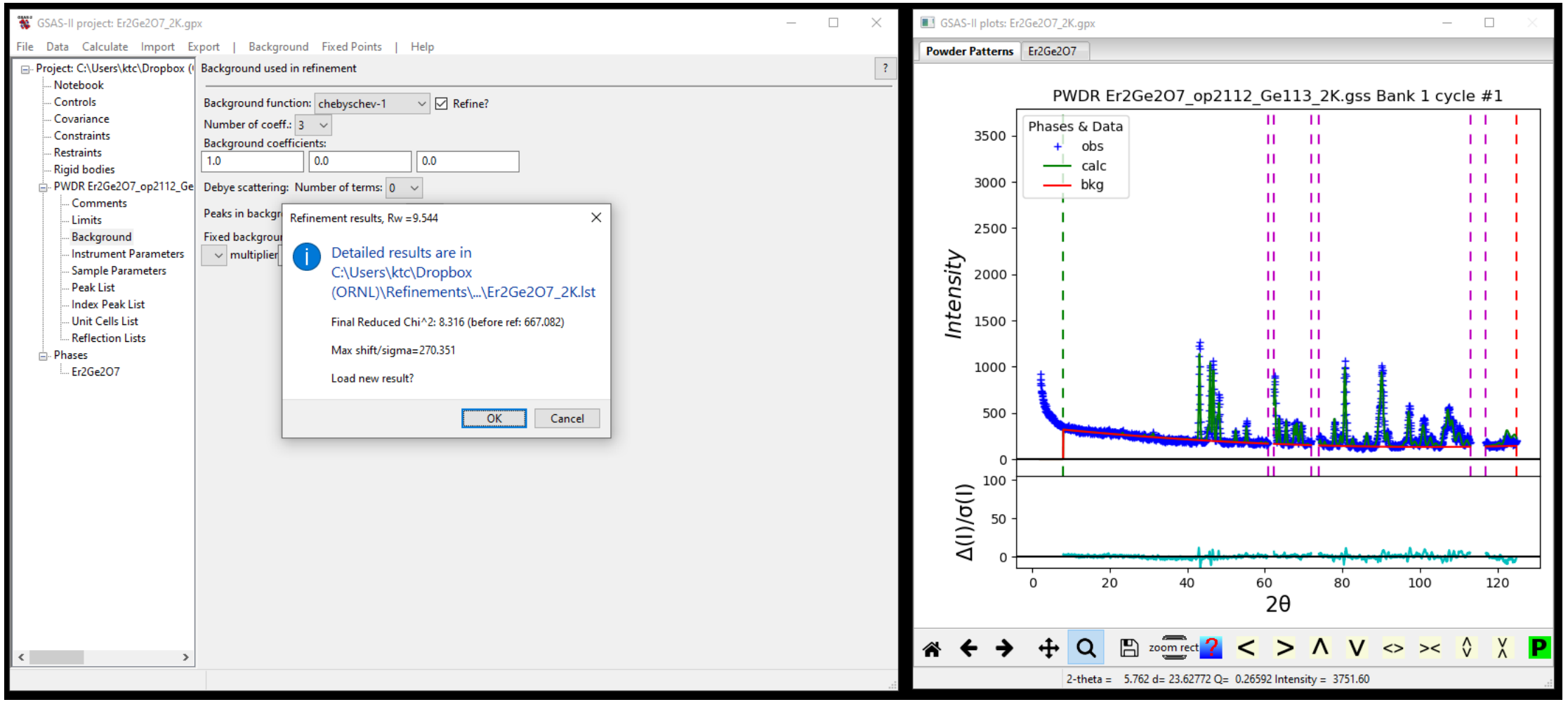
Ope, need to save it first (make sure to do so in the same location as the rest of the files). I'll name it Er2Ge2O7_2K.

Running a refinement



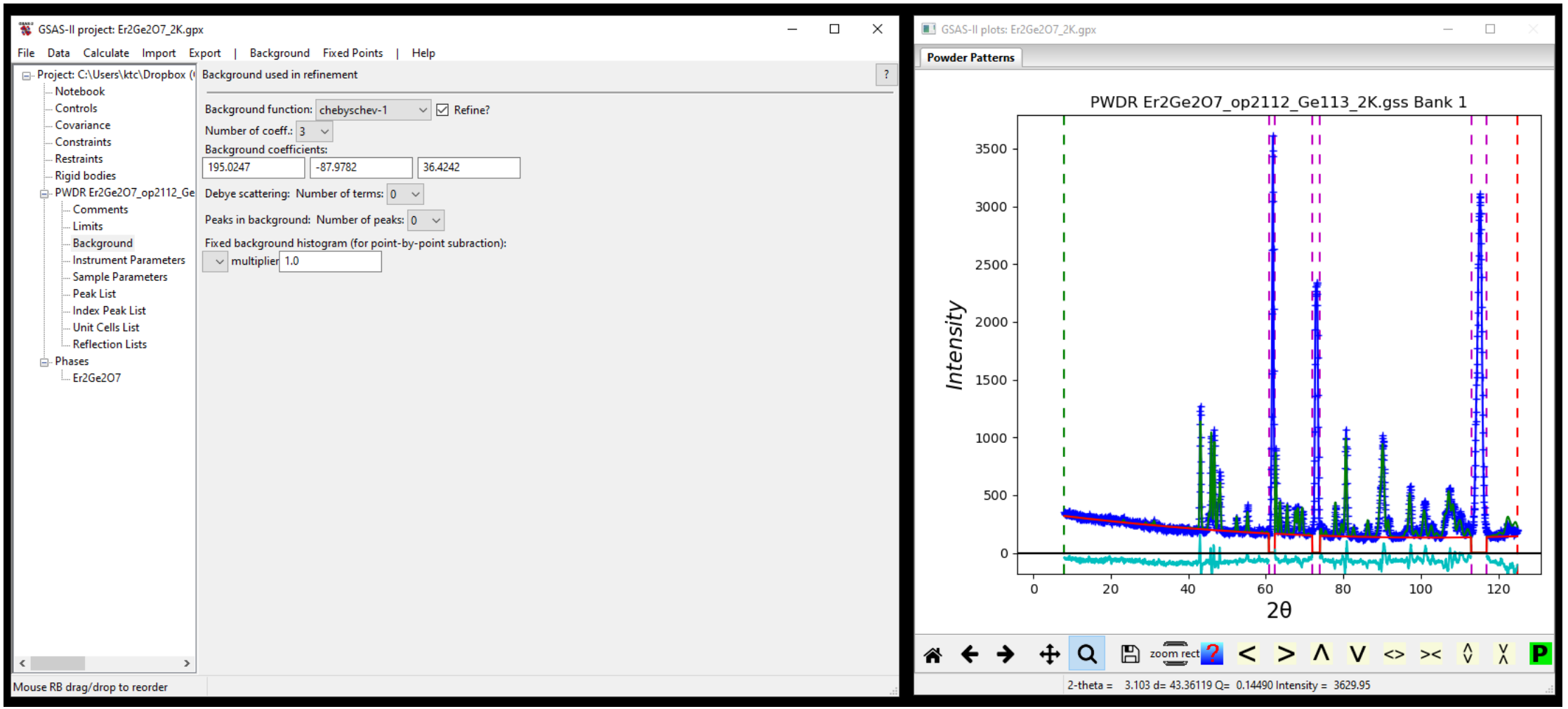
Should get something like this, which will go through a couple cycles…

Running a refinement



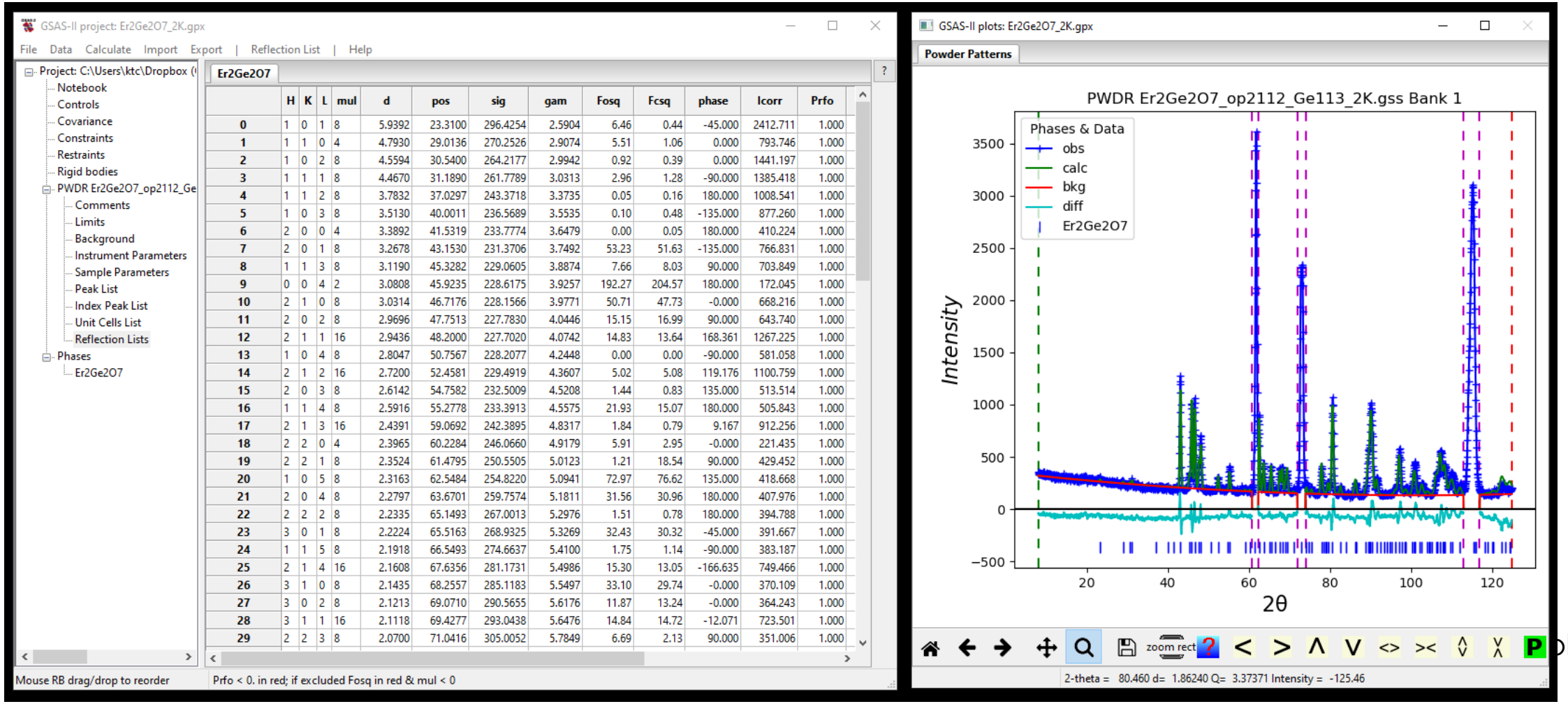
And then it will ask whether you wan to load the refinement results – click 'OK'

Running a refinement



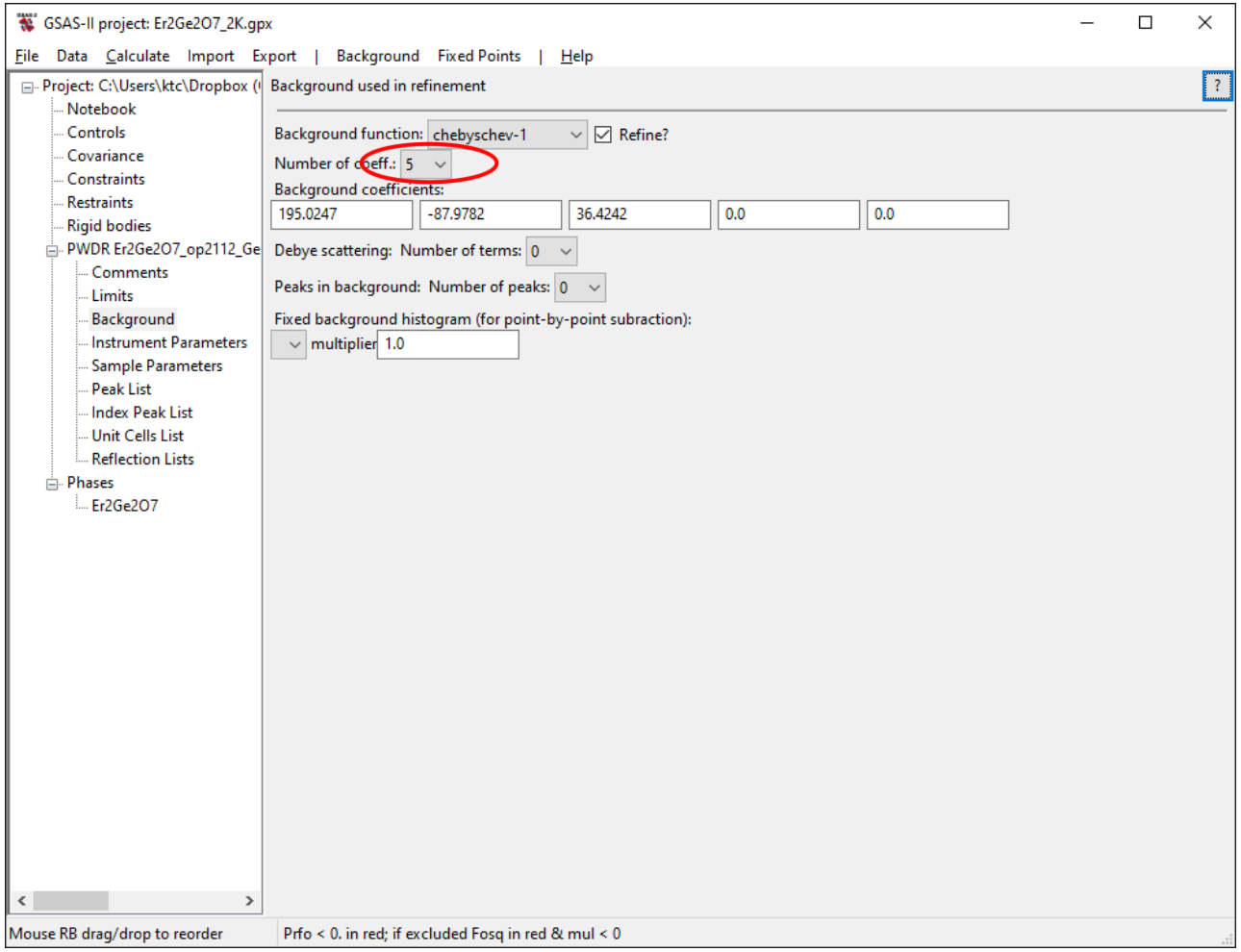
We can see the peaks are roughly in the right positions, but their intensities and the background is off – we need to add more background terms and flag some structural parameters to refine

Where are our peak indexes?



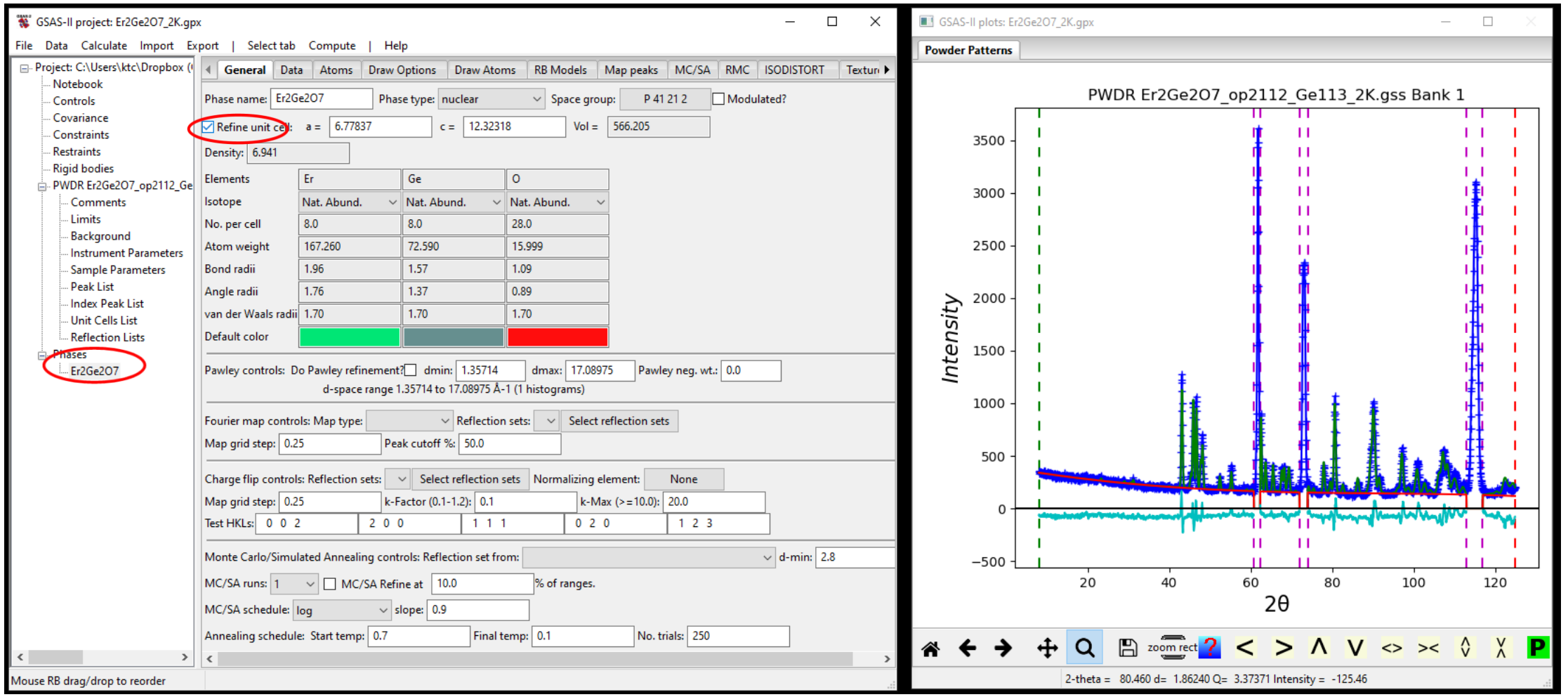
We can check a little more carefully by looking at the peak indexes. Go to the 'Reflection Lists' node in the Histogram tree and then click the home button on the Powder Patterns tab of our plotting window (to zoom out)

Optimizing the fit – background fitting



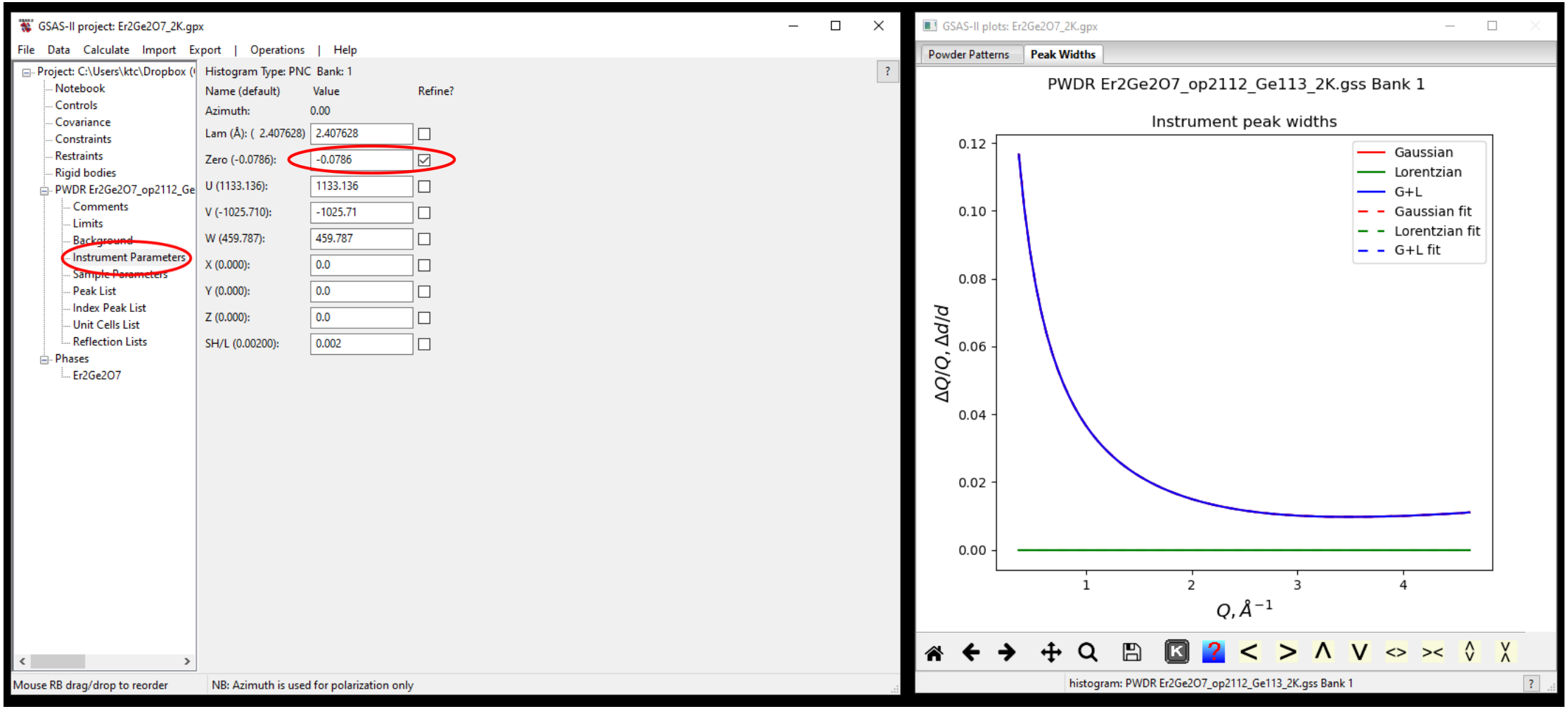
Okays let's start flagging some refinement parameters – starting with adding more background terms (I'm going to use 5 HB-2A's background is fairly isotropic, here we are seeing some paramagnetic scattering from Er's large moment).
Now refine.

Optimizing the fit – structural parameters



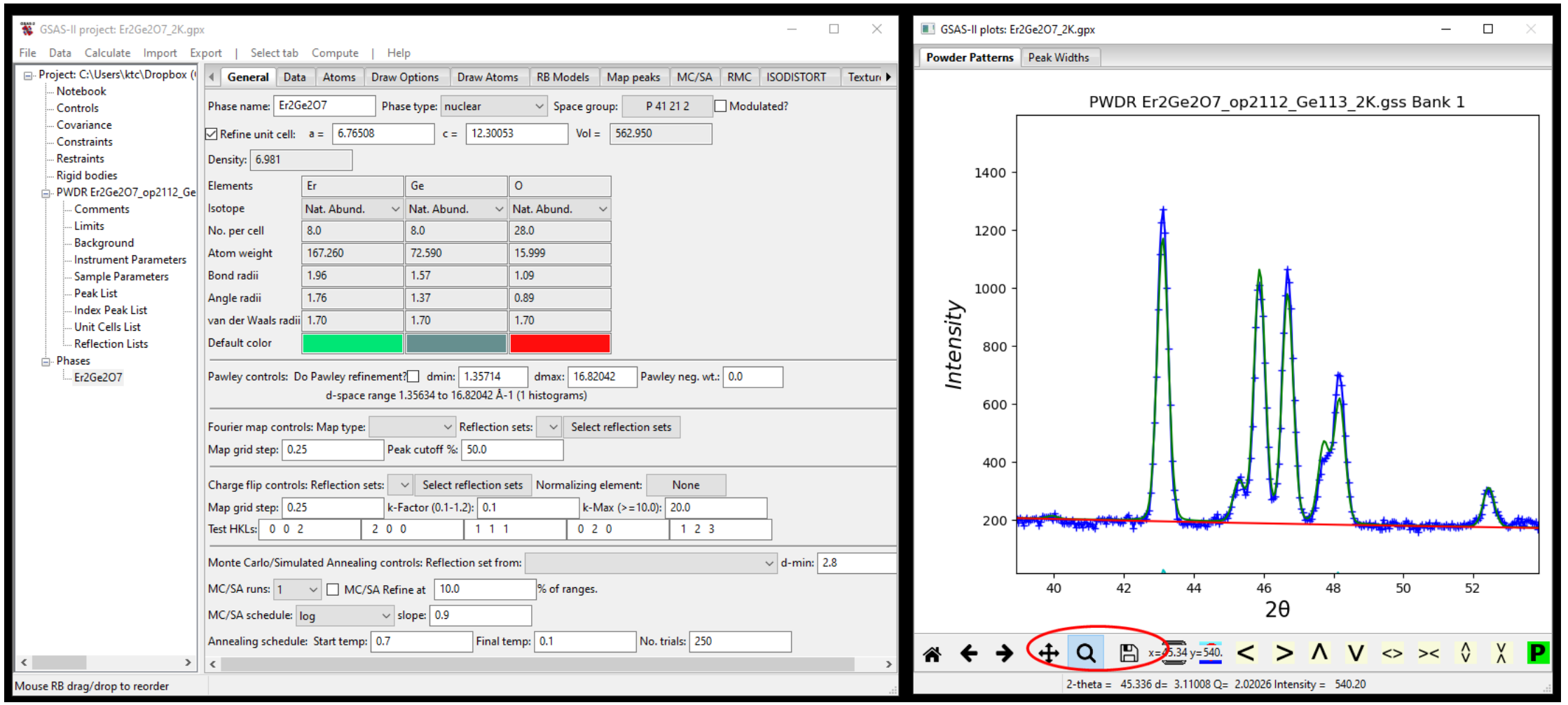
Looking better, now let's add the lattice parameters
Go to the phase node and click 'Refine unit cell'

Optimizing the fit – instrument parameters



Looking better, now let's add the 'zero' (this is the starting 2θ of the detector bank and is often off a bit)
Go to the Histogram node and flag the Zero parameter under Instrument parameters to refine

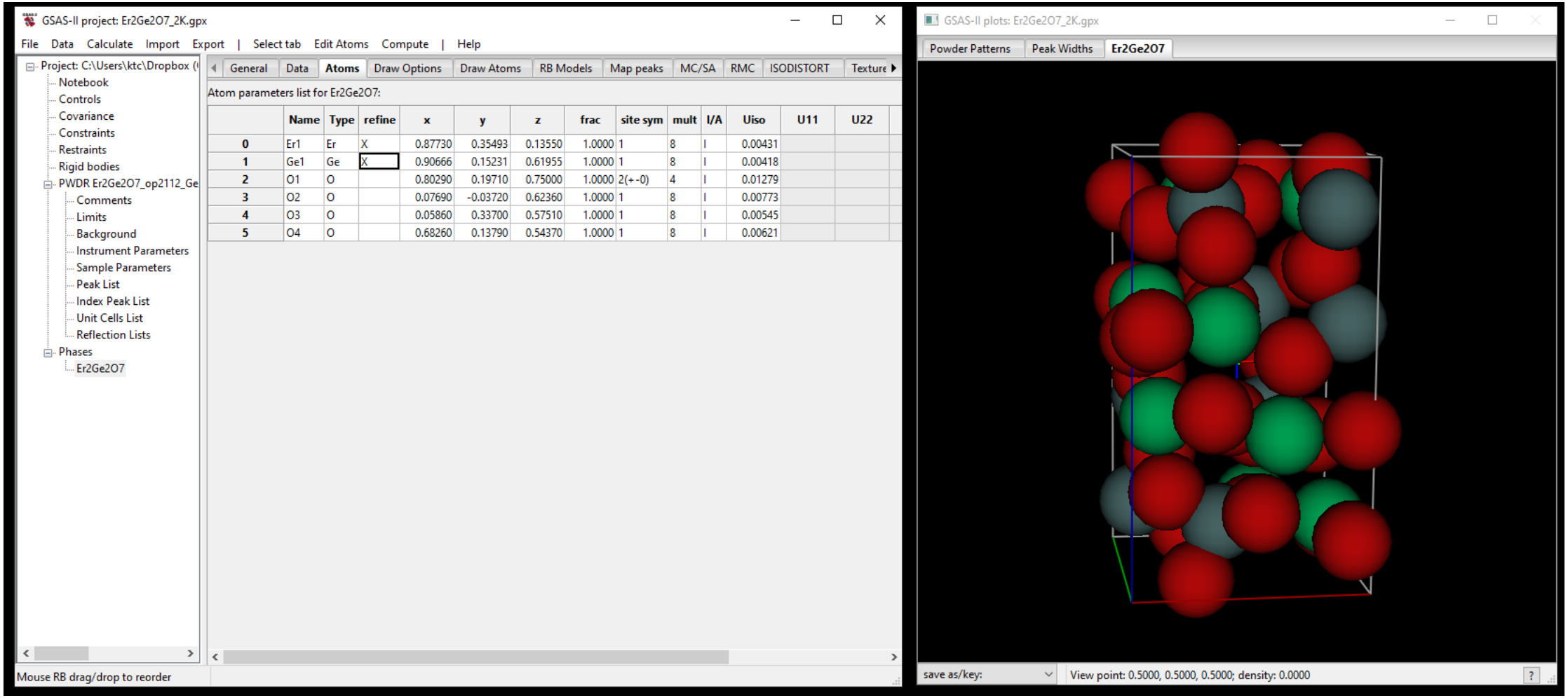
Optimizing the fit – instrument parameters



As you are fitting you should monitor your peaks to see how the model is working and to diagnose what might be off

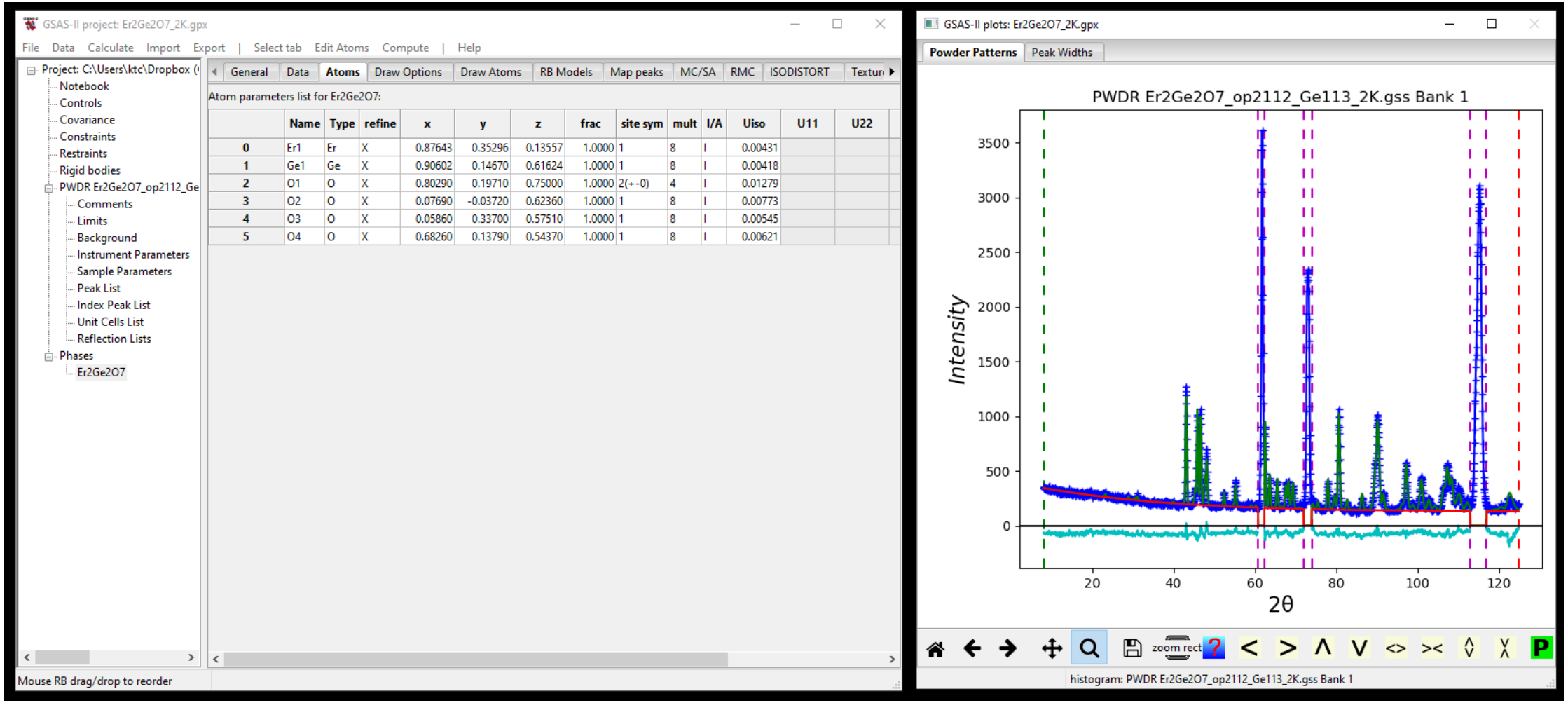
Here, the intensities are a little off so next we can flag internal parameters

Optimizing the fit – internal parameters



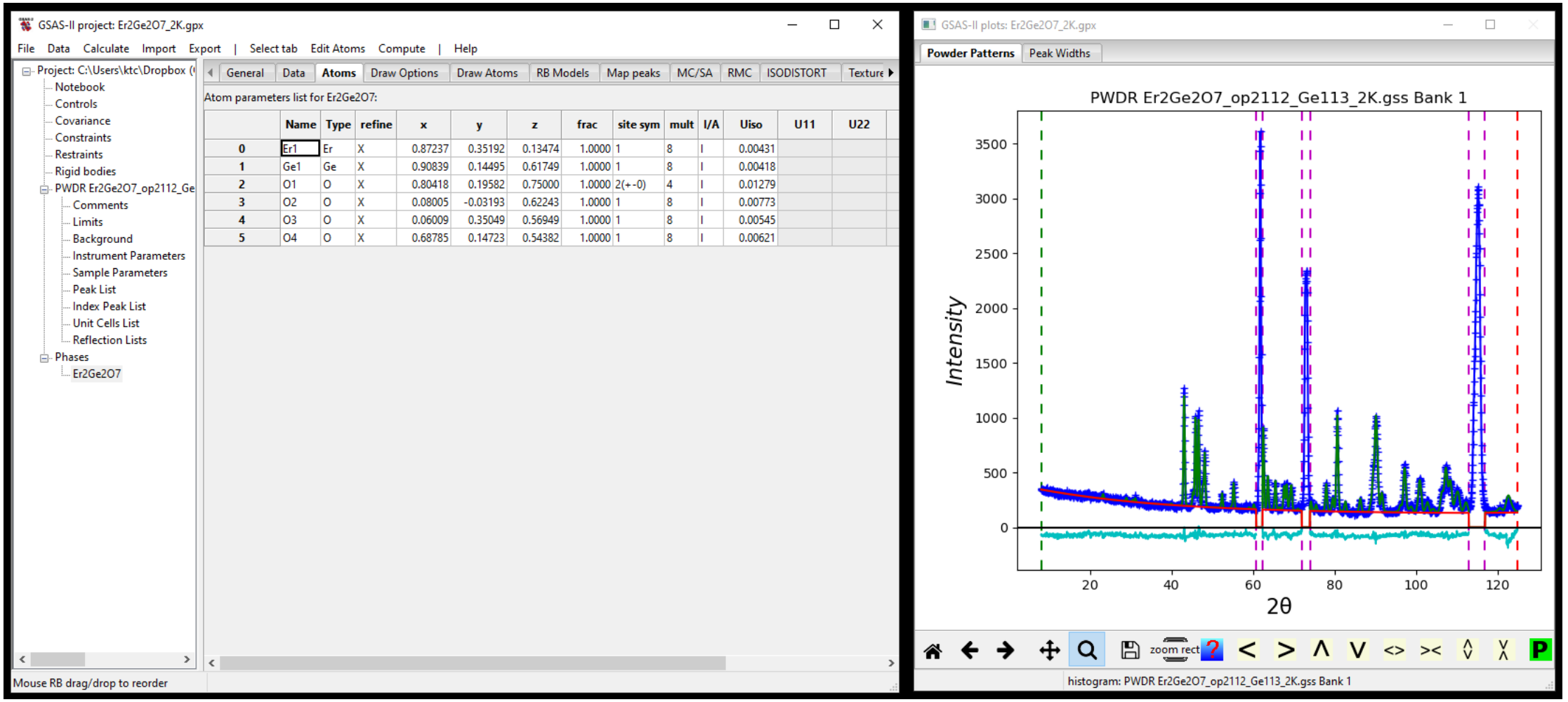
Now let's add the atomic positions and displacement parameters that were in the Atoms tab
NB: This should be done in small groups of atoms usually starting with the highest multiplicity and scattering strength to keep the refinement stable

Optimizing the fit – internal parameters



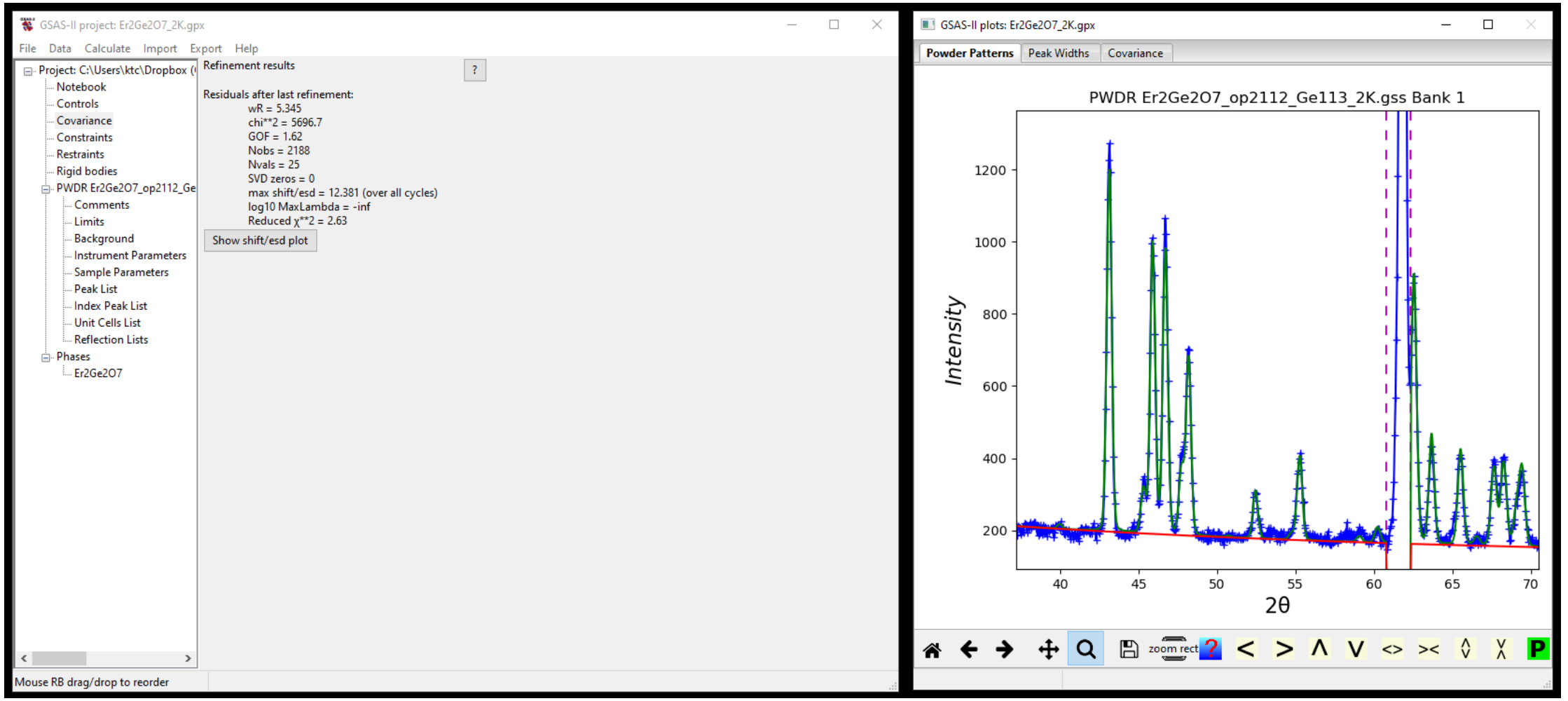
A little better, let's add the rest of the positions (we will not refine the Uiso parameters as this is very low temperature and we don't have very high in Q)

Optimizing the fit



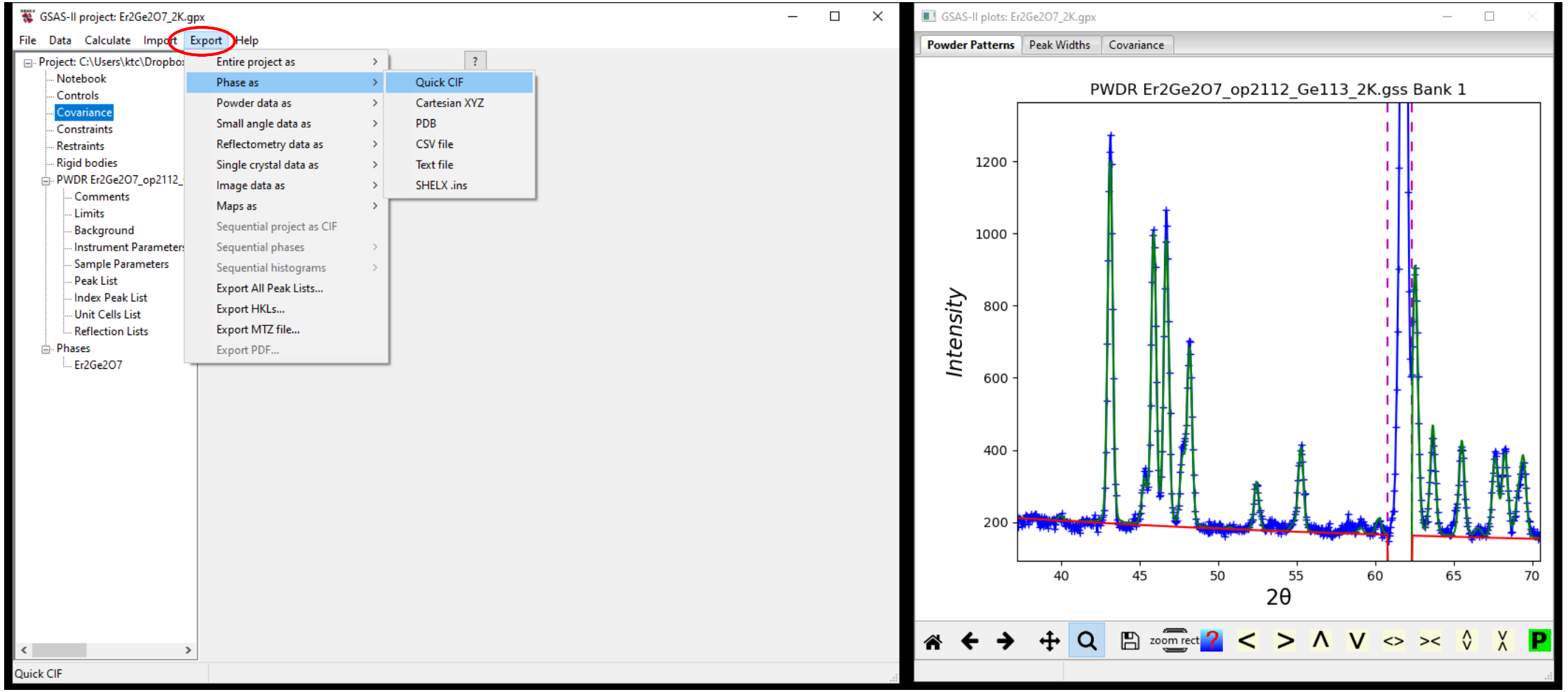
Another modest improvement, lets take a closer look

Optimizing the fit



Check the 'Covariance' node to see typical fit residuals
(it will also pop-up a plot of the Covariance matrix)
More importantly zoom in on peaks in the Powder Pattern to check fit quality, this looks pretty good. This is good enough for this tutorial but note that were this to be your work you should dig deeper (check out the tutorials on the GSAS page)

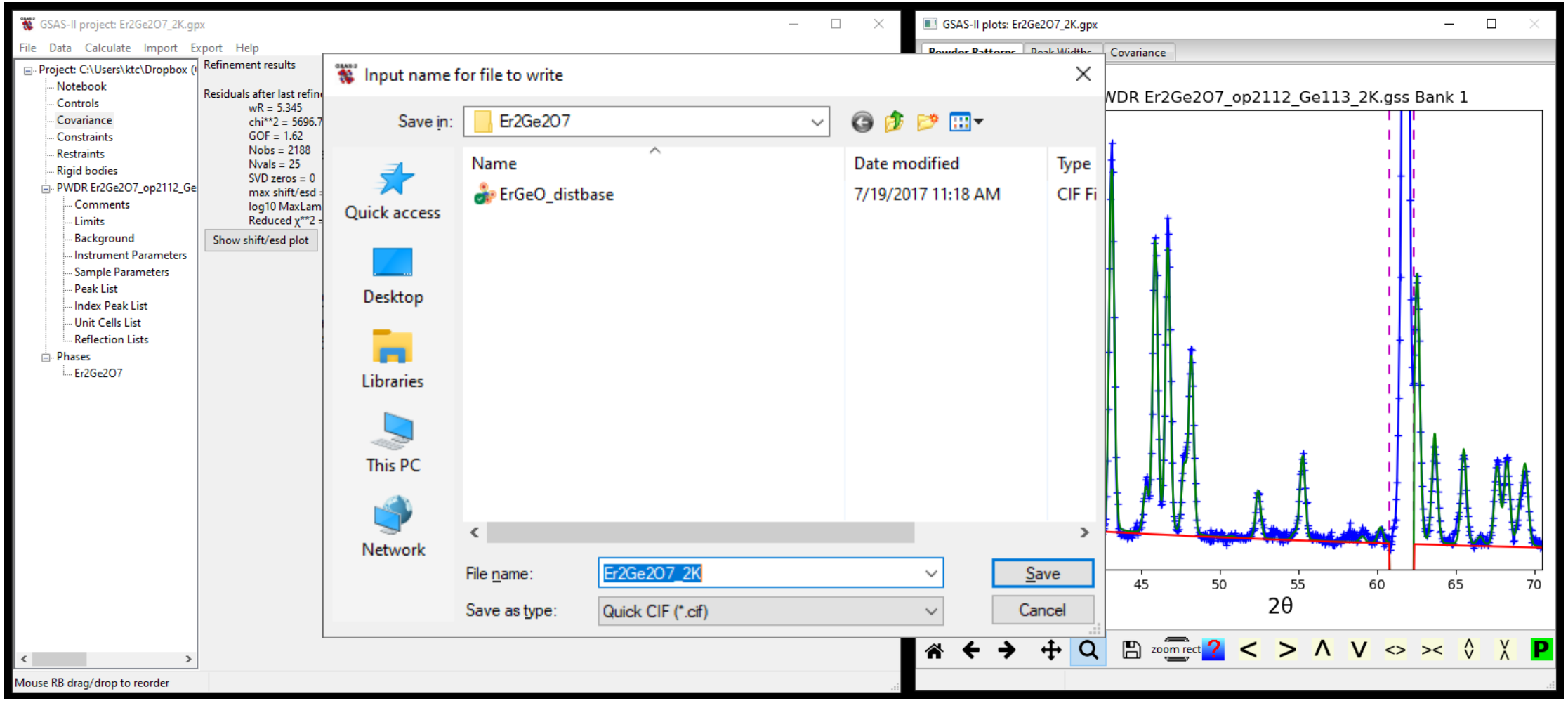
Exporting a cif for use at low temperature



Now let's export this structural data to use in our 0.5 K refinement

Go to Export -> Phase as -> Quick CIF

Optimizing the fit



Go ahead and save

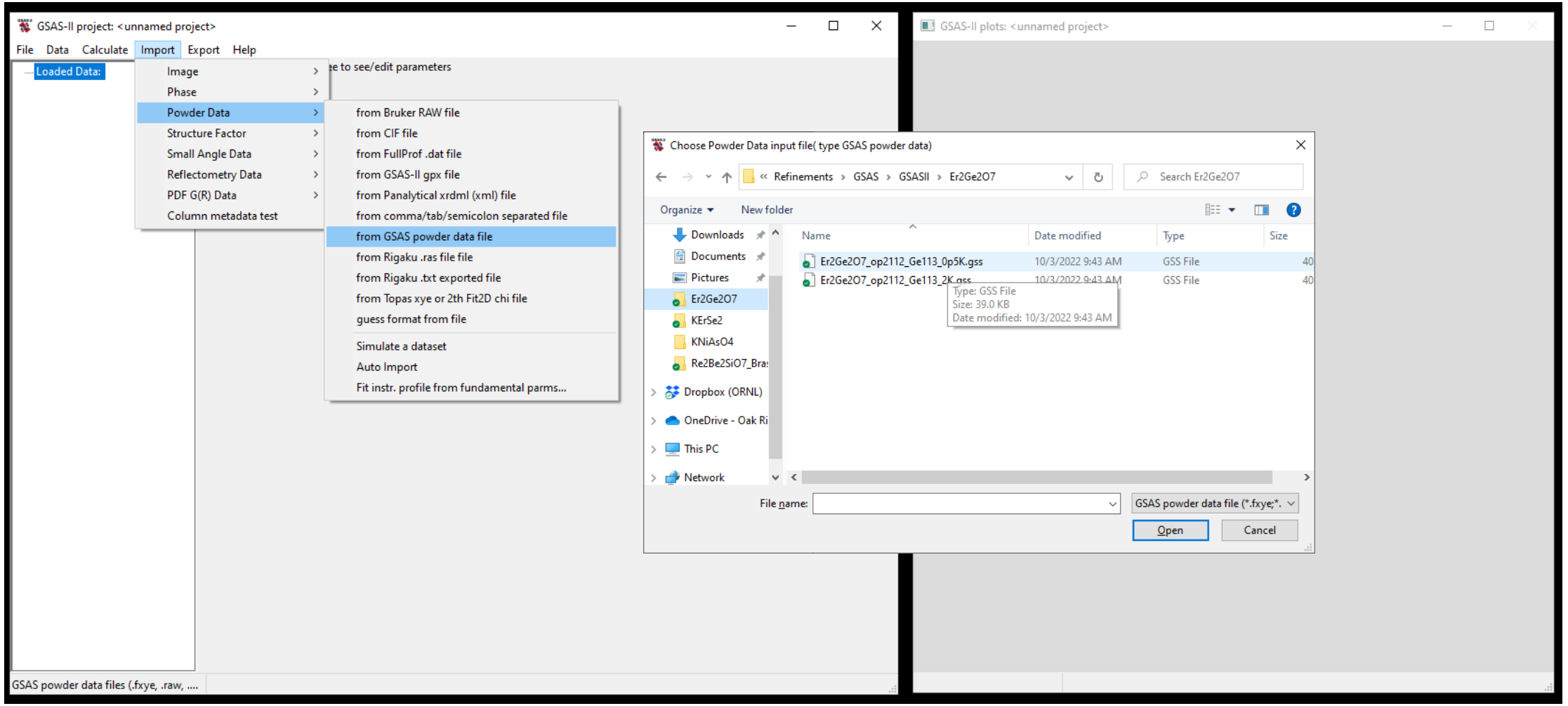
We are ready to move on to the magnetic refinement now!

OAK RIDGE National Laboratory

Magnetic Refinement

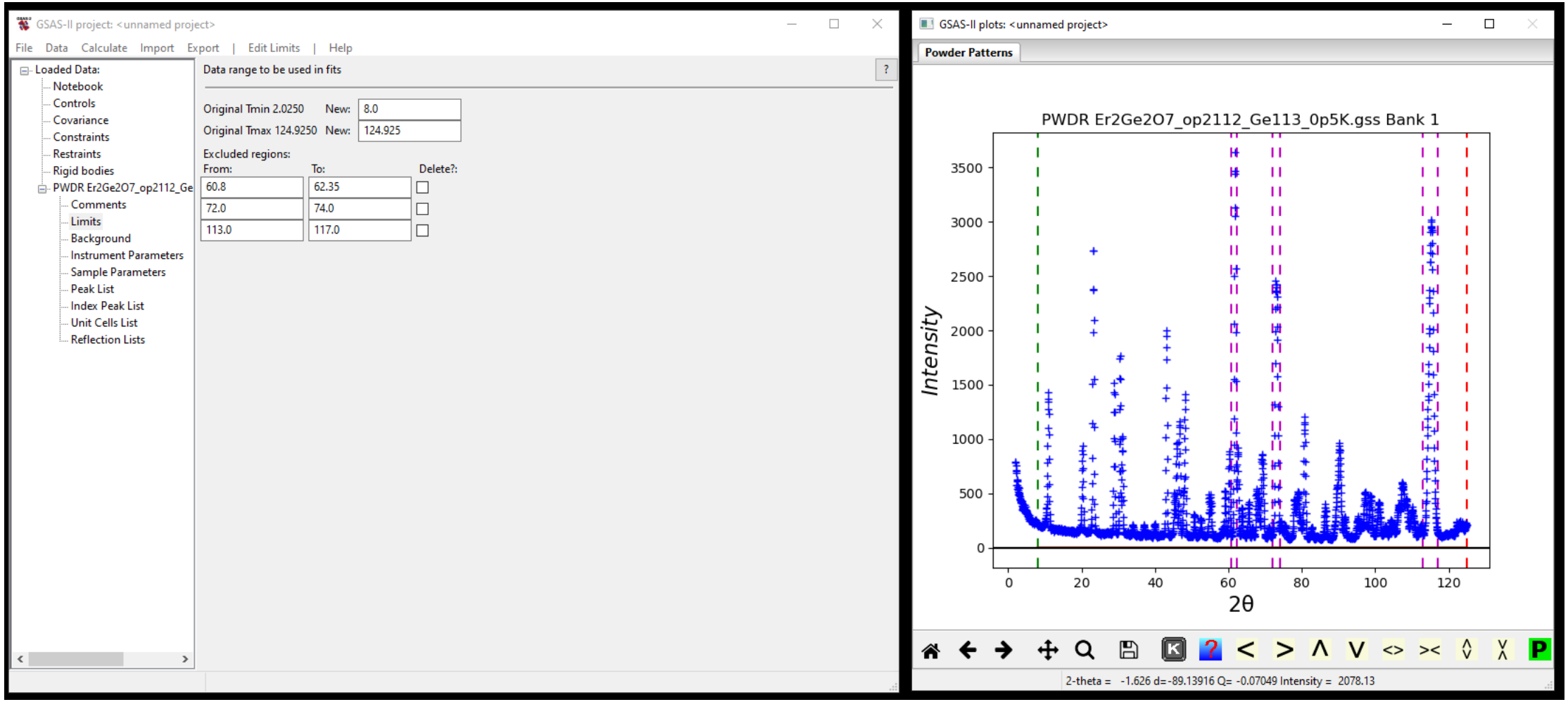
- We'll start from the 2 K structure we just optimized and use it to first fit the 0.5 K nuclear structure and then solve the magnetic structure

Starting the low temperature refinement



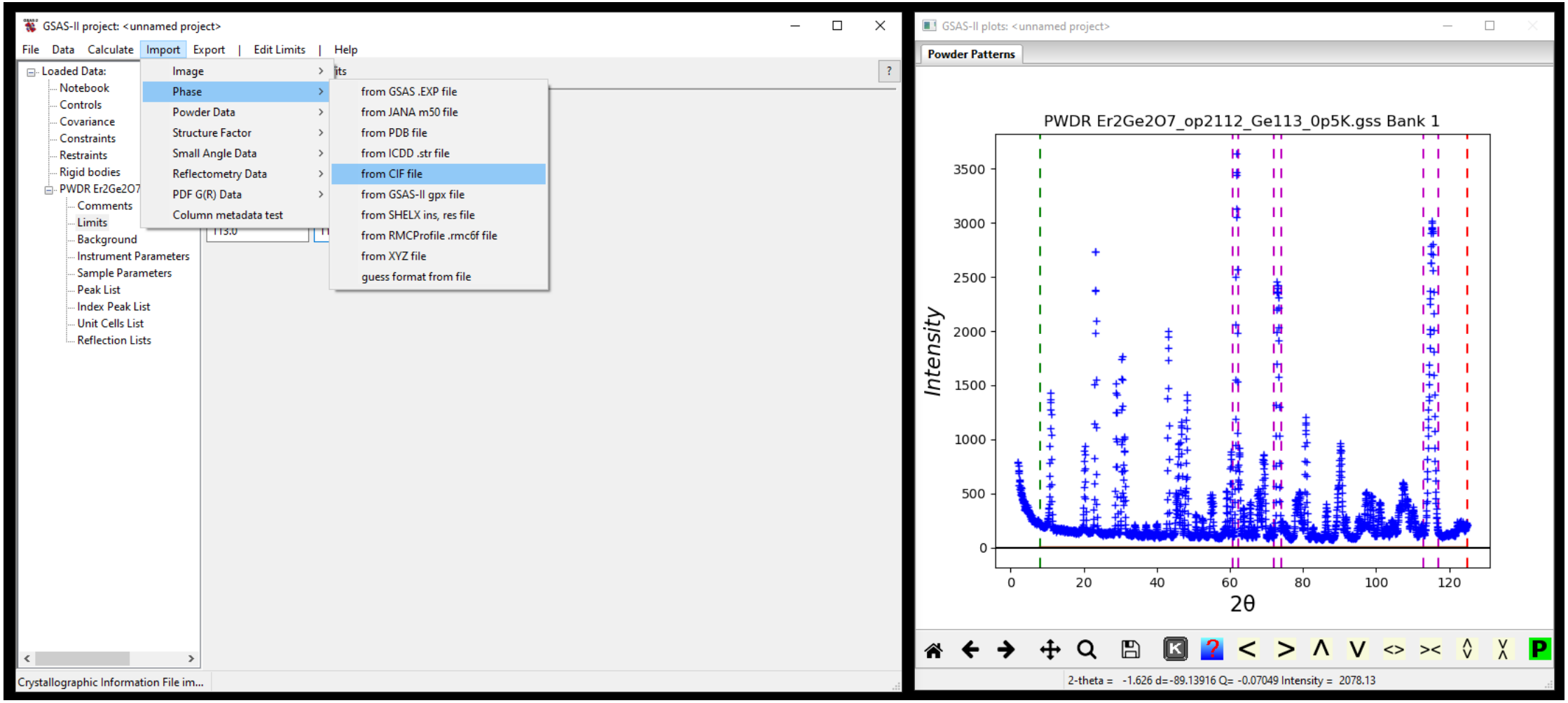
Start a new GSAS-II project
And load the low temperature data (Er2Ge2O7_op2112_Ge113_0p5K) and instrument parameters file as before

Starting the low temperature refinement



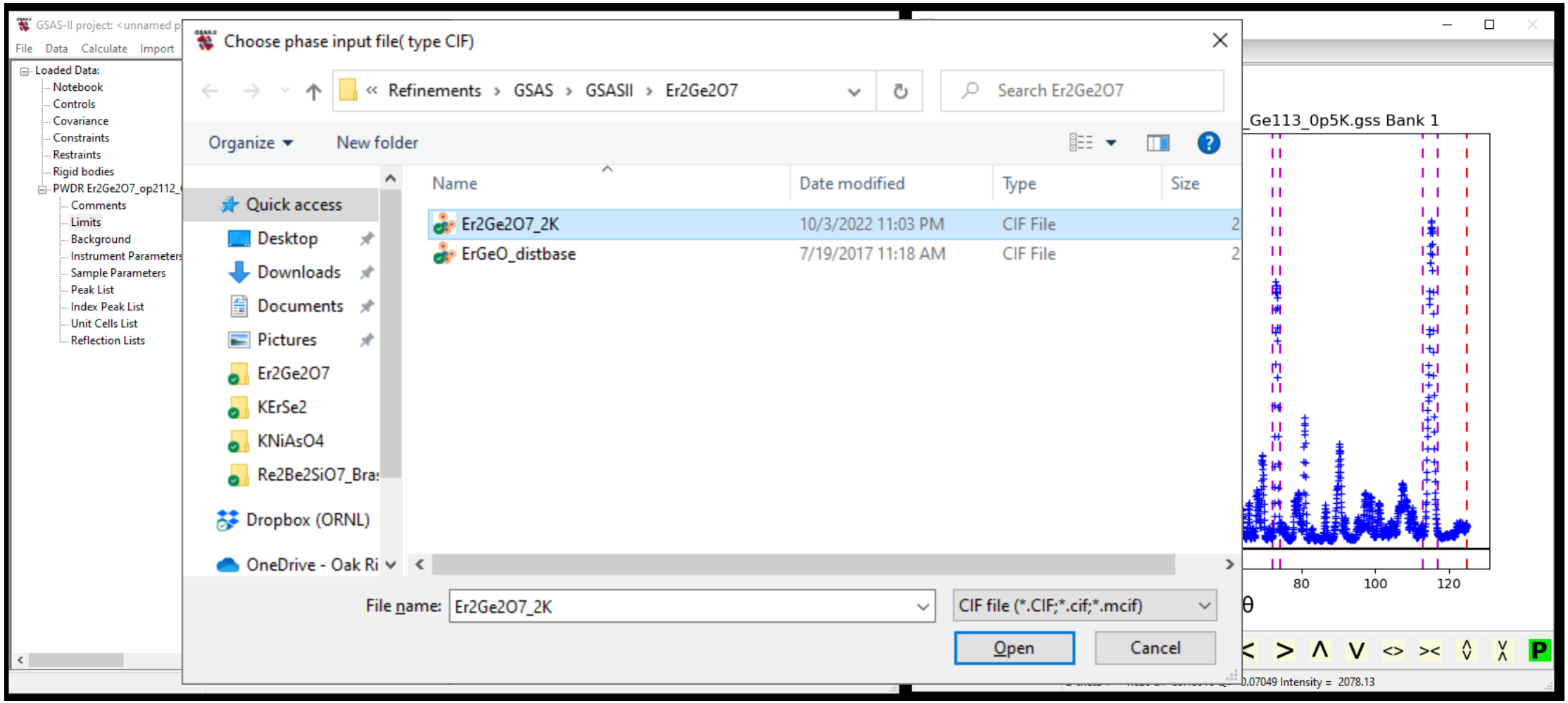
Set the limits as before
(look at all those new peaks!)

Loading the 20 K structural data



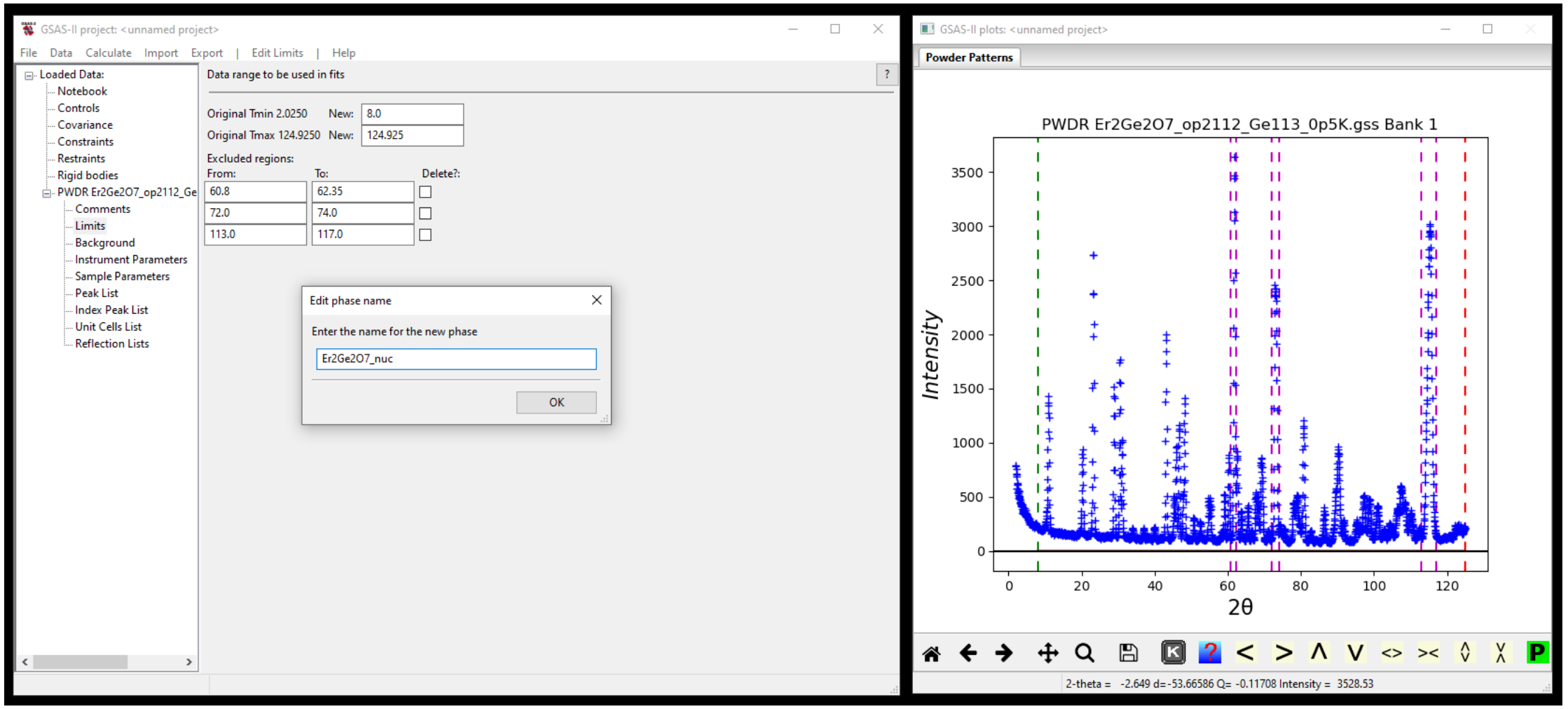
Go to Import -> Phase -> from CIF file (as before)

Loading the 20 K structural data



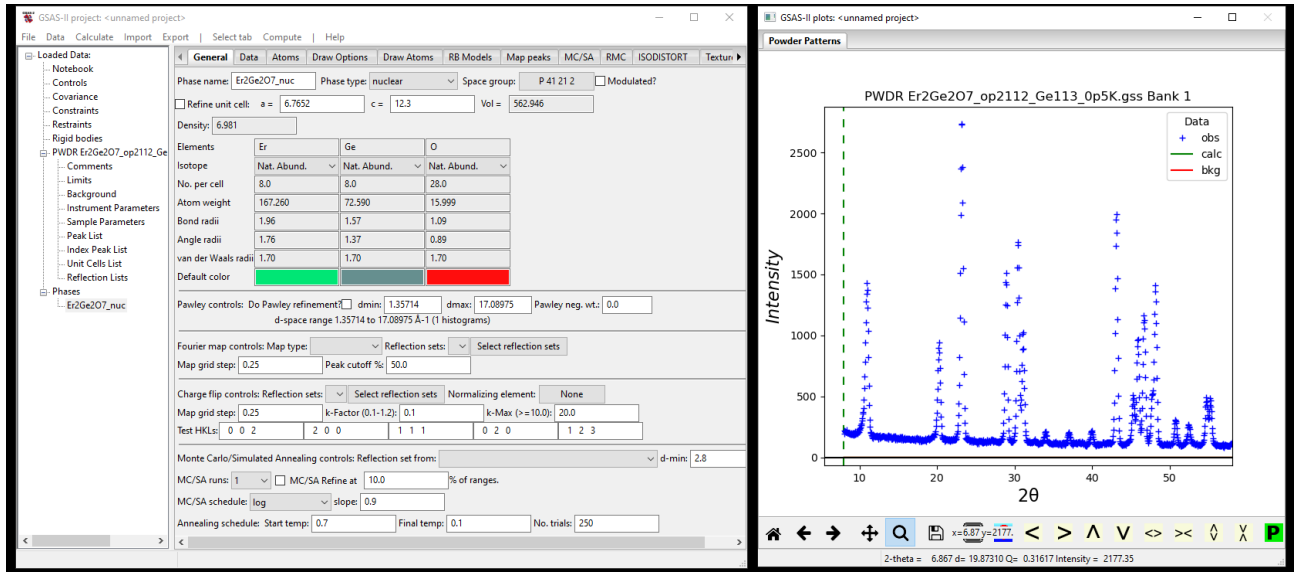
Select your 2 K cif file

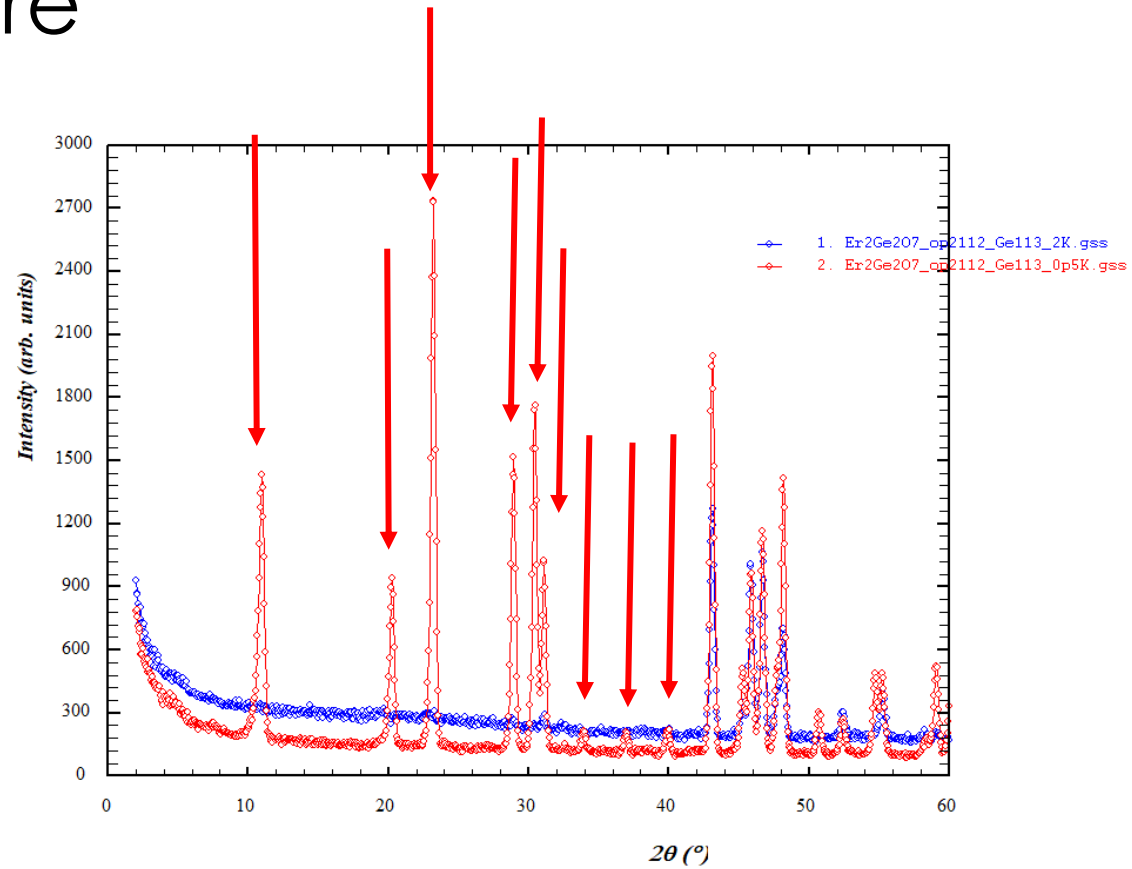
Loading the 100 K structural data



Again, give it a name meaningful to you – this time I'll name it Er2Ge2O7_nuc and add it to the histogram (as before)

Starting with the nuclear structure

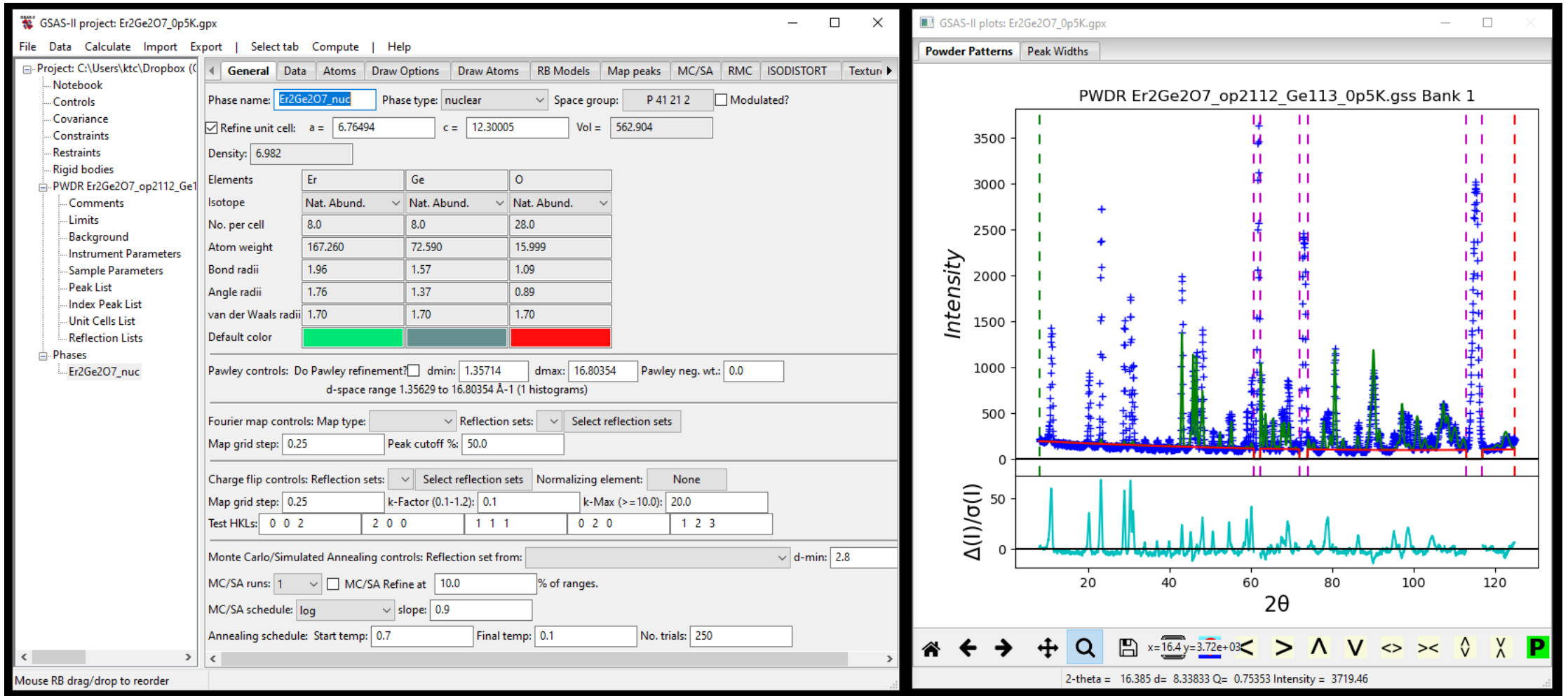




Now we have a nuclear phase which should be pretty close to where it needs to be (not too much thermal expansion expected between 2 and 0.5 K)

Looking at the Powder Pattern we can see a couple new low angle peaks already. If we plot both the 2 K and 0.5 K data (using your favorite plotter) we'll see there are many new peaks. However, before trying to solve the magnetic structure let's try to optimize the nuclear phase a little again (add 4 background terms, flag the unit cell, etc but do it one at a time and start with 3 background terms)

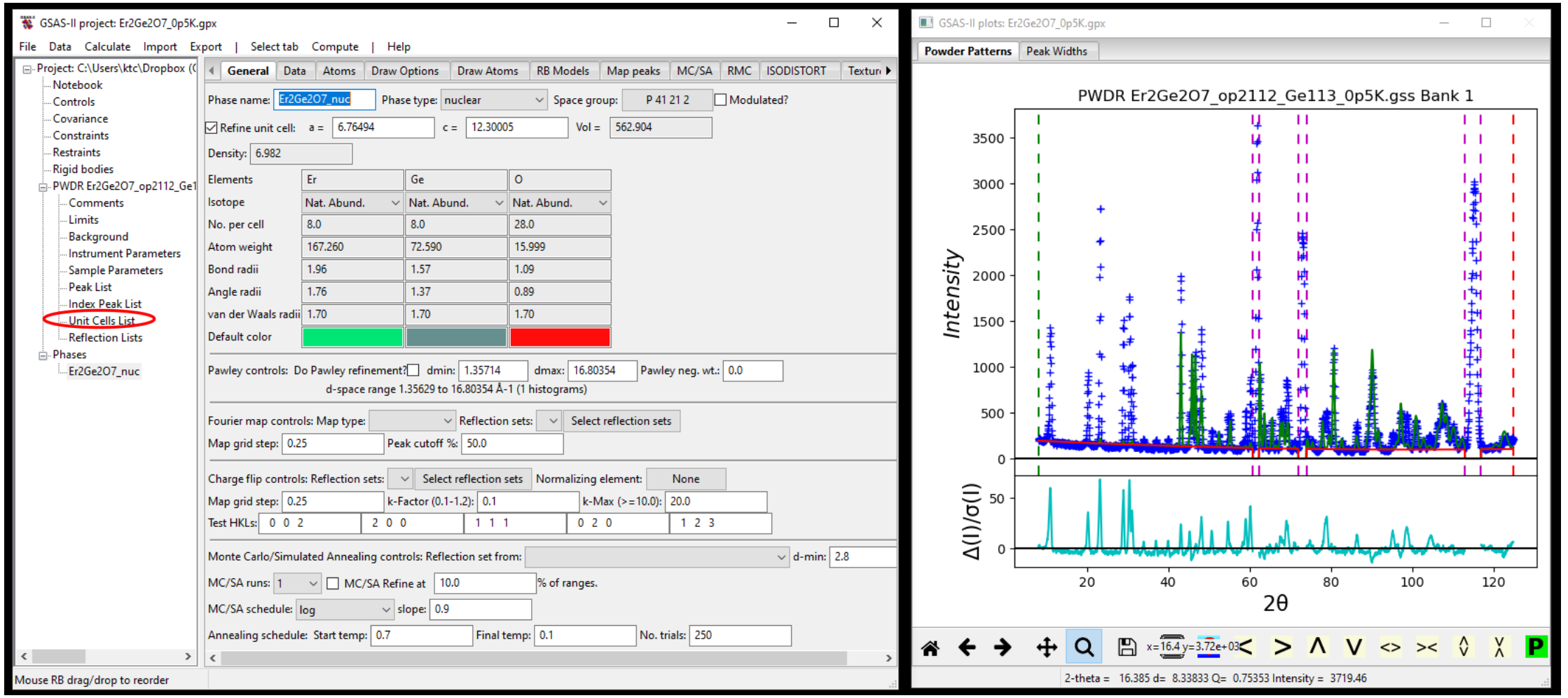
Starting with the nuclear structure



Looks bad but is a starting point – background is a bit off but we have large unfit peaks that it is trying to correct for. For us, this is a good enough position to start trying to solve the magnetic structure.

Our first step will be to use the Unit Cells List in the Histogram Node to try indexing the new peaks using integer multiples of the nuclear unit cell.

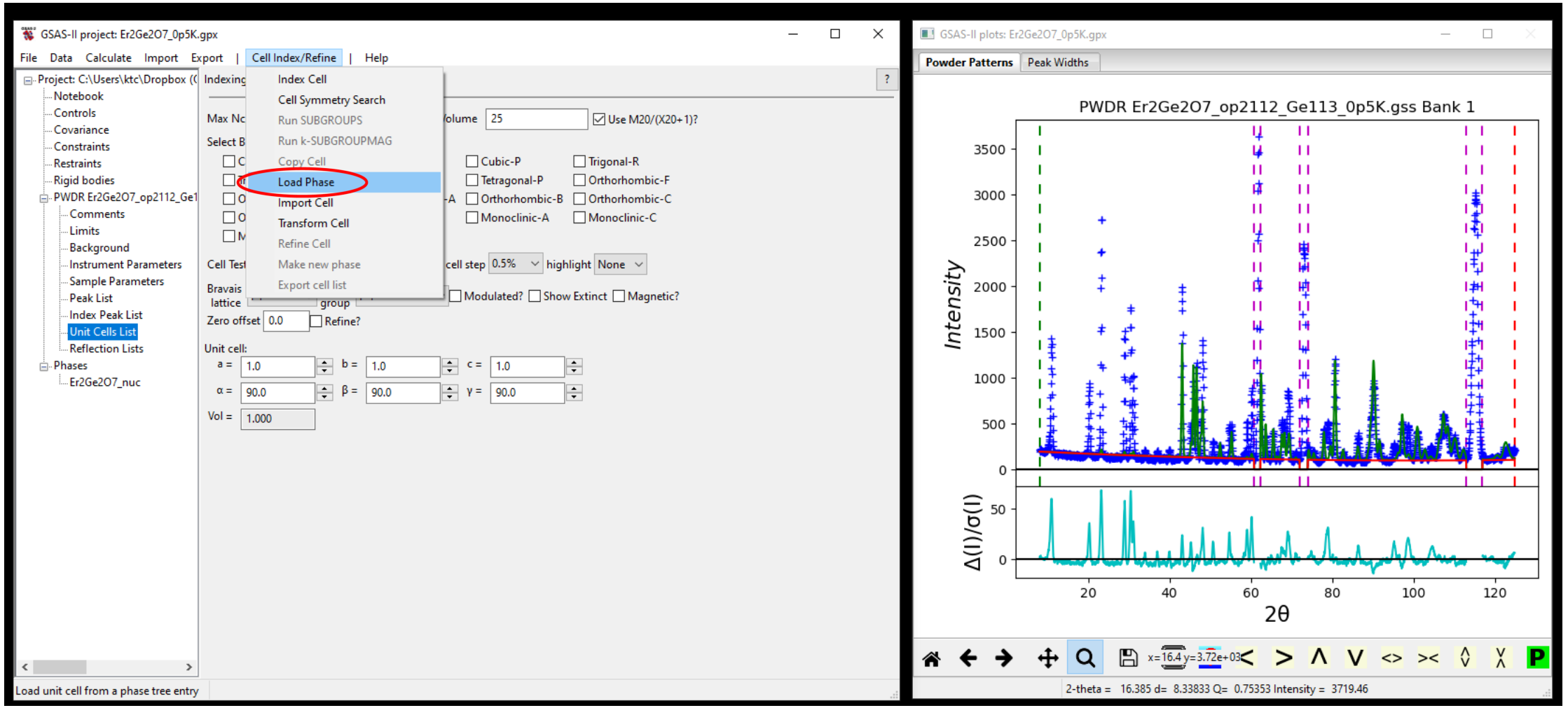
Indexing the new peaks



First go to the Unit Cells Node

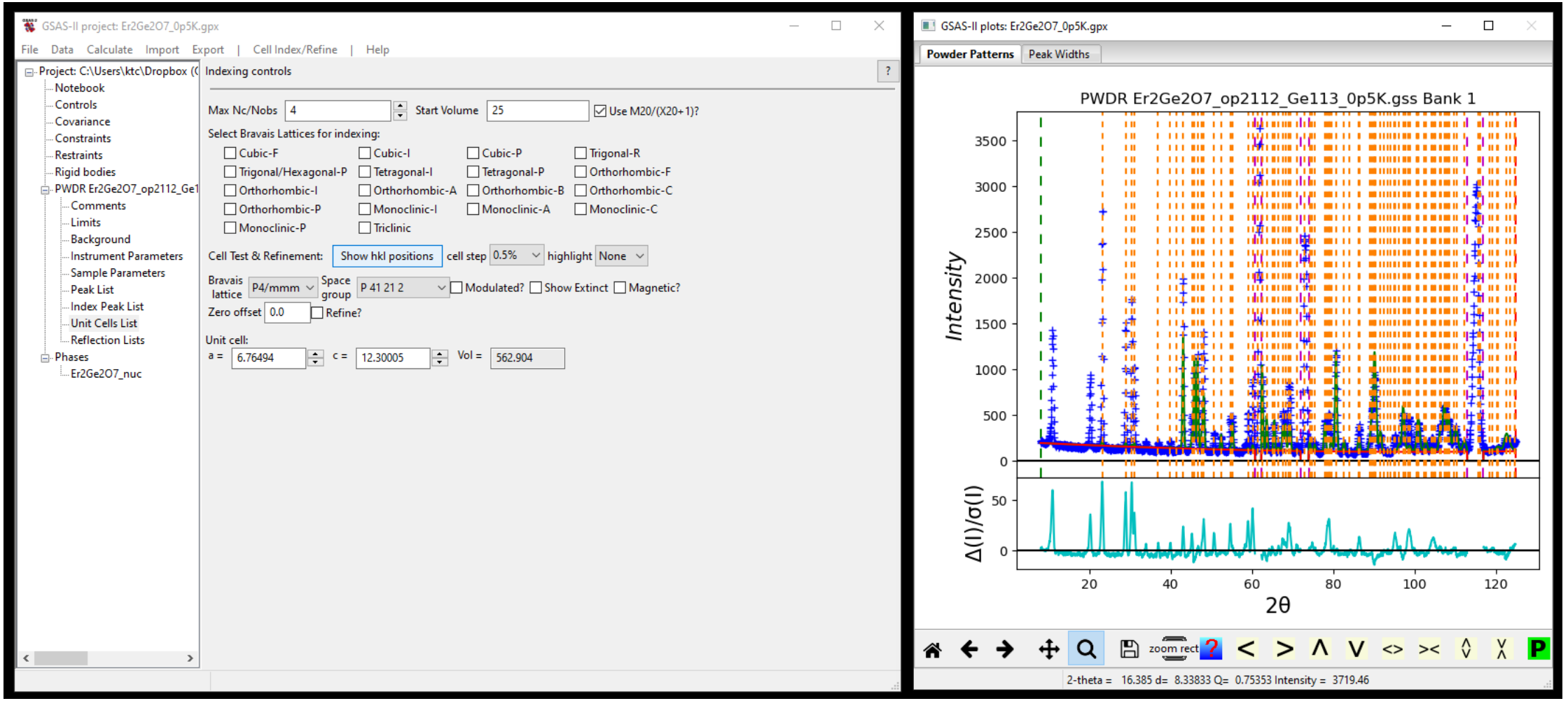
This will bring up a series of controls/options. We could put this all in manually, but you can also load the information from the nuclear phase. Let's do the latter.

Indexing the new peaks



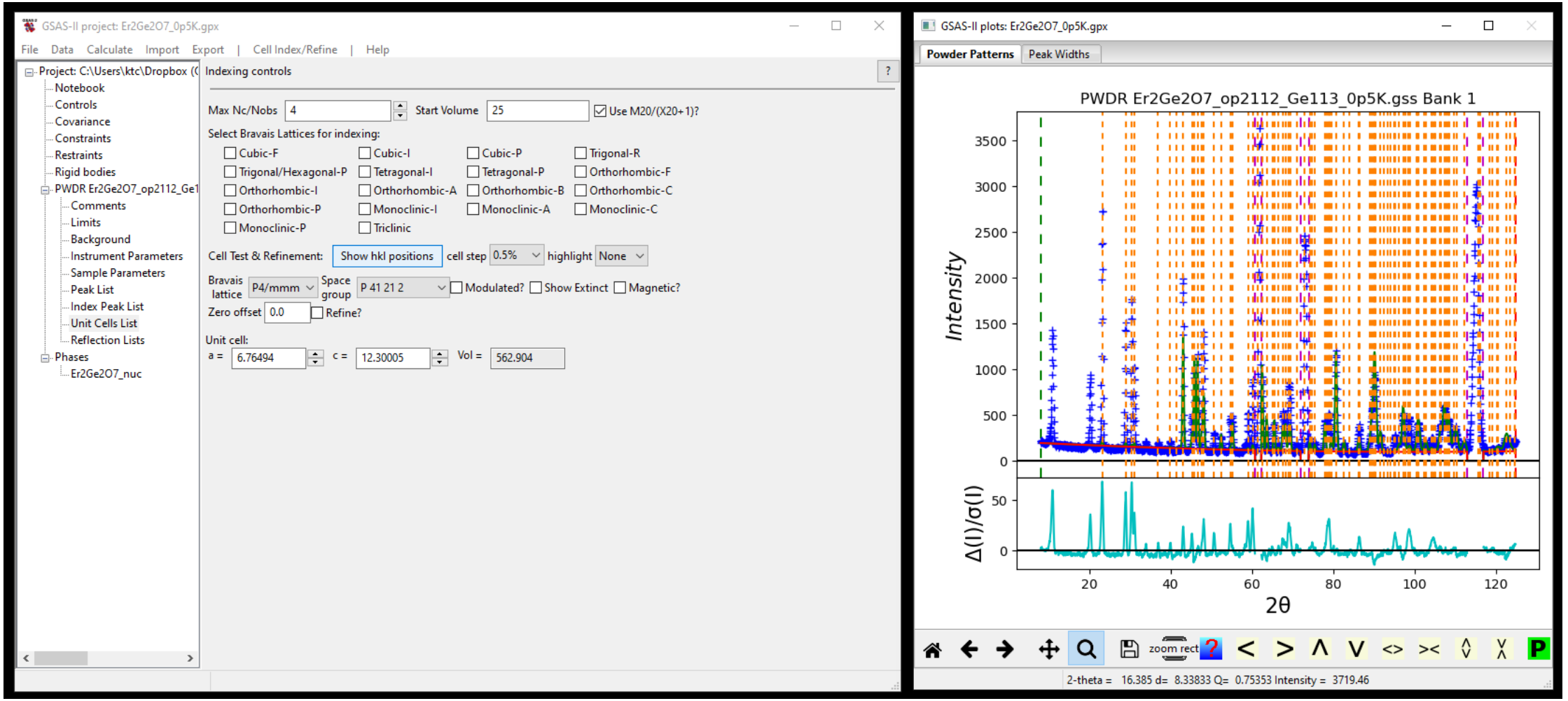
Click on ‘Cell Index/Refine’ in the toolbar and choose ‘Load Phase’

Indexing the new peaks



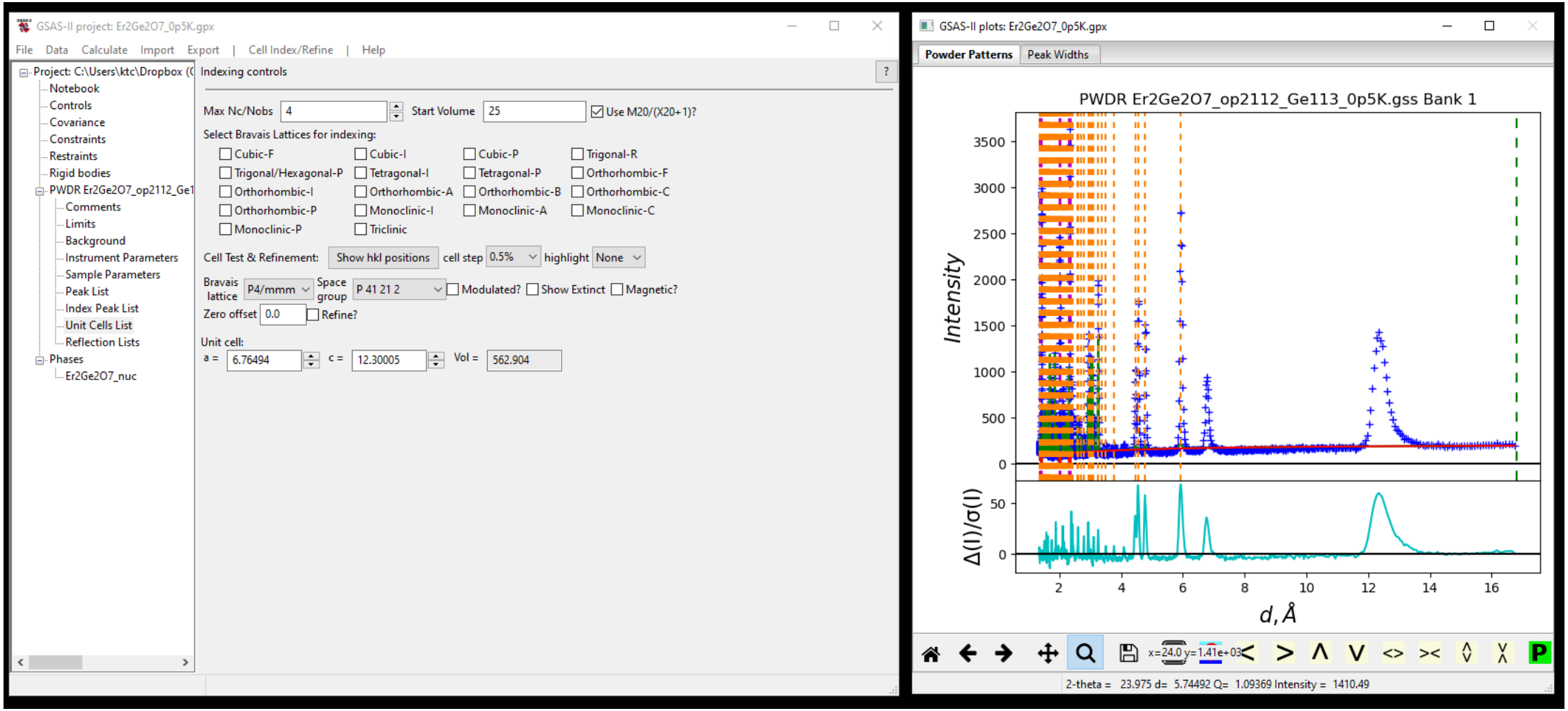
Since we only have one nuclear phase, it will automatically load it. You should see a bunch of vertical lines in the powder pattern which correspond to peak indices.

Indexing the new peaks



From here we can play with the lattice parameters and cell symmetry to try and index the new peaks.

Indexing the new peaks

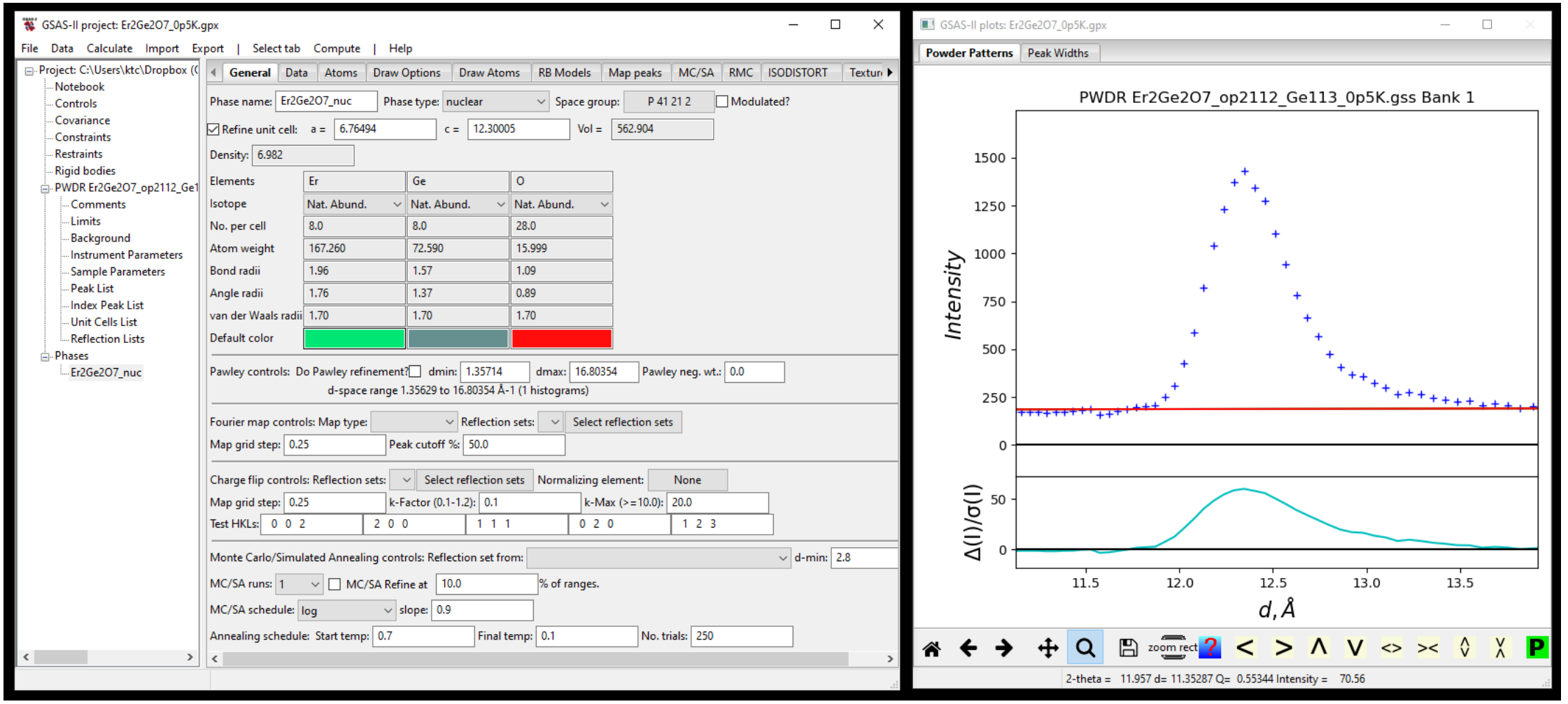


If we are lucky, changing to d-spacing might help us identify this low angle peak – in particular if it can be located at some multiple of a lattice parameter

To change the x-axis click on the plot and press ‘t’ on your keyboard. The units should shift to d-space (I’ve done so already in the plot above). If you wanted to view it in Q instead you could press ‘q’ on the keyboard.

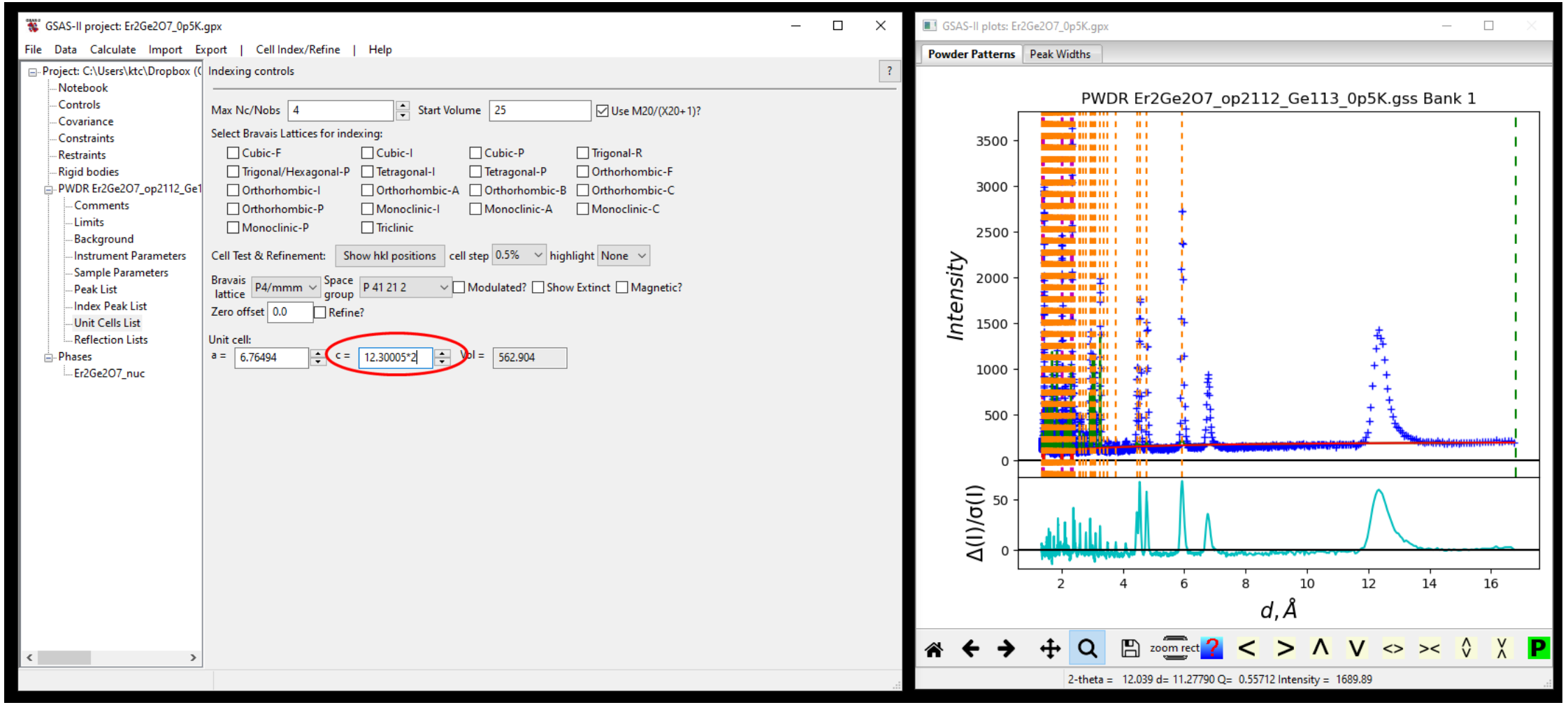
Indexing the new peaks

$$\text{Tetragonal:}\quad \frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2}$$



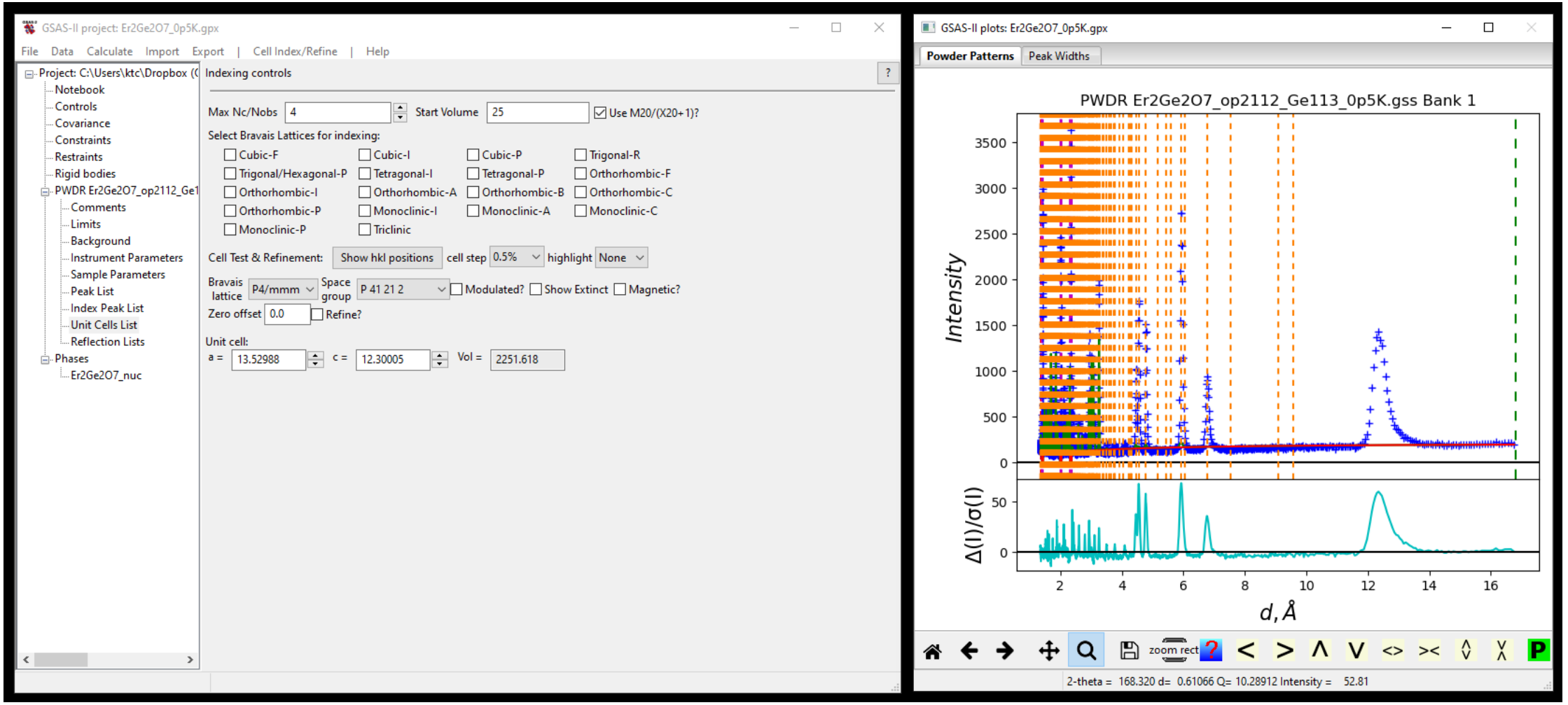
Let's zoom in on that large d-spacing peak. Its at 12.3 Å, very near the c lattice parameter as a 001 reflection. This would indicate a k=(000). We can test this:

Indexing the new peaks



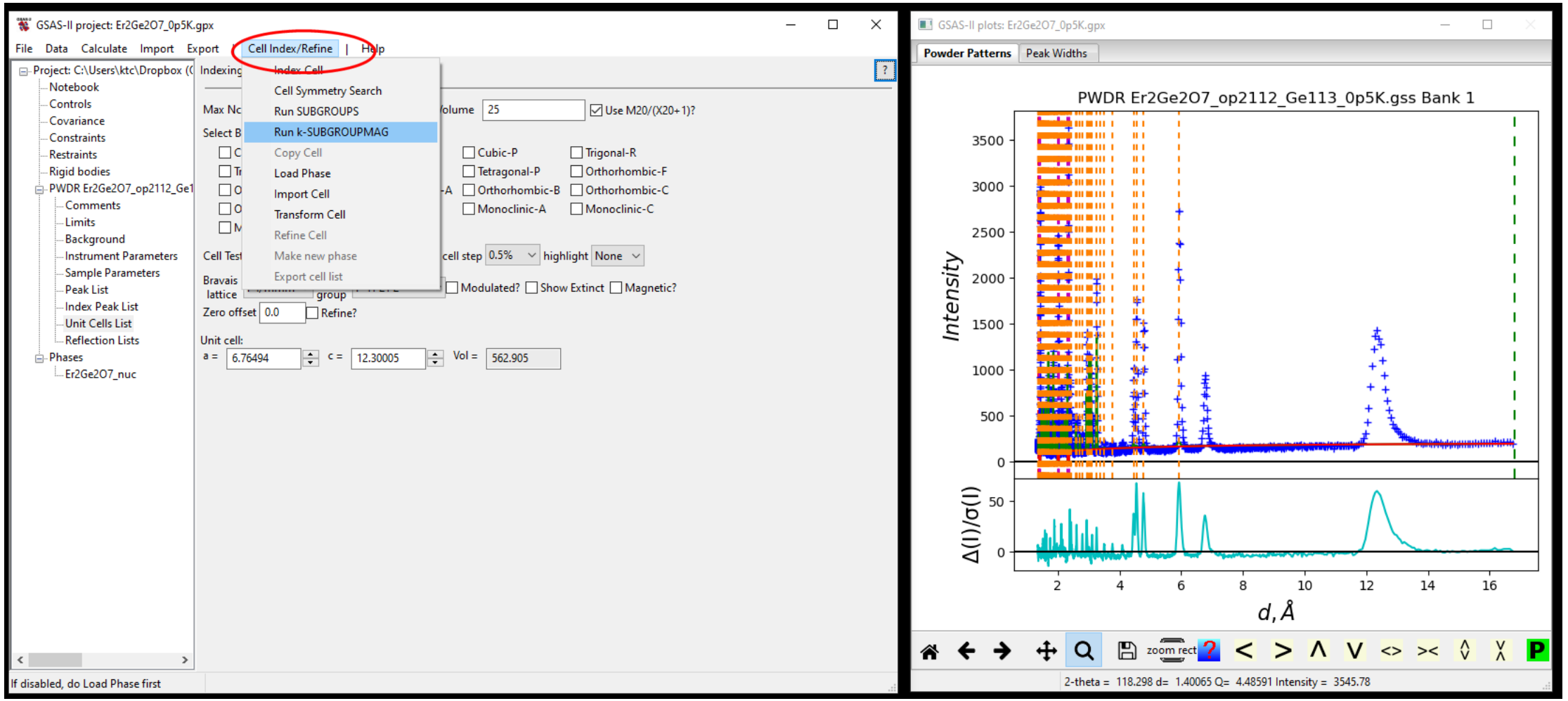
To do so, let's try playing with larger cells type in a doubled lattice parameter into any of the boxes in the Indexing controls window (or type '*2' in the box after the current lattice parameter.) I've tried doubling c above. It doesn't seem to help much – lets try 2*a.

Indexing the new peaks



Maybe gets some of the peaks, but it also adds peaks where we don't see any. Let's stick with k=000 for our first try
We could also try changing the space group and breaking some centering symmetries for example. But here since out largest d-spacing peak seems to correspond to our c lattice parameter we have a good lead

Solving the structure

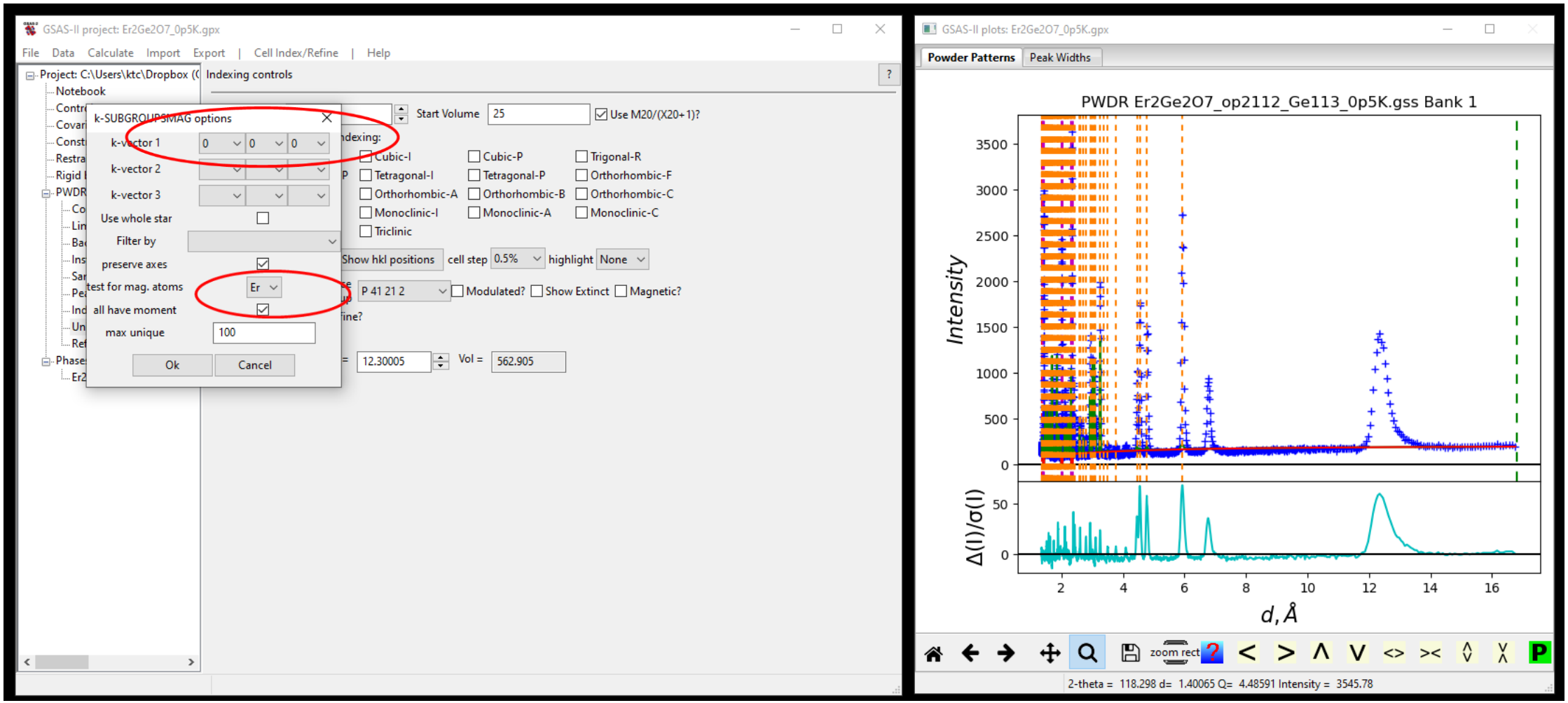


With a guess of the k-vector we can now use GSAS-II to link to the Bilbao server and run k-SUBGROUPMAG. This will provide a list of all the possible magnetic subgroups of our nuclear structure's space group given our found k-vector.

First, CHANGE THE UNIT CELL BACK TO THE ORIGINAL PARAMETERS!

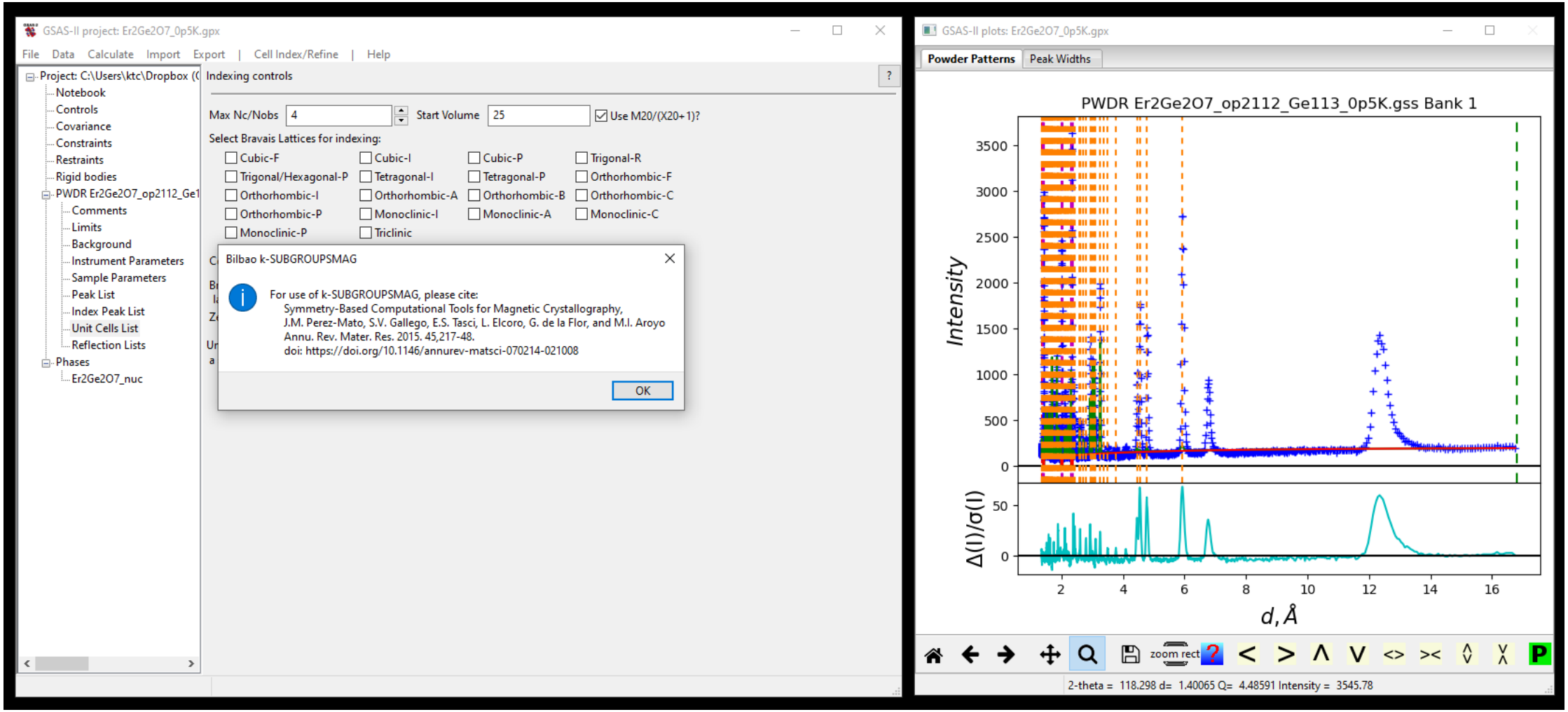
Then, Go to 'Cell Index/Refine' and click 'Run k-SUBGROUPMAG'

Solving the structure

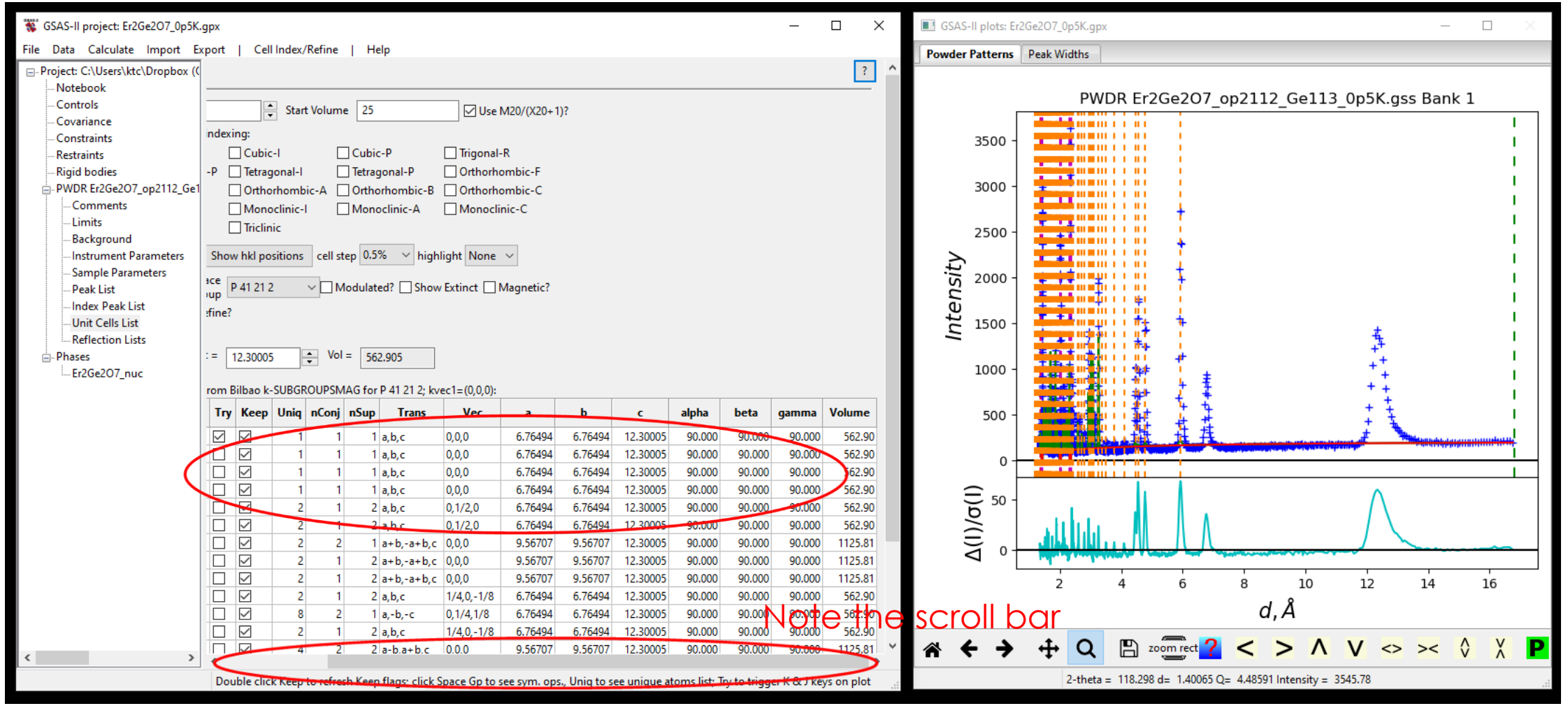


This should open a window allowing you to input one (or several) k-vectors and set some search criteria.
We'll put in our k-vector (0, 0, 0) and tell it to test for magnetic Er atoms.
Since we have no indication otherwise, let's start with structures that have moments on all Er.

Solving the structure

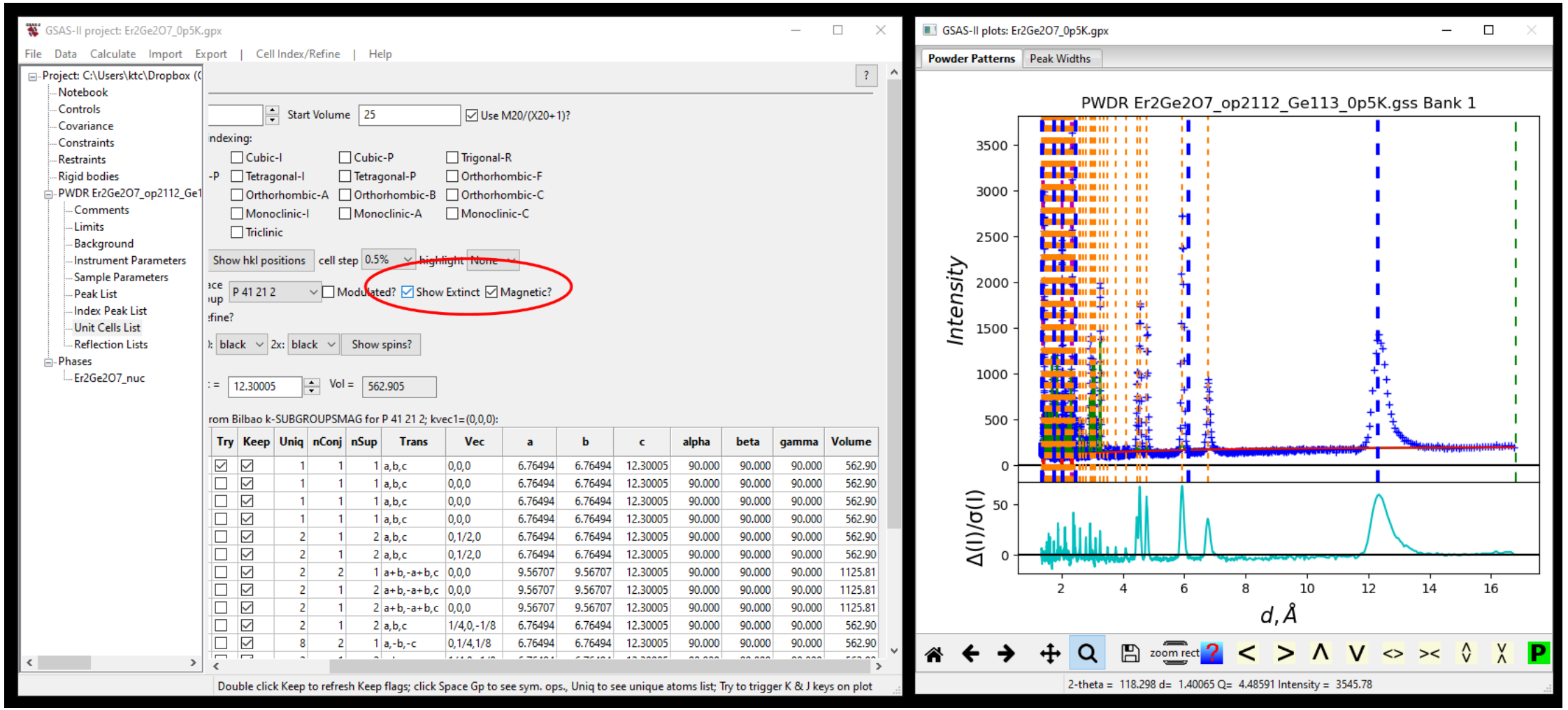


Solving the structure



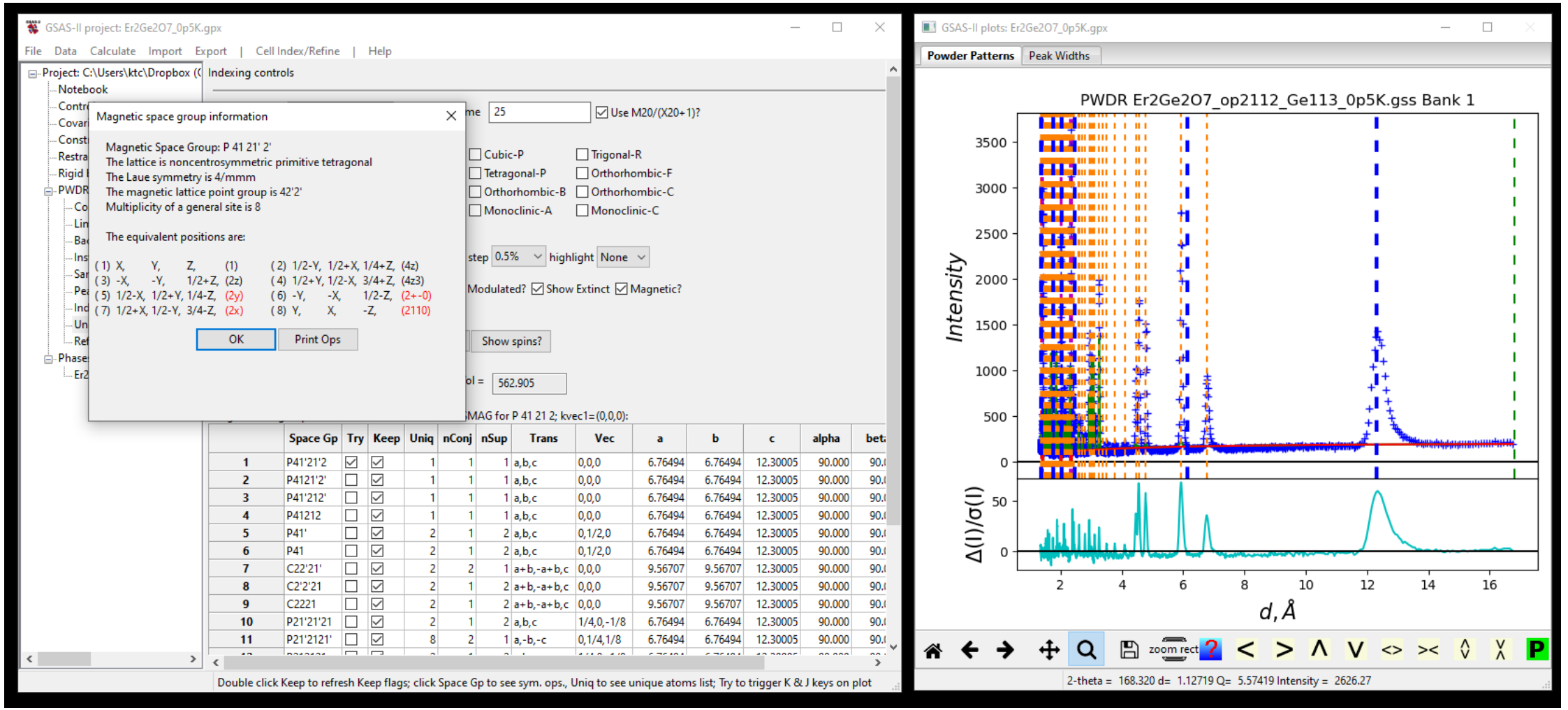
Some progress windows will show and then (hopefully) a series of possible magnetic subgroups should load complete with information on the magnetic space group, the transformation from the nuclear cell, etc. You can click through the 'Try' column to see how the different symmetries index the peaks. Here, first select 'show Extinct' and 'Magnetic'

Solving the structure



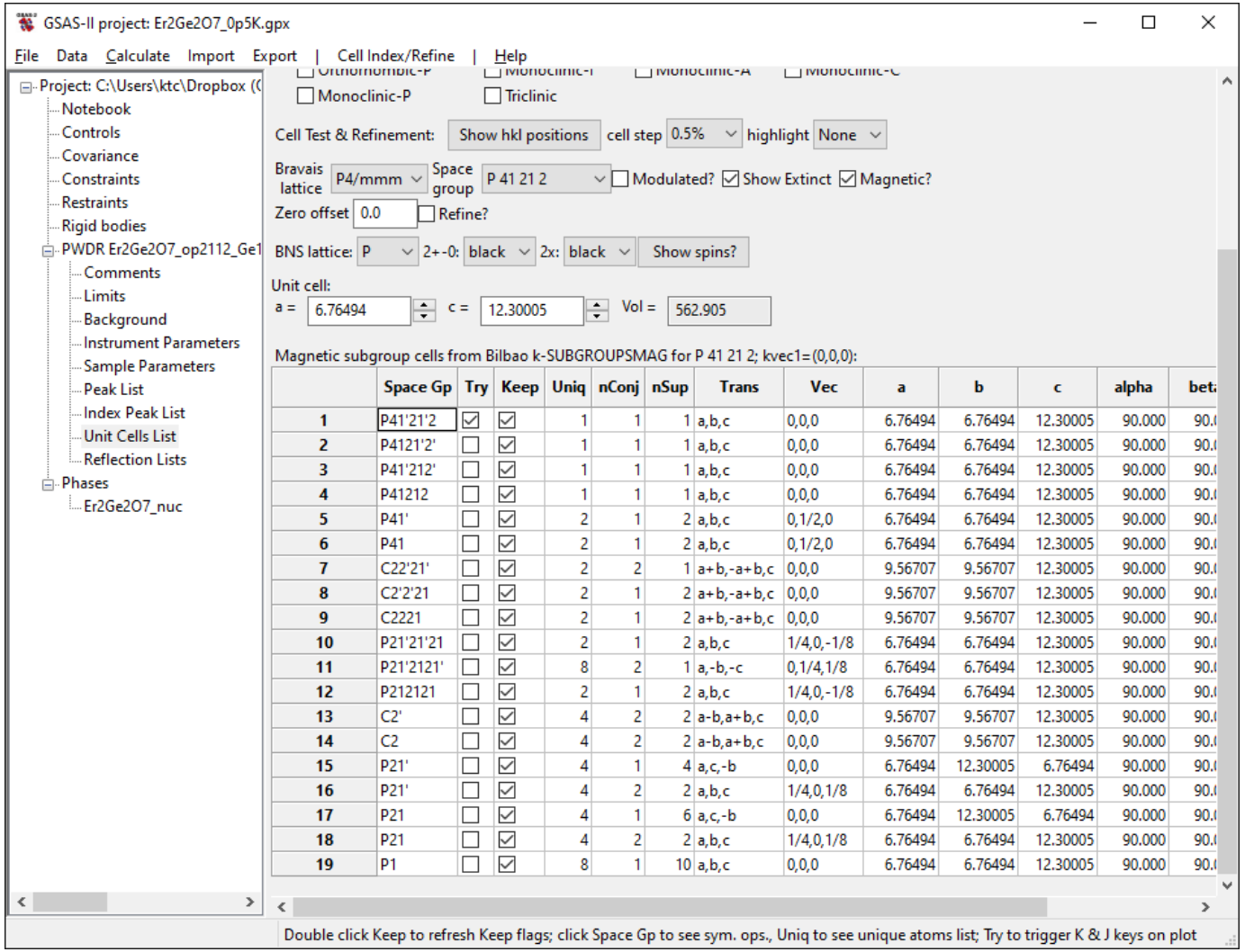
Now we see that all the peaks are indexed even for the first magnetic subgroup.
Note we select to show extinction here as a work around for the moment.

Solving the structure



You can also click on the space group to get a table of the symmetry operations

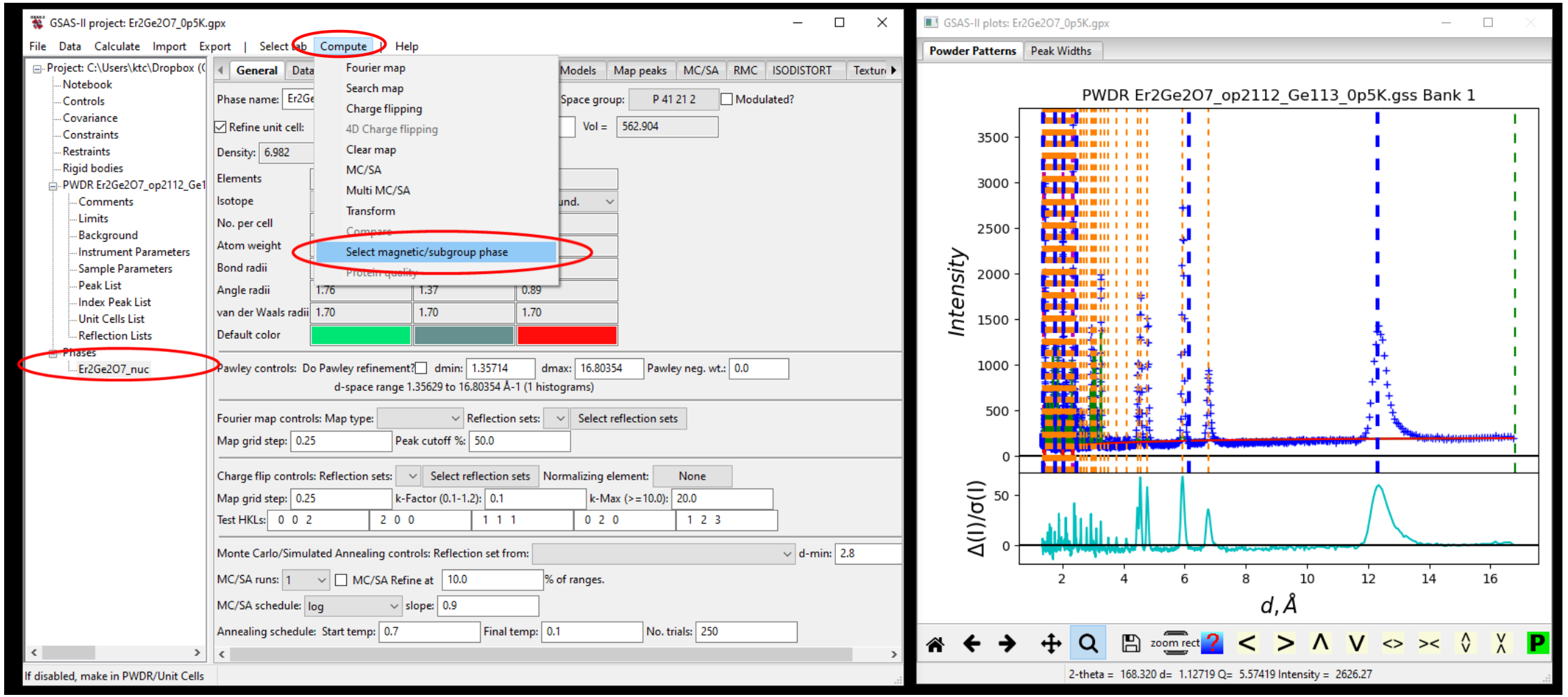
Solving the structure



	Space Gp	Try	Keep	Uniq	nConj	nSup	Trans	Vec	a	b	c	alpha	bet
1	P41'21'2	☑	☑	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.
2	P4121'2'	☐	☑	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.
3	P41'212'	☐	☑	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.
4	P41212	☐	☑	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.
5	P41'	☐	☑	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.
6	P41	☐	☑	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.
7	C22'21'	☐	☑	2	2	1	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.
8	C2'2'21	☐	☑	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.
9	C2221	☐	☑	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.
10	P21'21'21	☐	☑	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.
11	P21'2121'	☐	☑	8	2	1	a,-b,-c	0,1/4,1/8	6.76494	6.76494	12.30005	90.000	90.
12	P212121	☐	☑	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.
13	C2'	☐	☑	4	2	2	a-b,a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.
14	C2	☐	☑	4	2	2	a-b,a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.
15	P21'	☐	☑	4	1	4	a,c,-b	0,0,0	6.76494	12.30005	6.76494	90.000	90.
16	P21'	☐	☑	4	2	2	a,b,c	1/4,0,1/8	6.76494	6.76494	12.30005	90.000	90.
17	P21	☐	☑	4	1	6	a,c,-b	0,0,0	6.76494	12.30005	6.76494	90.000	90.
18	P21	☐	☑	4	2	2	a,b,c	1/4,0,1/8	6.76494	6.76494	12.30005	90.000	90.
19	P1	☐	☑	8	1	10	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.

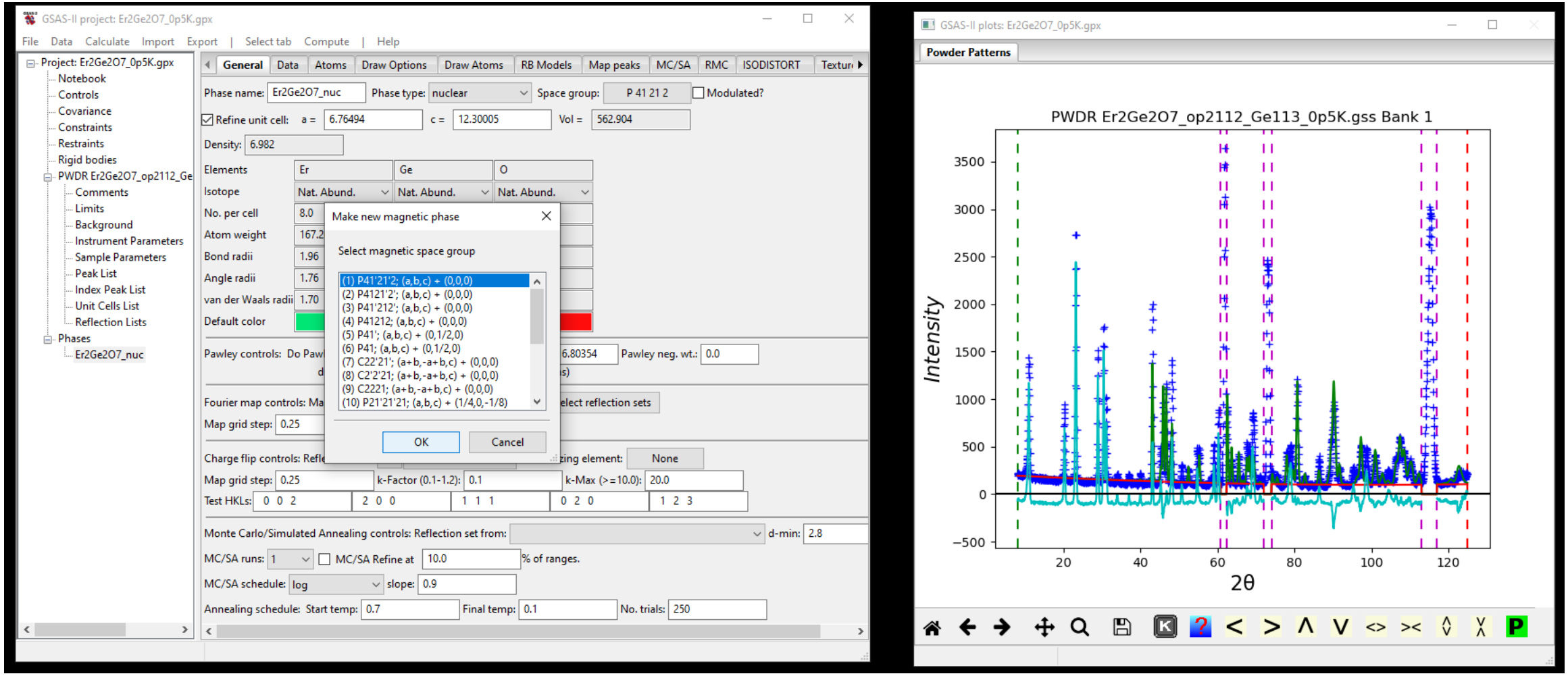
If you scroll around the table you can see the symmetry gets lower the lower down you go. The first four magnetic space groups keep the nuclear structure and just 'prime' its operations for the magnetic structure. Just playing around it looks like these would be good to try

Solving the structure



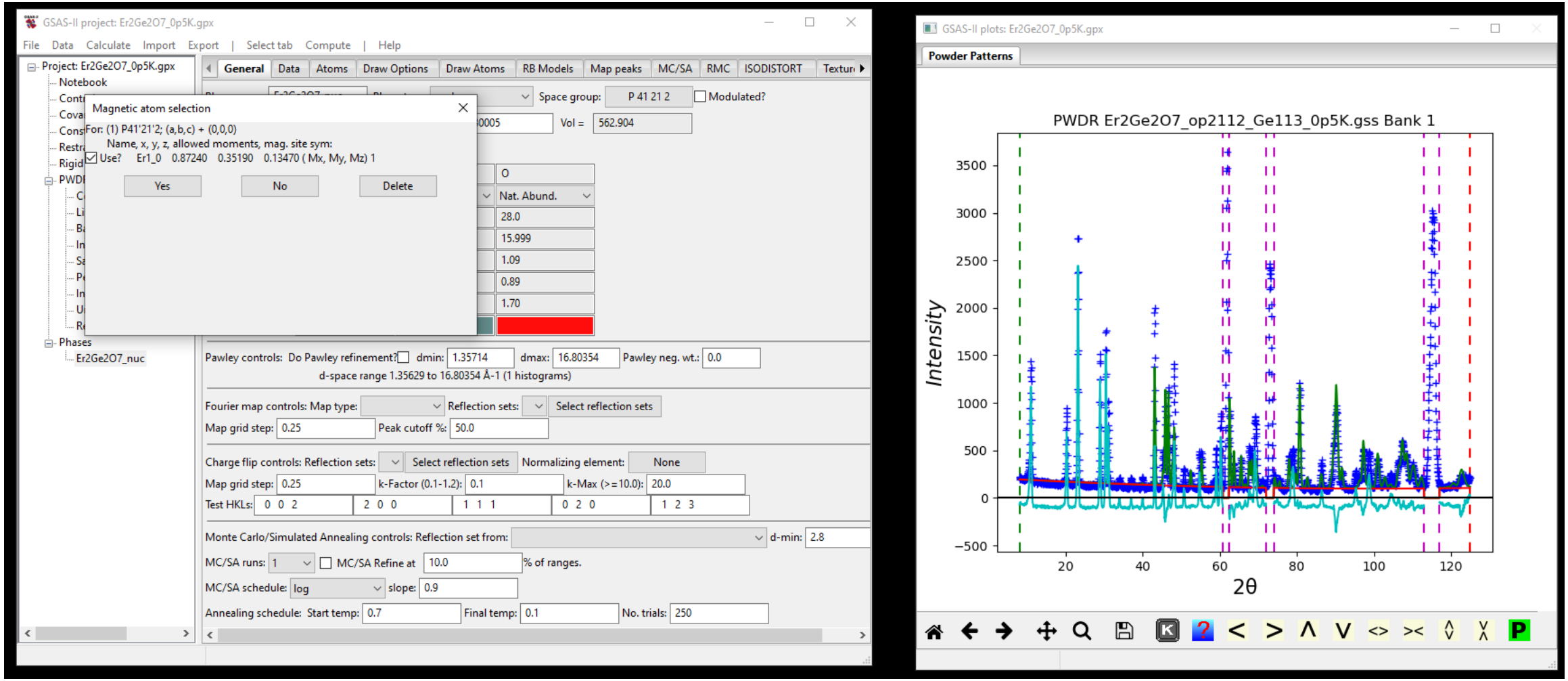
Now to actually try some of these out in the refinement we'll go back to the Er2Ge2O7_nuc Node under phases and click on Compute -> Select magnetic/subgroup phase (You may want to save a backup of this working nuclear refinement at this point!)

Solving the structure



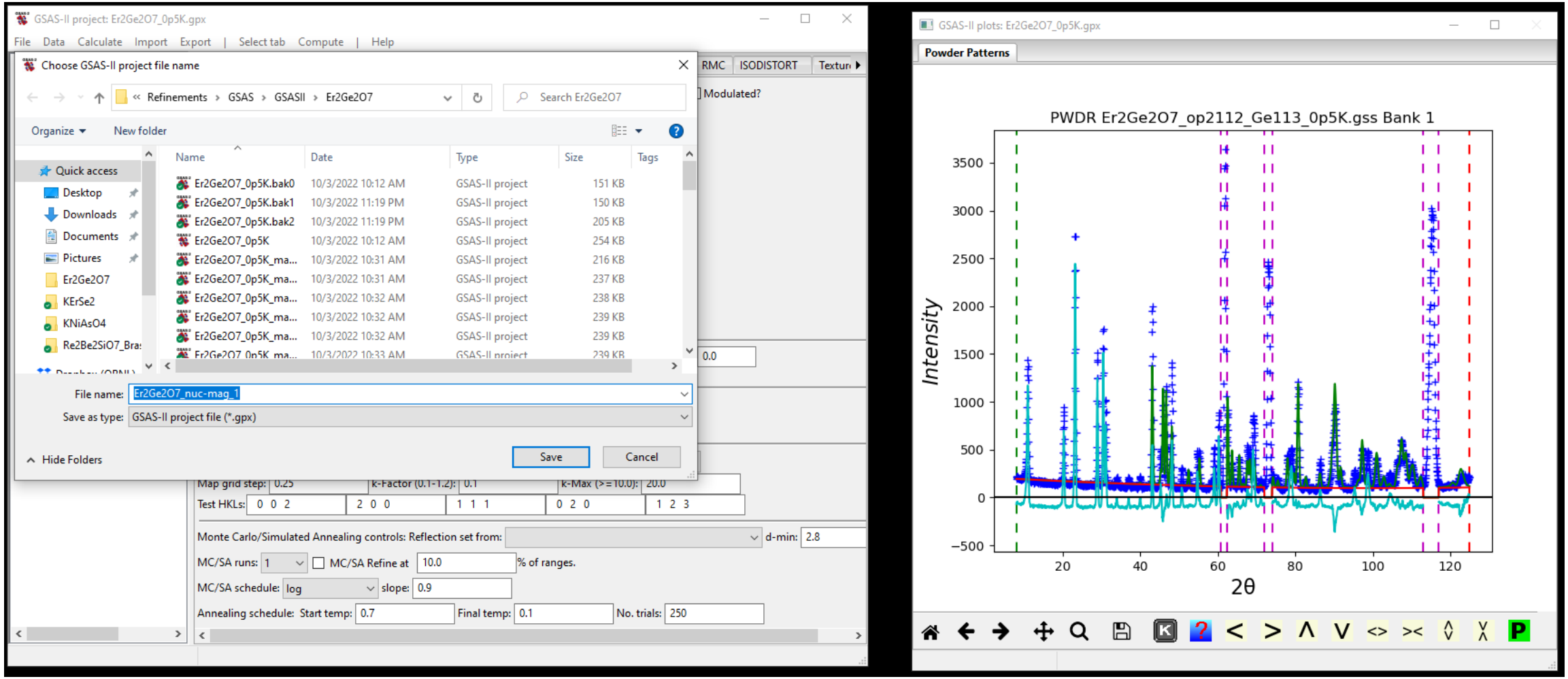
Let's try these one by one
Starting with the first option

Solving the structure



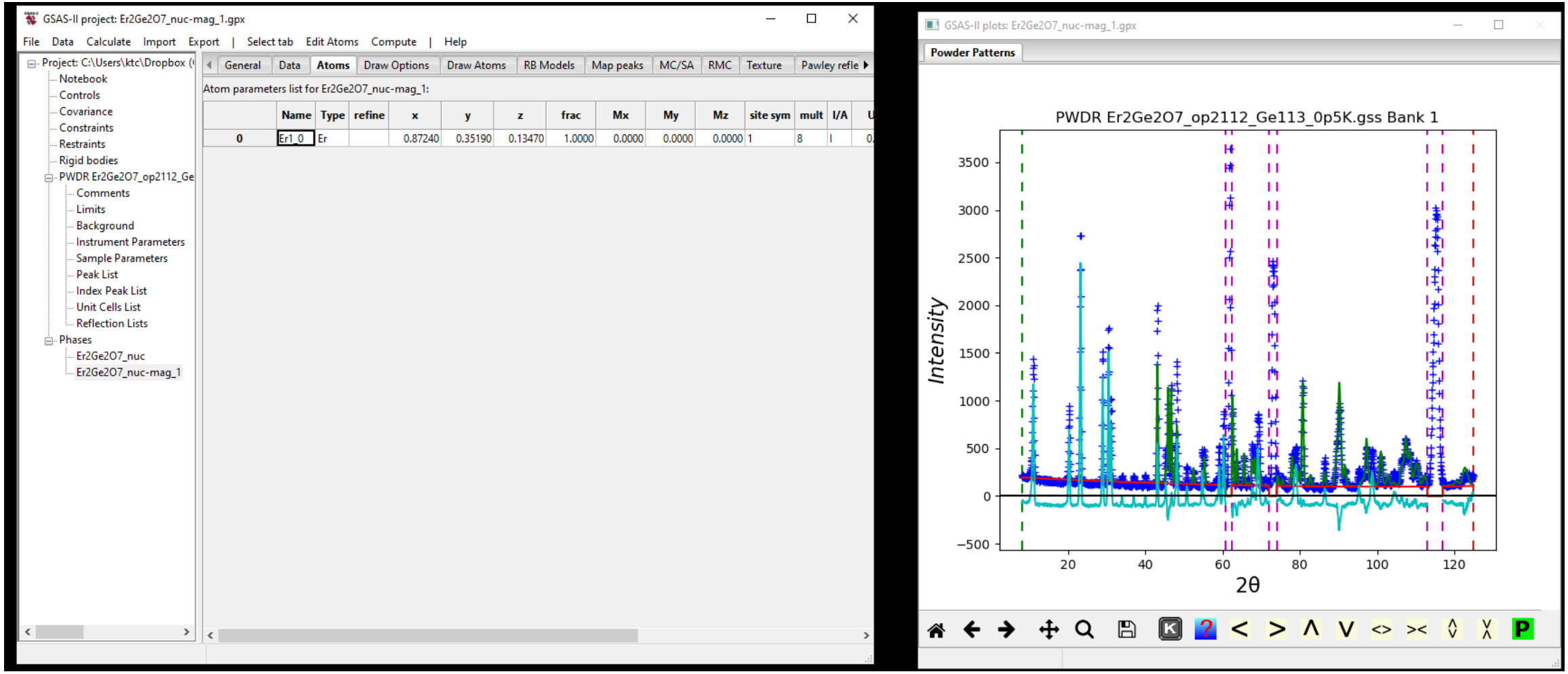
A window will pop-up with the magnetic ions. There is only one symmetry distinct site in this case.
Click Yes.

Solving the structure



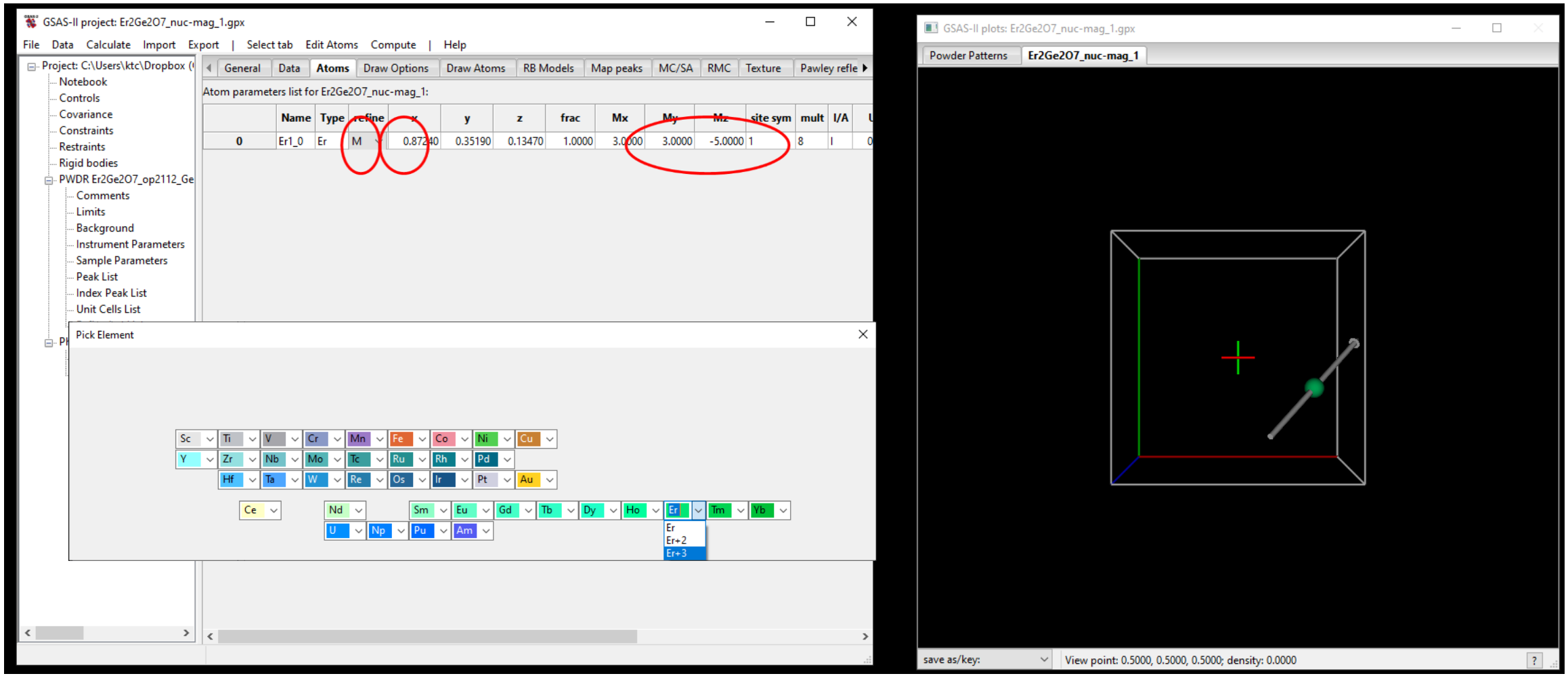
You should get a window prompting you to save the gpx under a new name. This gives you a good branch to try and then go back to the original to try a different structure

Solving the structure



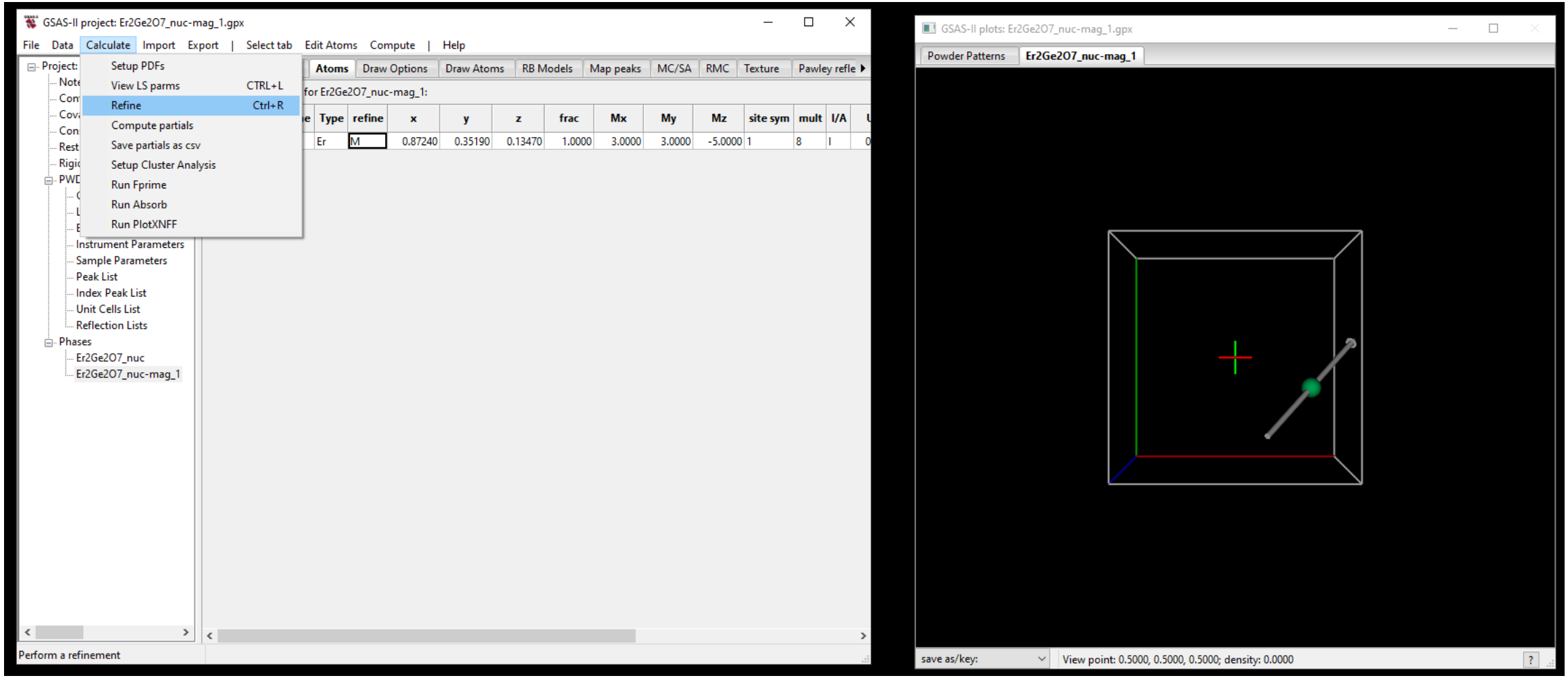
In the tree you will now see a second, magnetic, phase. If you go to the Atoms tab you'll find only 1 atom – the single Er site – now with new columns for the magnetic moment components. In this symmetry the magnetic moments are allowed to have moment components along all three crystallographic directions.

Solving the structure: refining the moments



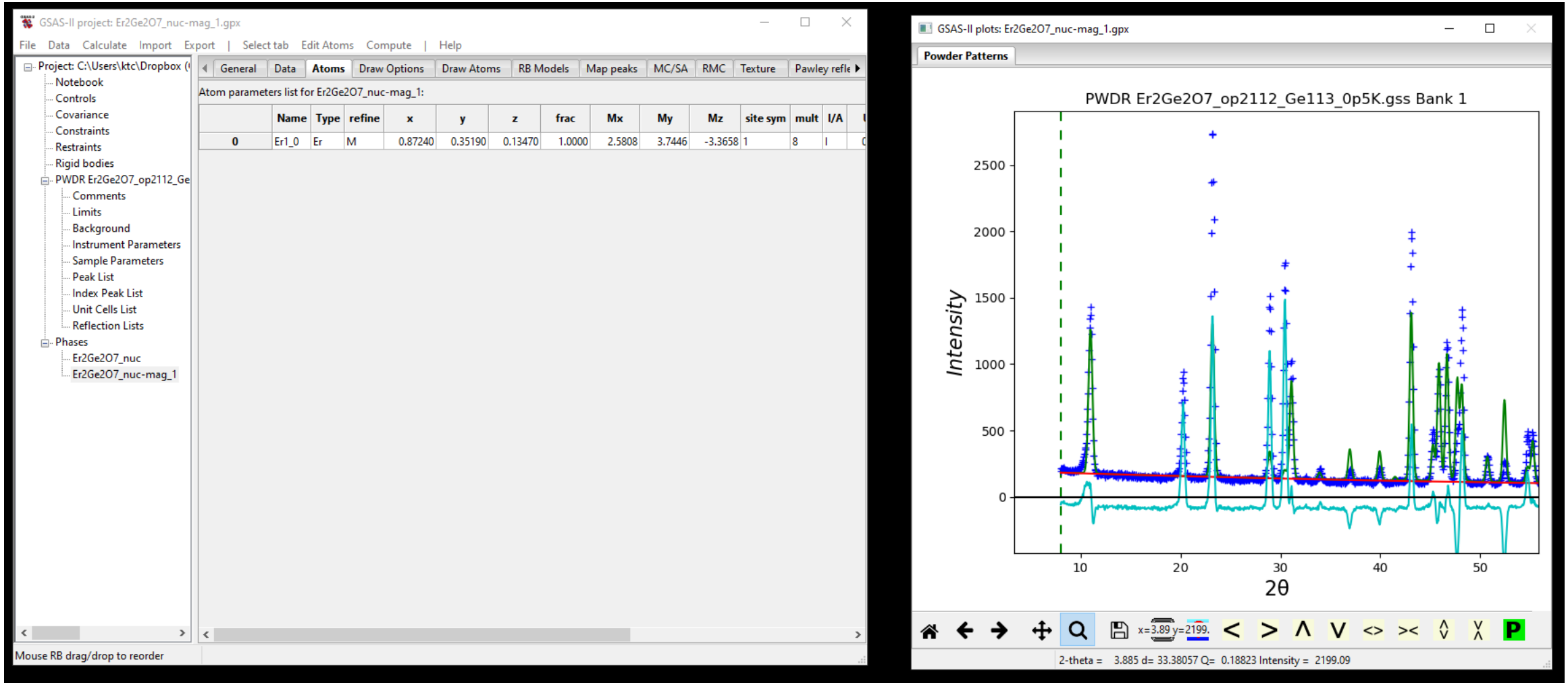
Let's flag these for refinement (set the refinement column to M), give them nominal starting values (I'll do Mx, My, Mz = 3,3,-5) and see how it goes we can motivate these choices from comparing the relatively intensity of the 001 peak to HK0 peaks.
Also, we should change the atom type to reflect the valence – Er^{3+}

Solving the structure: refining the moments



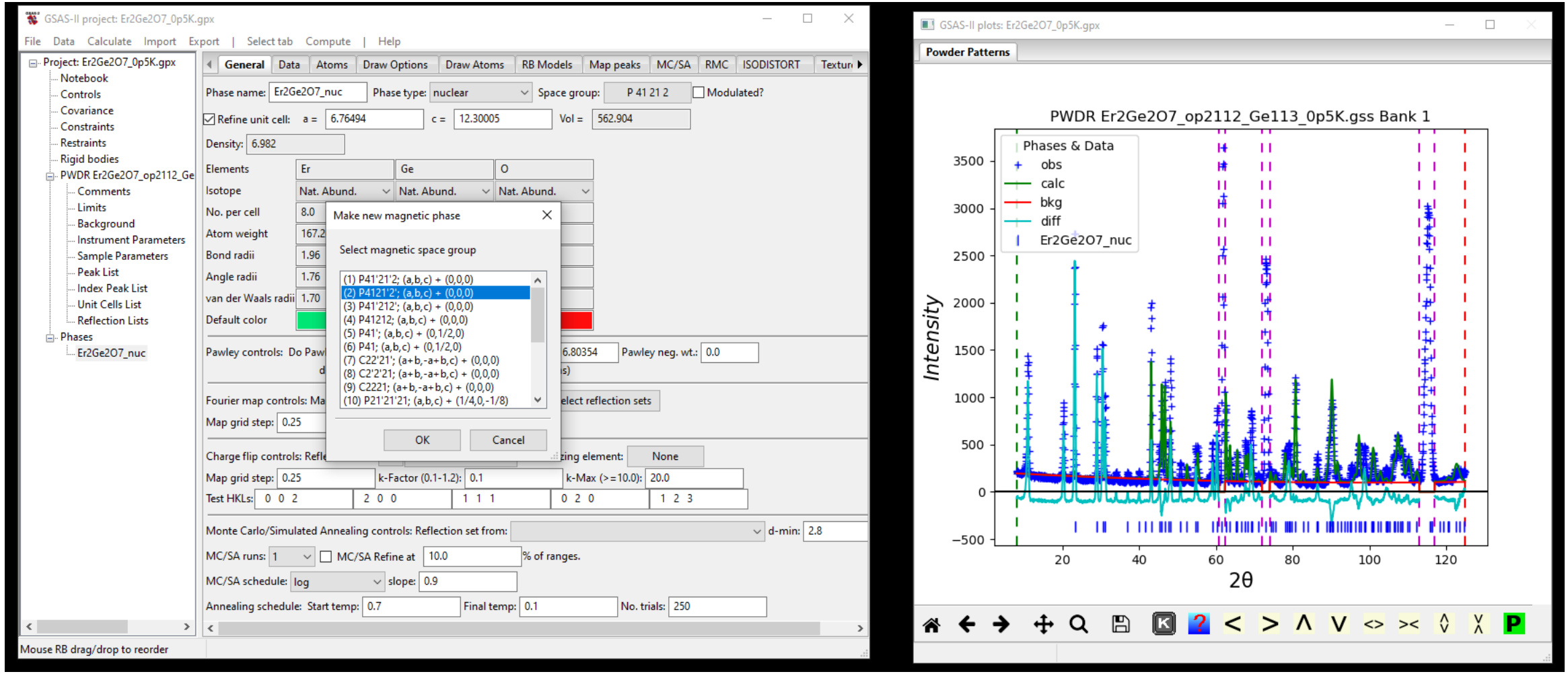
Let's try a refinement

Solving the structure: refining the moments



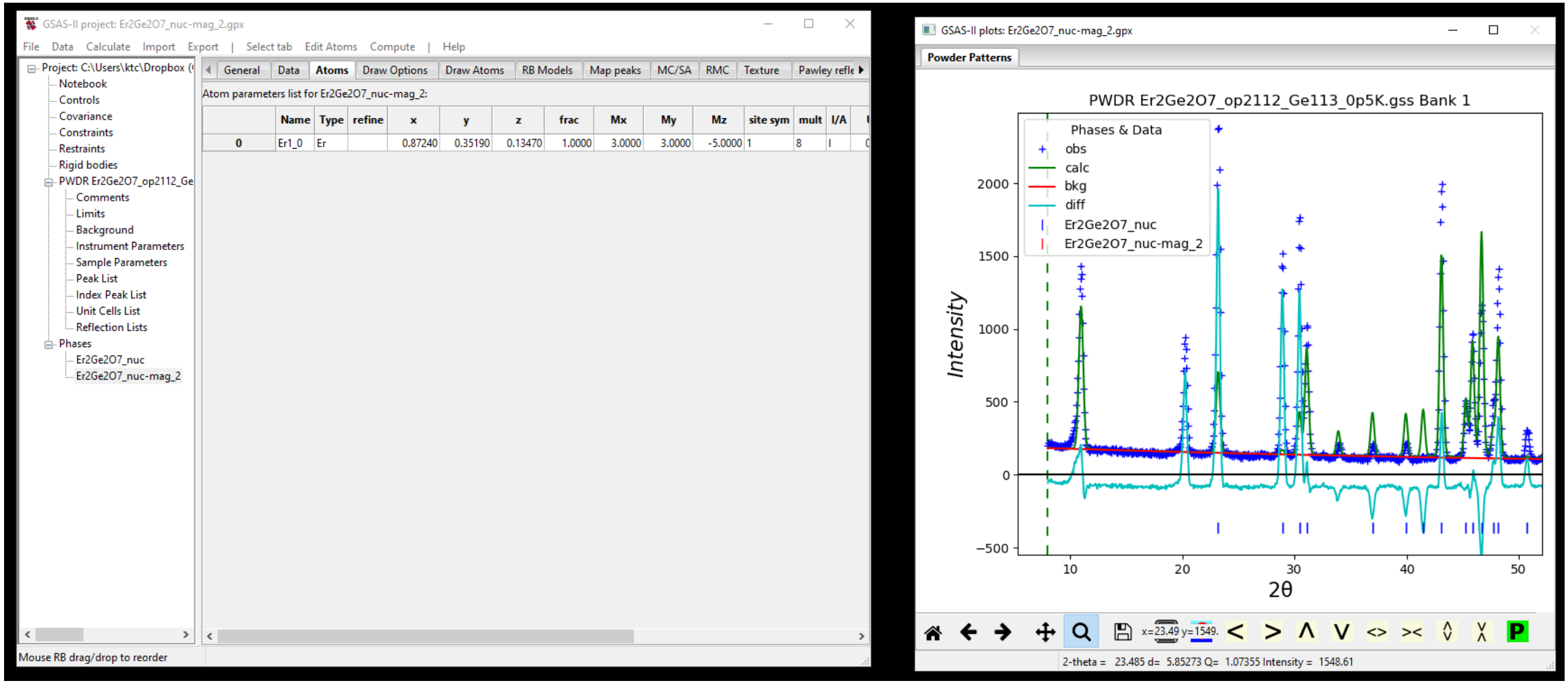
This doesn't look so great, intensity is off but we are also completely missing some peaks.
Let's try the second structure

Solving the structure: iterating through the structures



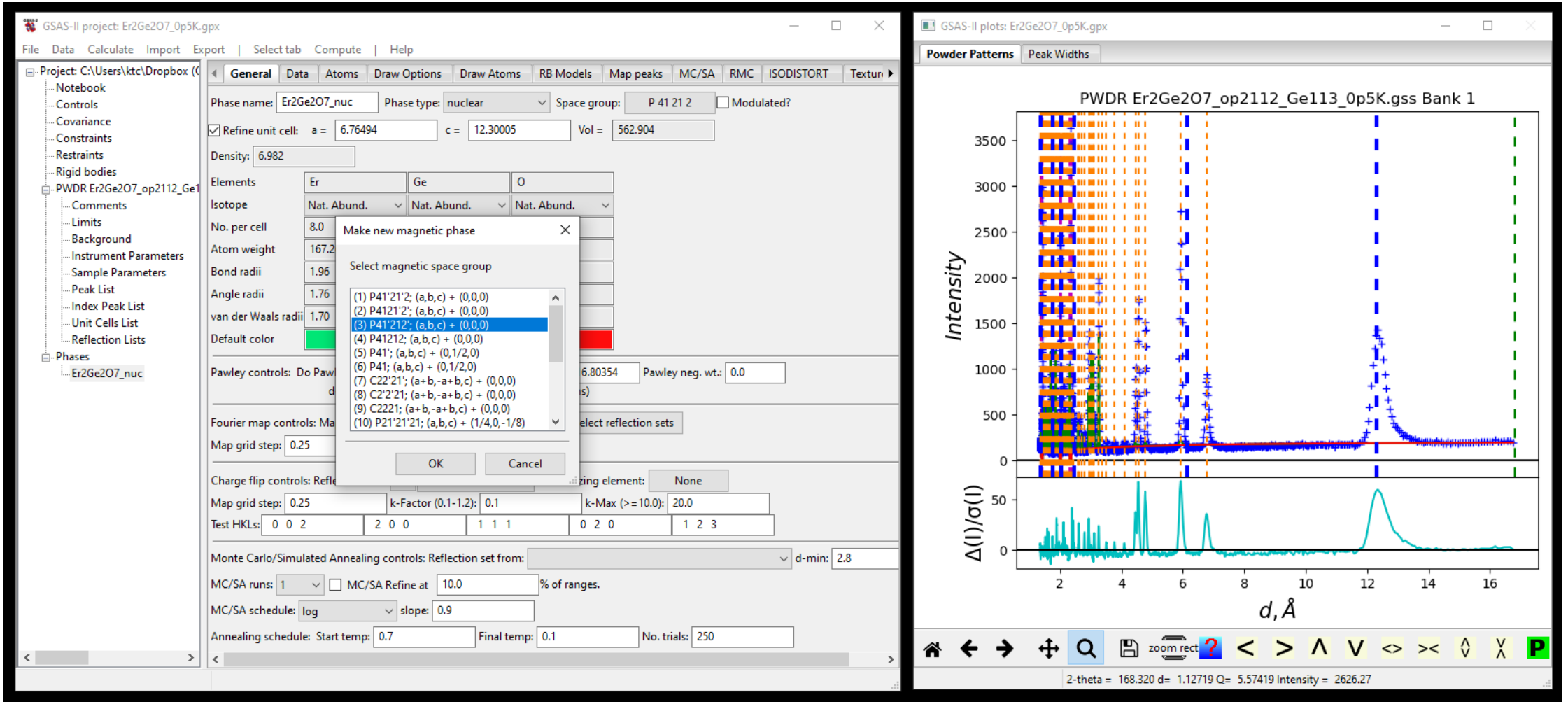
Reopen your 0.5 K fit and select the second structure and redo the last couple steps

Solving the structure: iterating through the structures



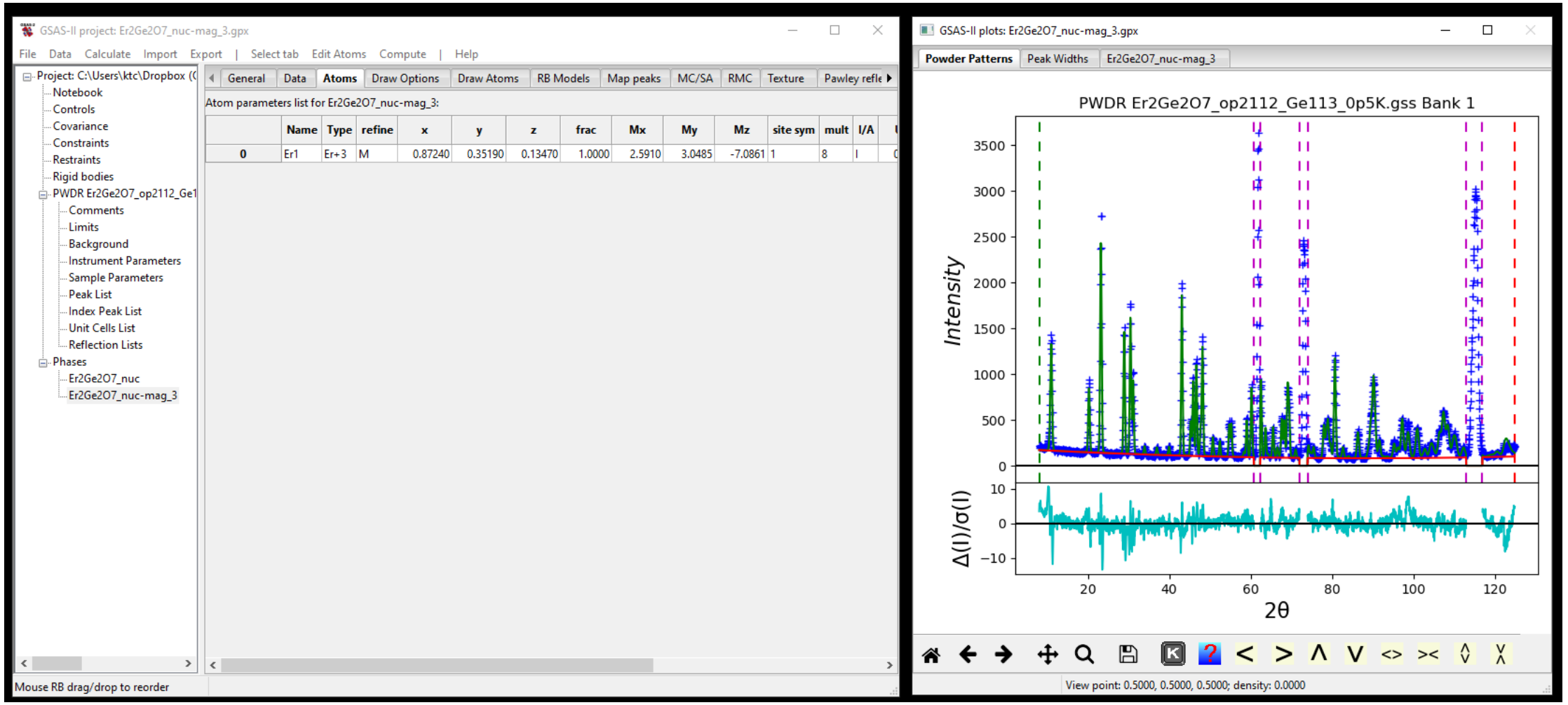
Also not even close, let's try the third.

Solving the structure



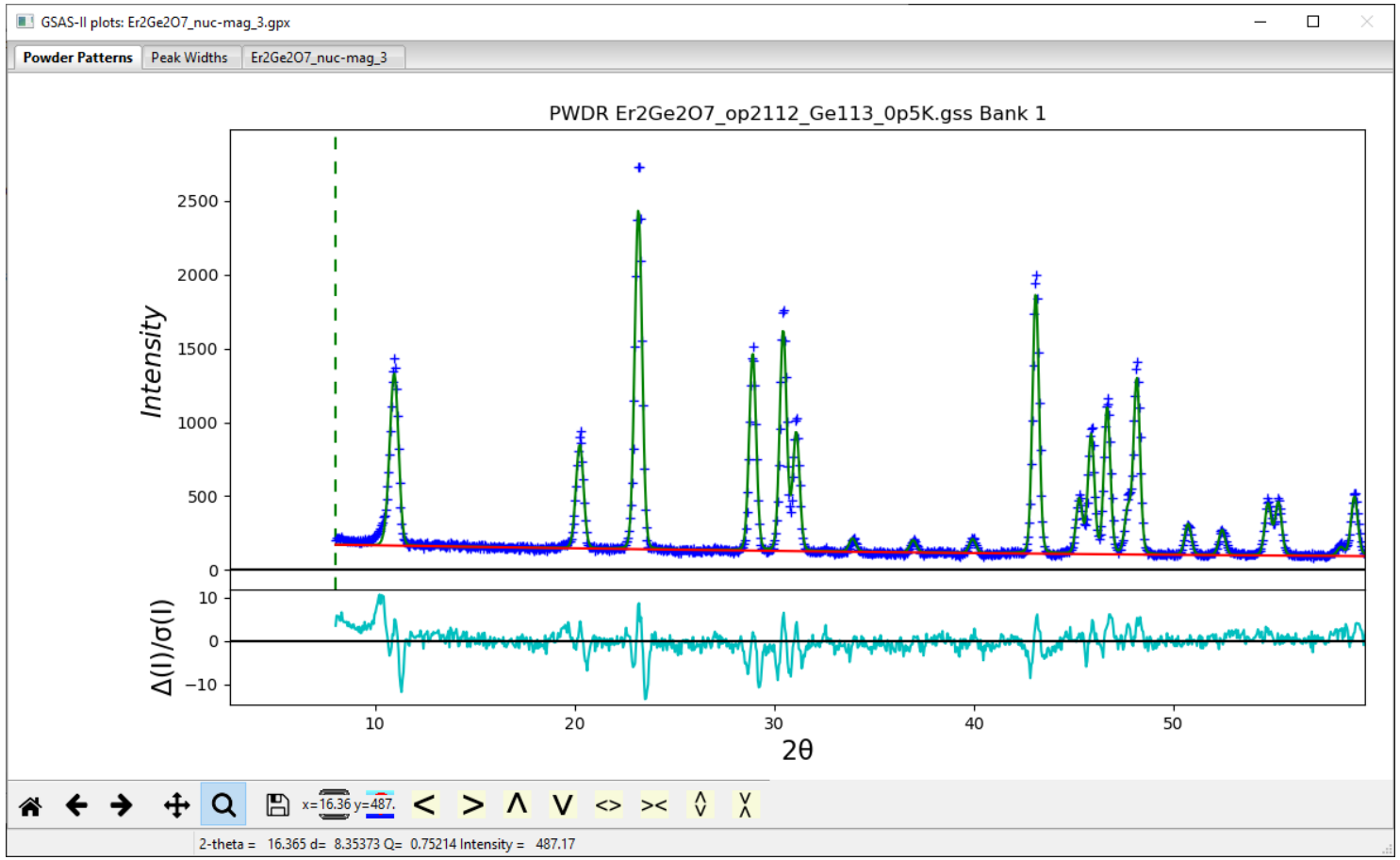
Go back to your 0.5 K save and try the third structure

Solving the structure: refining the moments



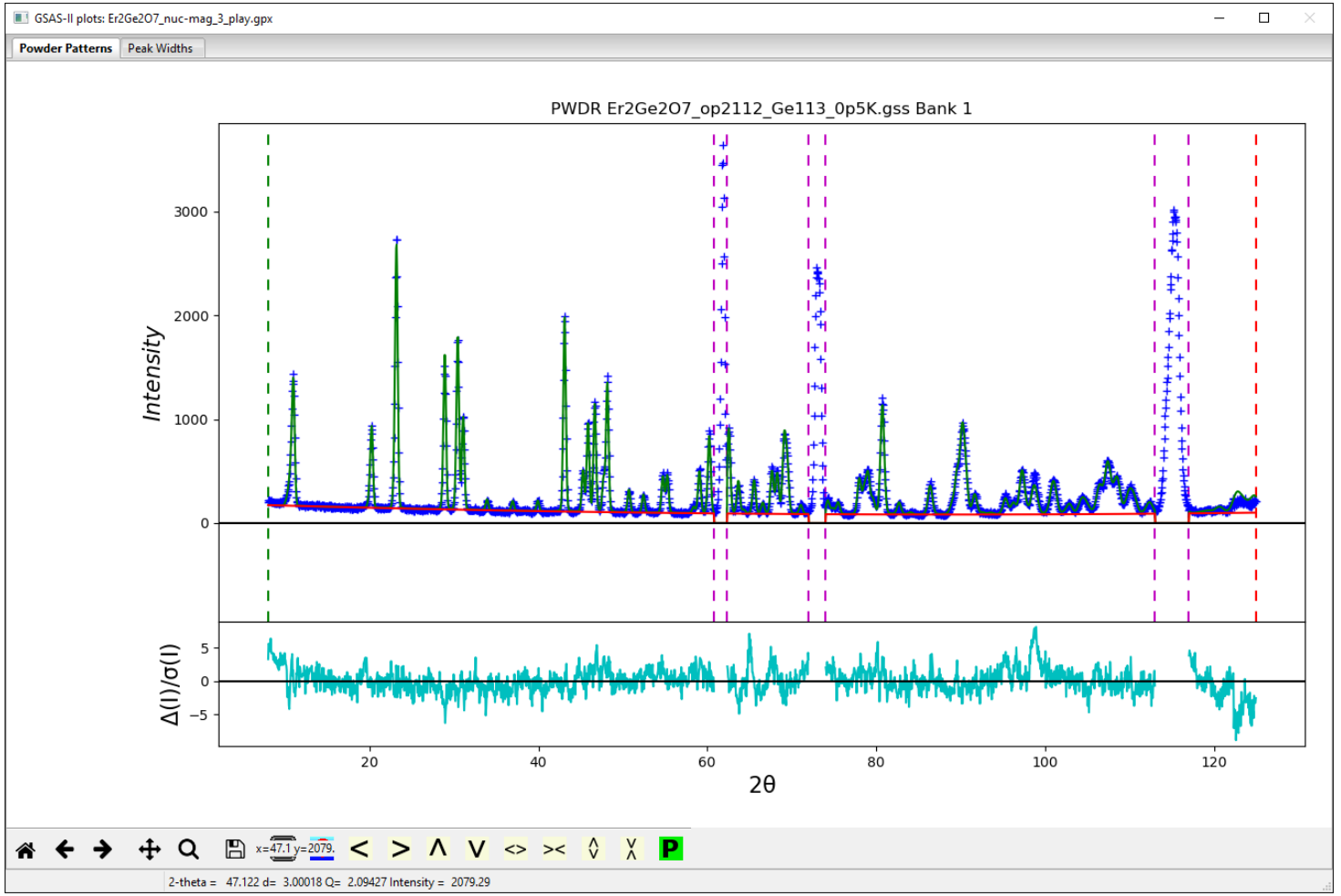
This one looks quite good

Solving the structure: checking the fit



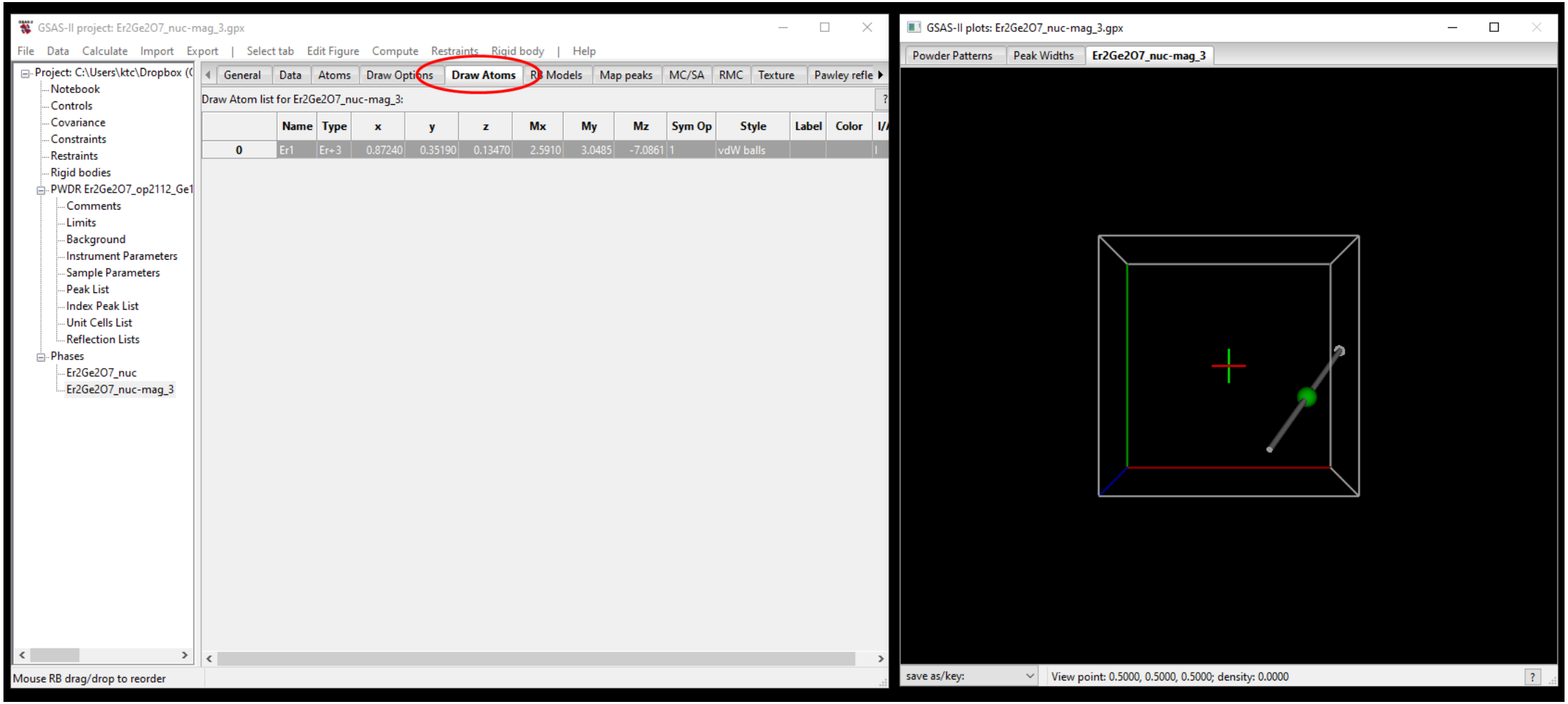
If we zoom in on the magnetic peaks, we see agreement in the positions and intensities. This might be a possible solution (we should try the final P41212 based structure as well)
I wouldn't recommend trying the lower symmetry models if you get a good fit with a higher symmetry model. Only if you see something indicating you need further symmetry breaking.

Solving the structure: checking the fit



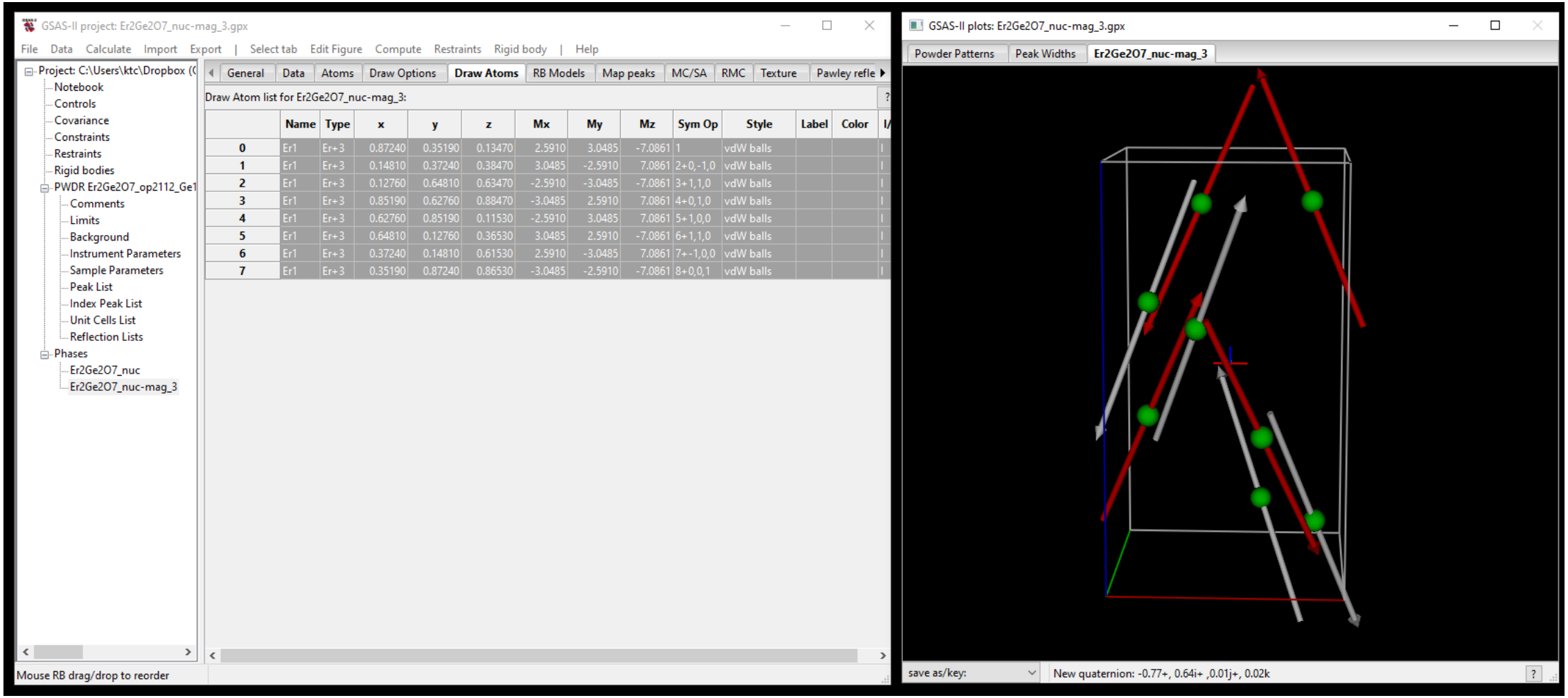
Fitting a little more carefully I can get a fit that looks like this – quite nice!

Visualizing the magnetic structure



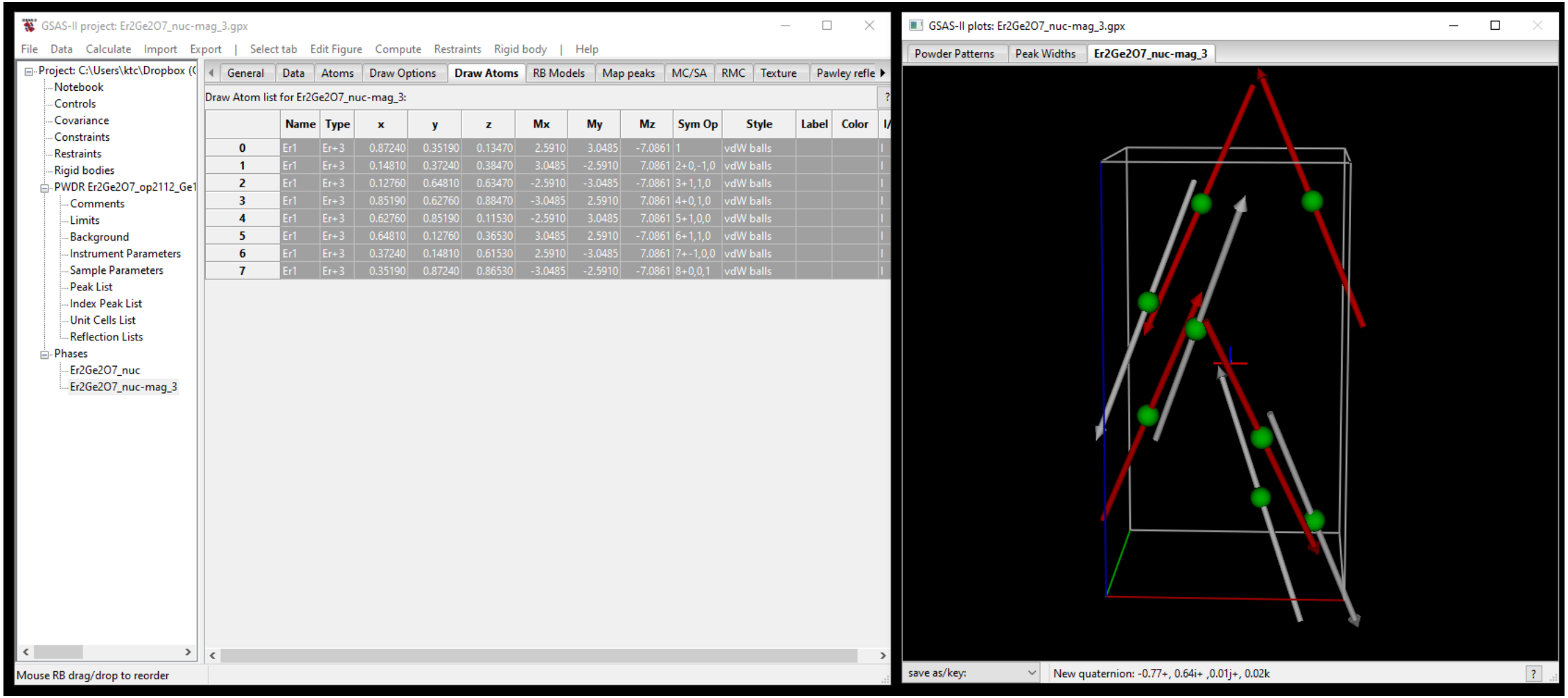
To see what this looks like click on the 'Draw Atoms' tab for the magnetic phase. Then use the same trick as before to show all the atoms

Visualizing the magnetic structure



That helps!

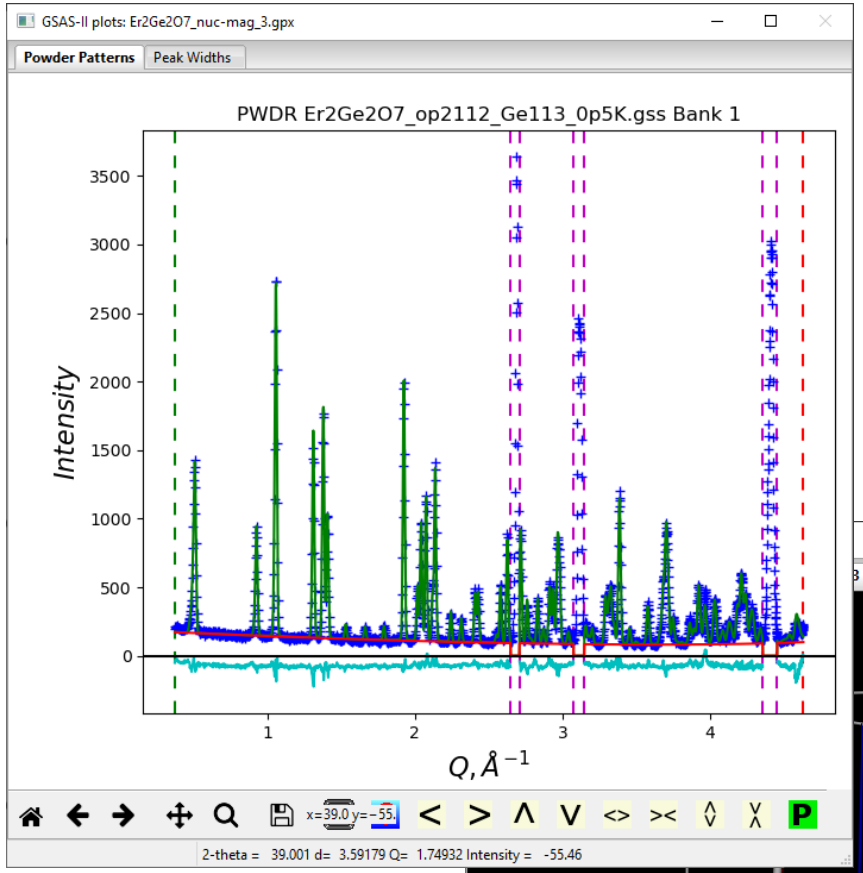
Visualizing the magnetic structure

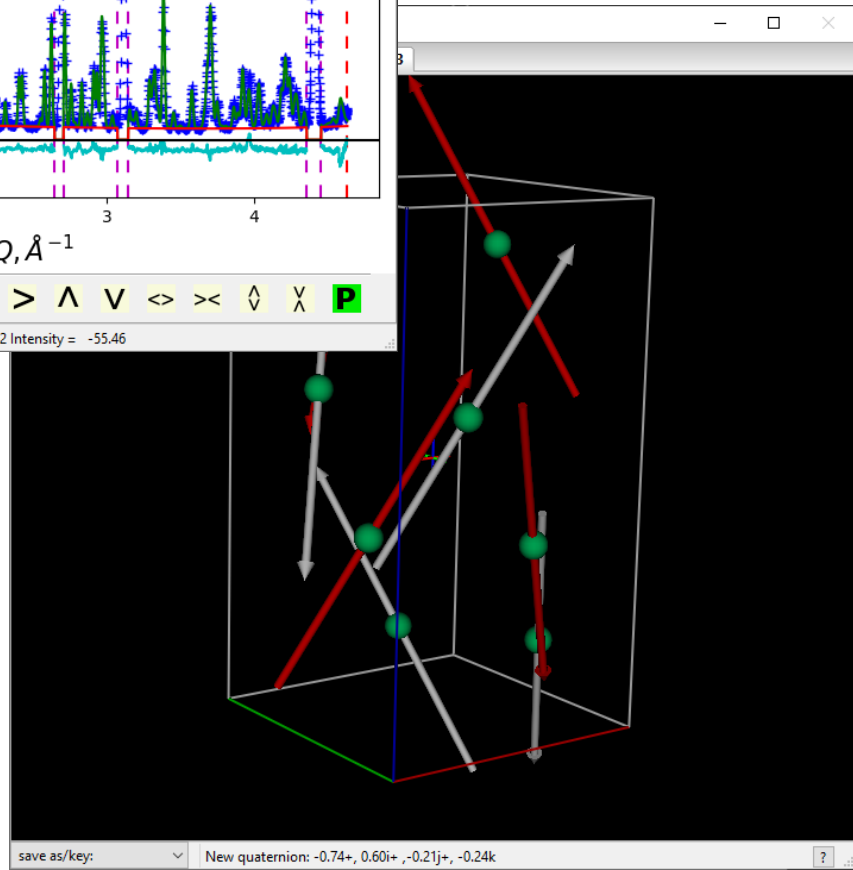


Now the structure looks to have the reported 'local Ising' behavior.
We should next go through and kick the moments a bit – see if they go back to these values. Is this a unique solution? Do the other models work? Better? But for now, we've shown how to get started at least!

Concluding thoughts

- From here (after checking that fourth structure)we can export a nice plot of the fit, check the moment size, try pushing the structure to see if it returns to this solution.
- As you can see, GSASII's linking to k-subgroupmag is very nice and makes trying solutions very convenient!
- Always good to check your structure solution using multiple methods





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