

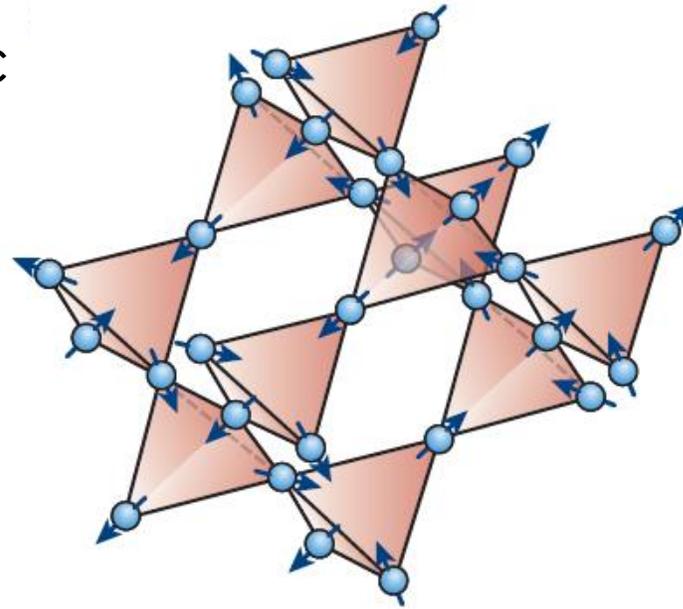
Using CW data and GSASII to solve magnetic structures (HB-2A data)

Keith M. Taddei

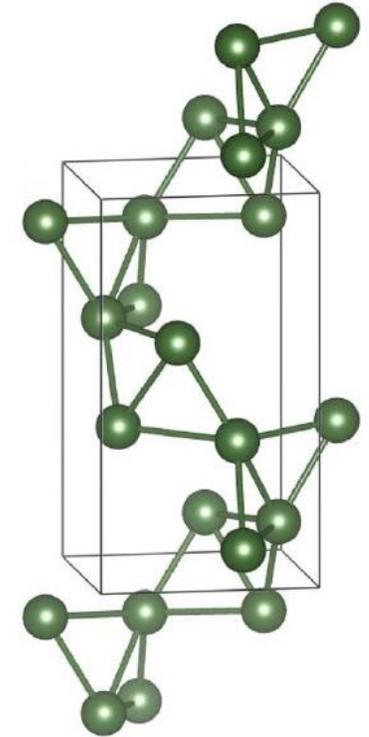
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Starting point – study frustration in pyrogermanates

- 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

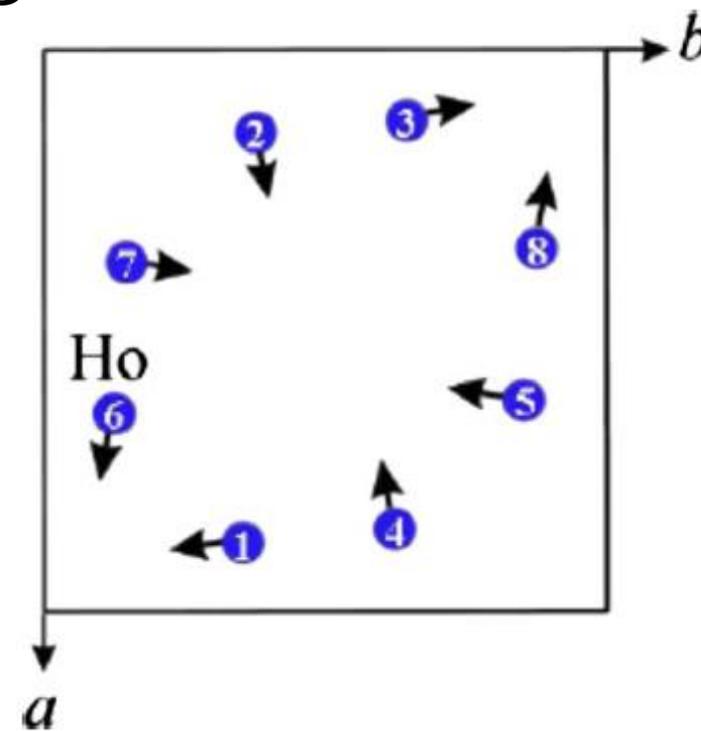
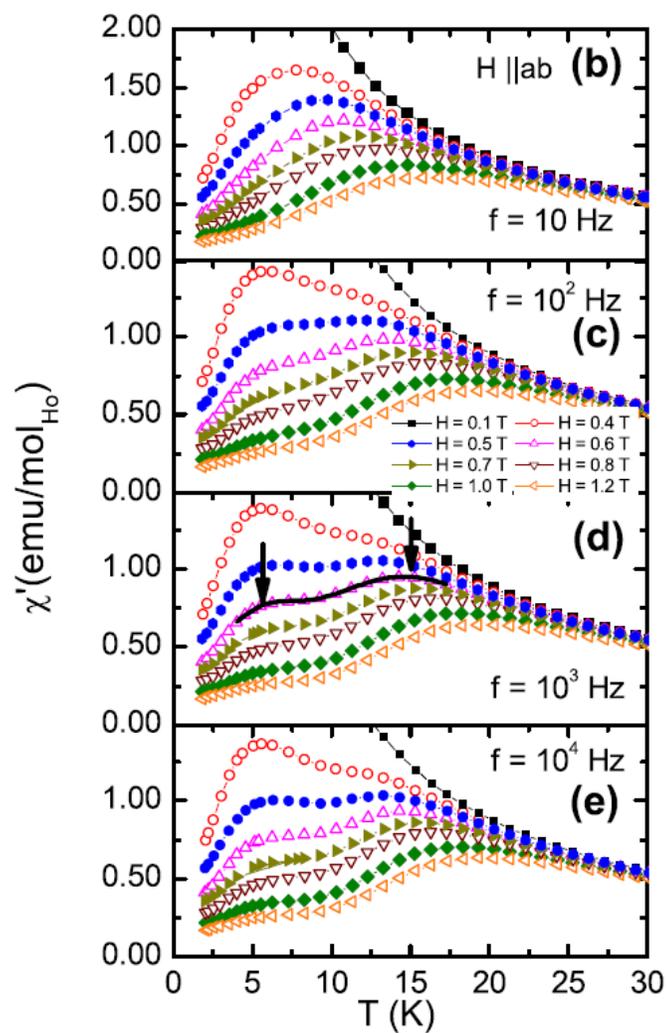
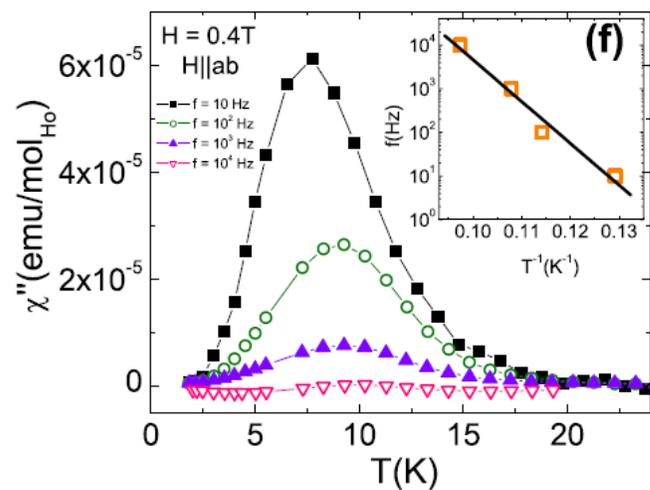
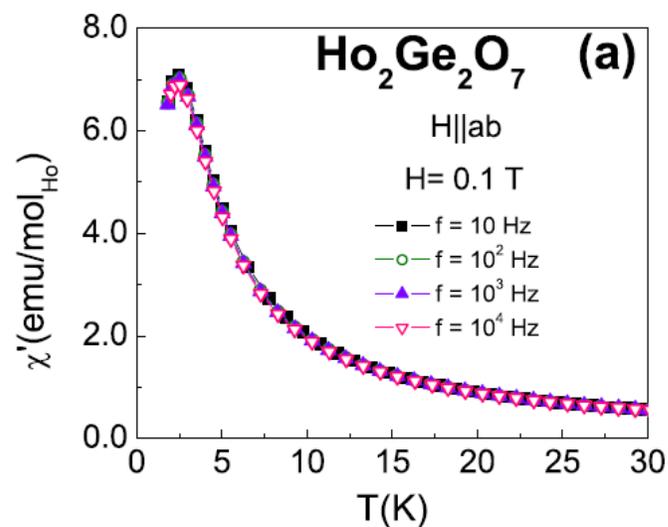


Pyrochlore Lattice:
 $RE_2M_2O_7$
Corner sharing *RE* tetrahedra –
geometric frustration



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

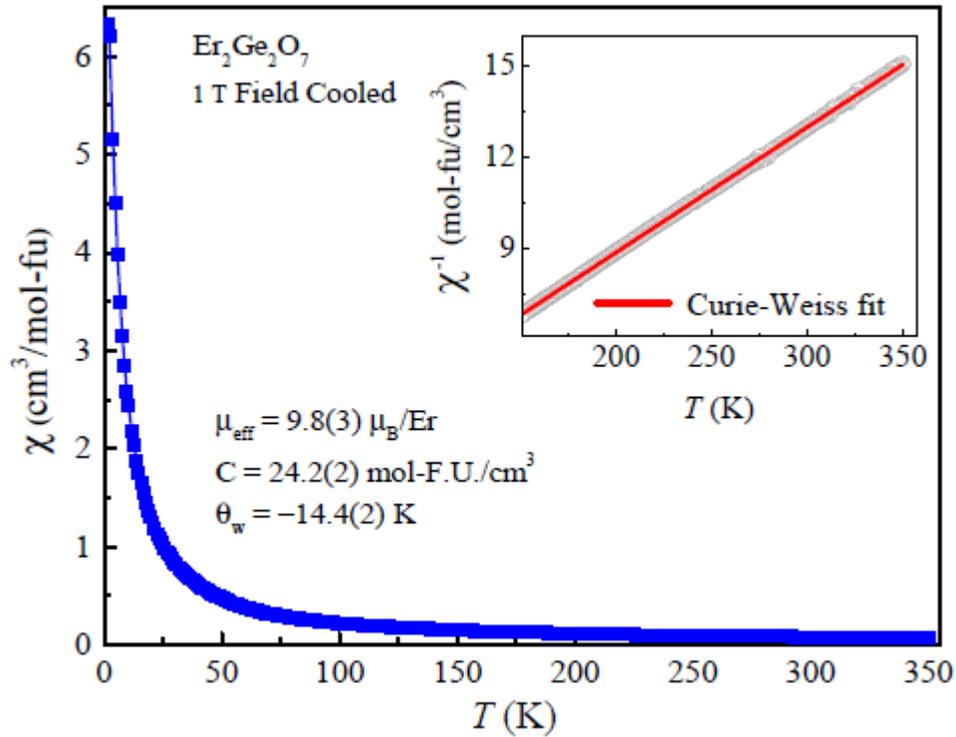
Evidence of frustration in Ho and Dy pyrogermanates



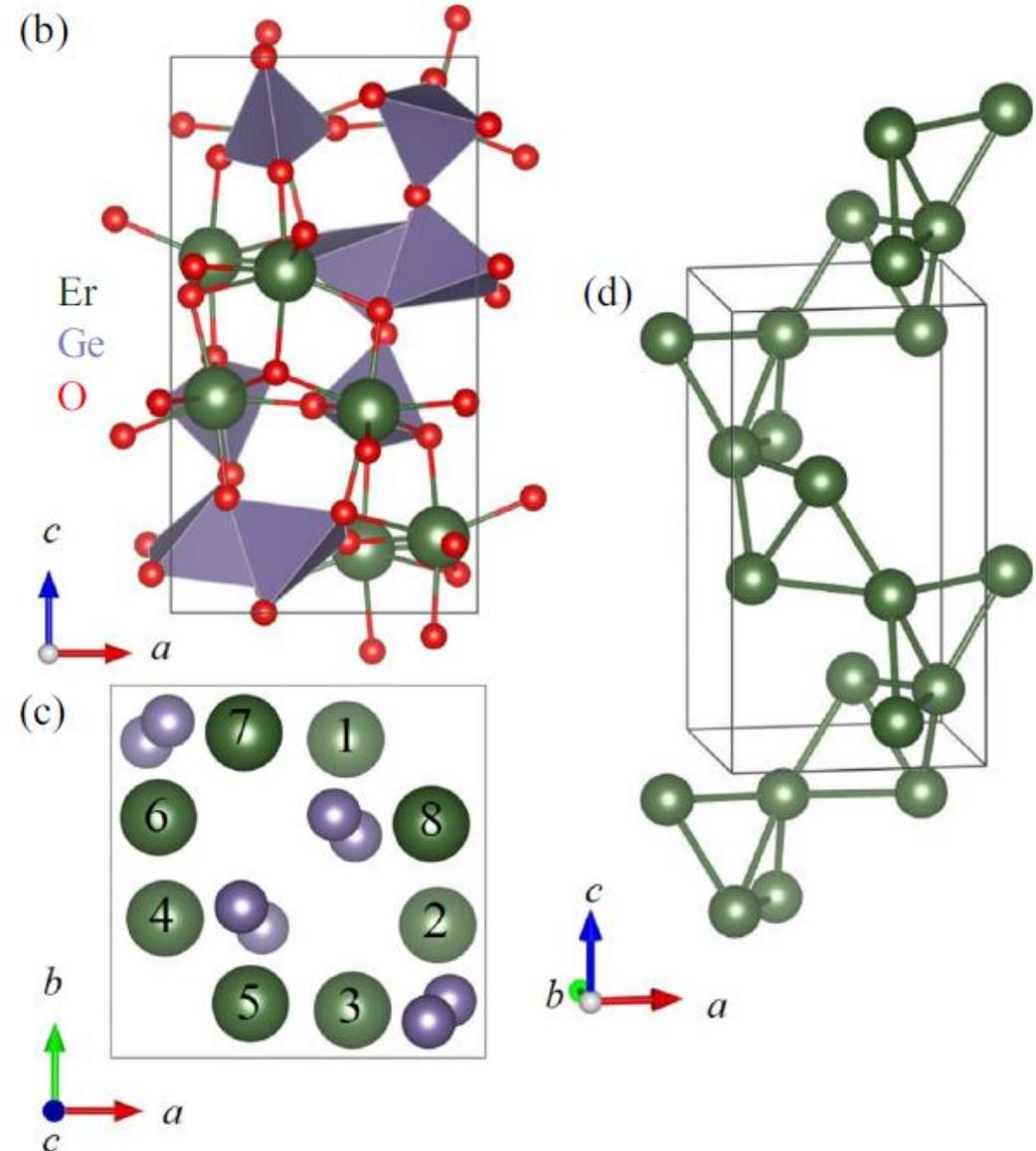
Similar frustration and spin-ice dynamics in Dy₂Ge₂O₇ and Ho₂Ge₂O₇

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

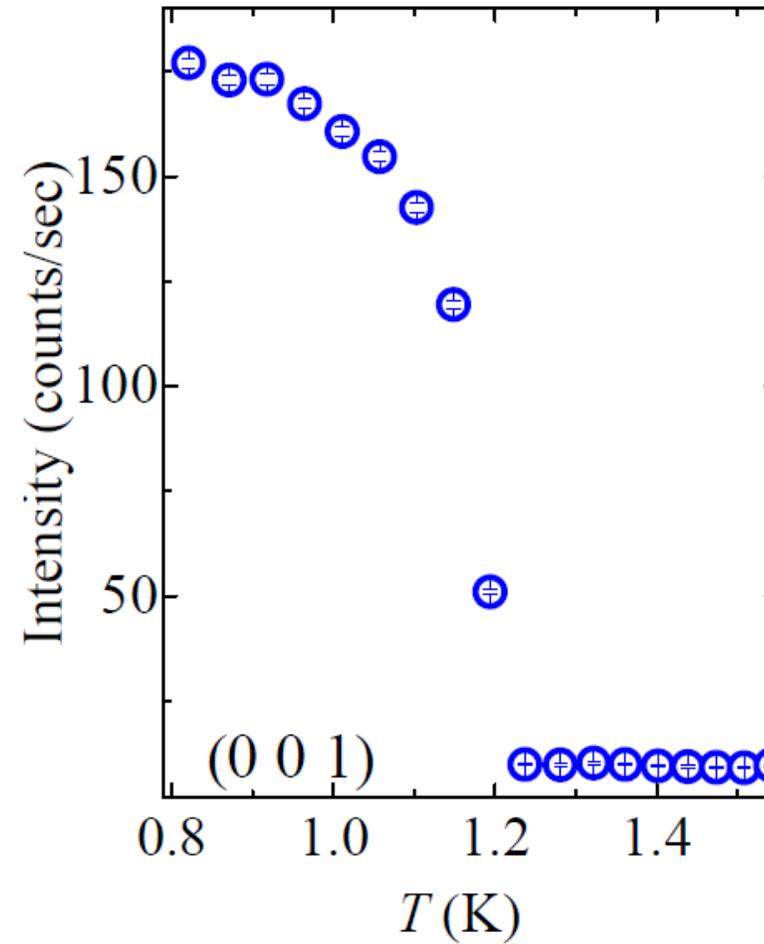
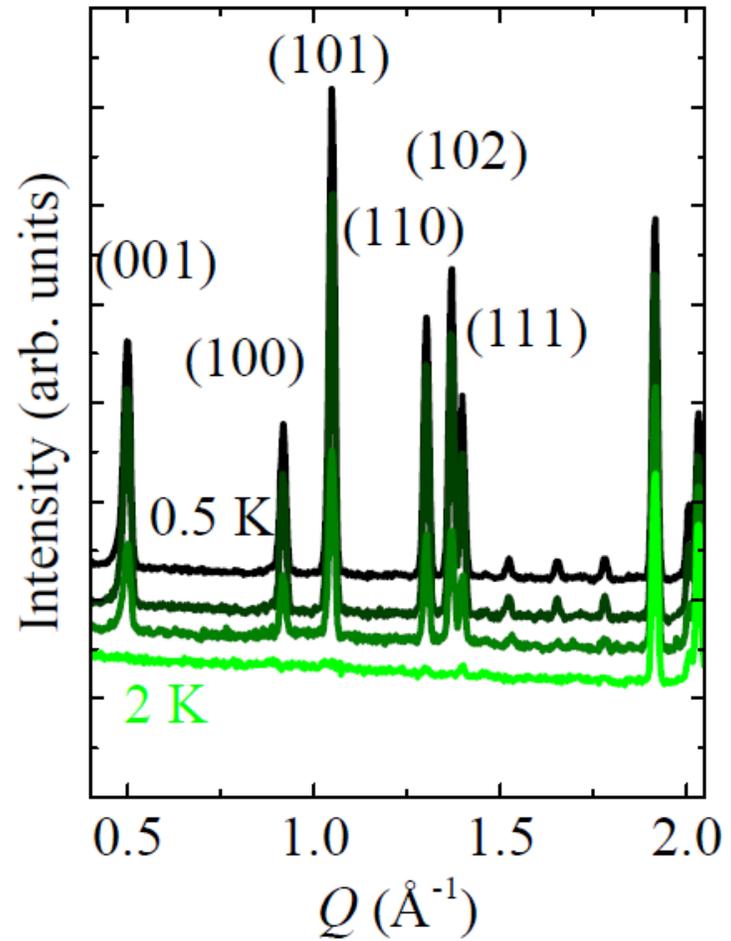
We'll look for similar behavior in $\text{Er}_2\text{Ge}_2\text{O}_7$



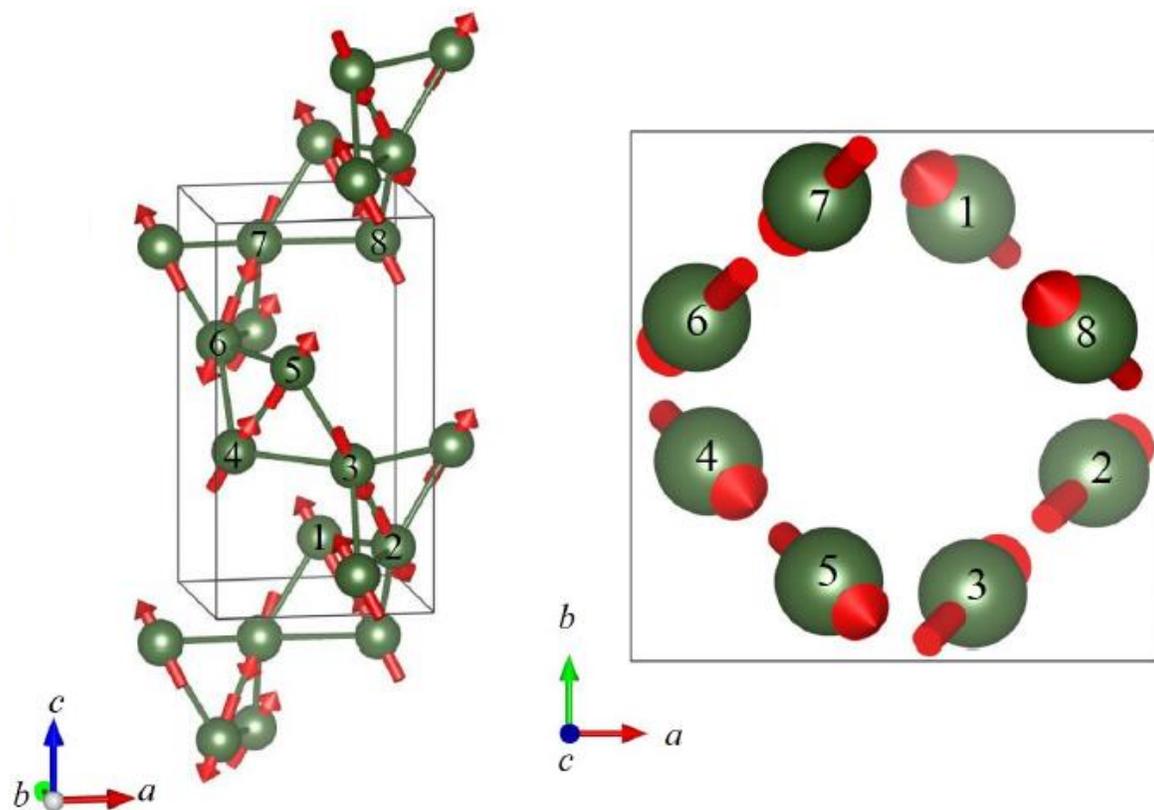
No evidence of magnetic order above 2 K, but we expect it from the Dy and Ho compounds



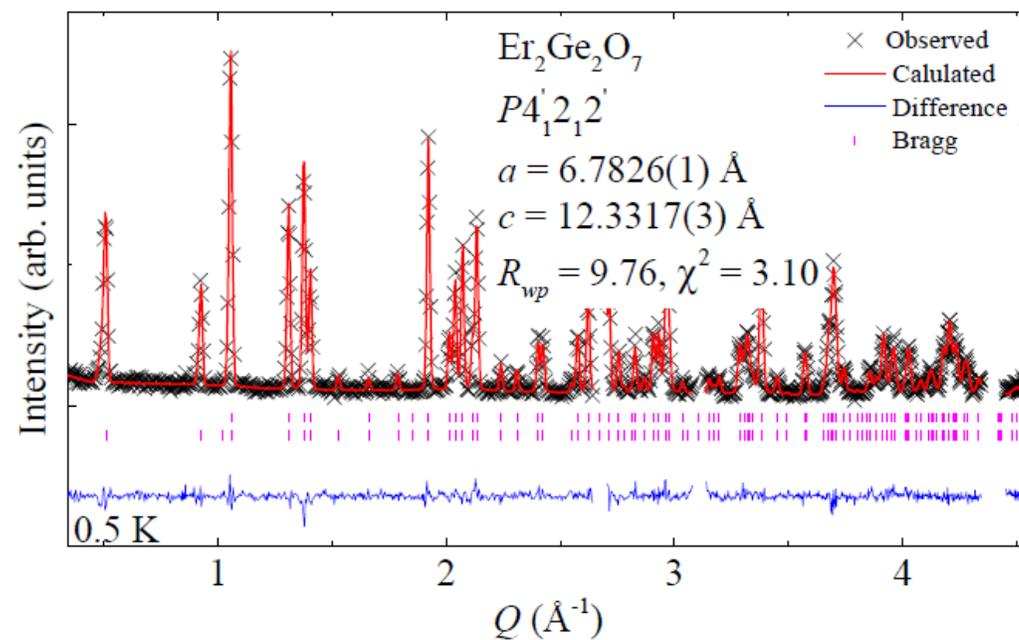
The neutron results we'll look at



The solved structure



Γ	Magnetic space group	ψ	R_{wp}	χ^2
Γ_1 (<i>mGM1</i>)	$P4_12_12$	3	48.6	76.8
Γ_2 (<i>mGM2</i>)	$P4'_12_12'$	3	9.76	3.10
Γ_3 (<i>mGM3</i>)	$P4_12'_12'$	3	49.8	80.7
Γ_4 (<i>mGM4</i>)	$P4_12'_12$	3	52.3	88.9
Γ_5 (<i>mGM5</i>)	$P2'_12'_12$	6	39.6	51.0
	$C22'_12'_1$	6	40.4	53.3
	$P2'_1$	12		



Installing GSAS-II

GSAS-2

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](#). If 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/GSASIIidistributions>, 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.
 - o 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.
 - o 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)

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Neutron Powder Diffractometer POWDER | HB-2A | HFIR

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HB-2A

Calibration Standards

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

POWGEN

Data Reduction and Analysis by Run Cycle

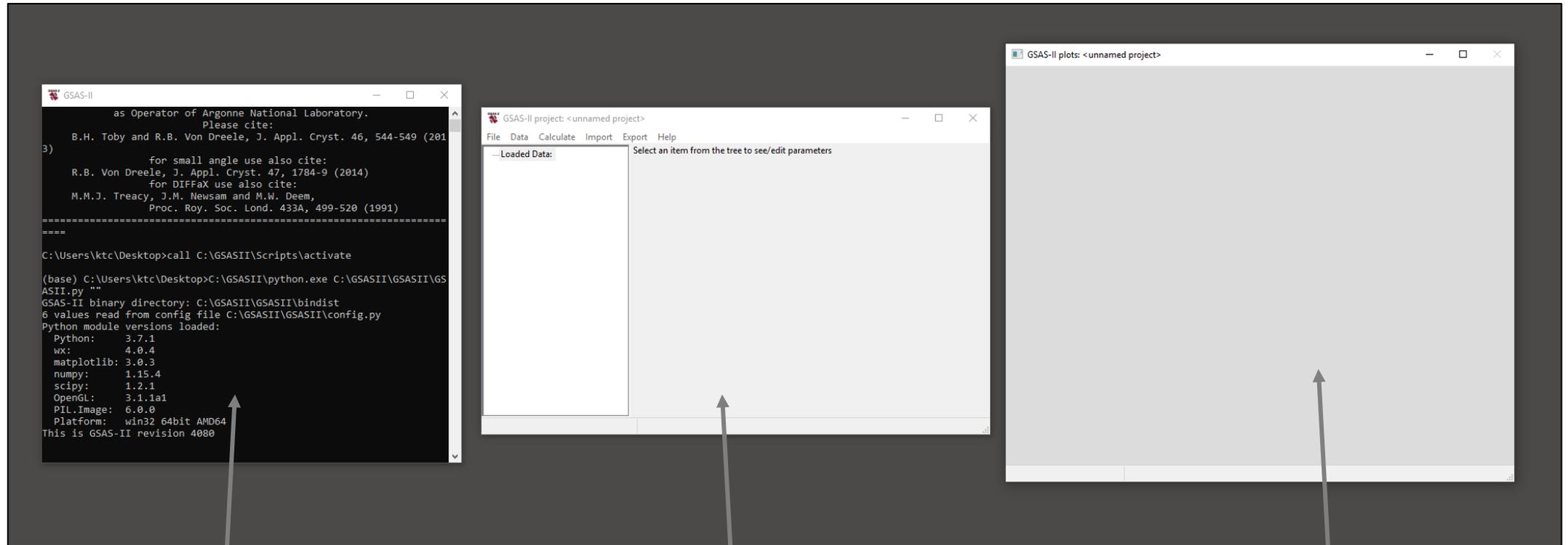
Parameter files for GSAS, GSAS-II, Fullprof and Topas can be downloaded from the table below. Be sure to pick the correct cycle. Also these files are stored in the /SNS/PG3/run_cycle_11A_CAL folder in the analysis computer where cycle is year_1 or year_2 based on cycle A or B.

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

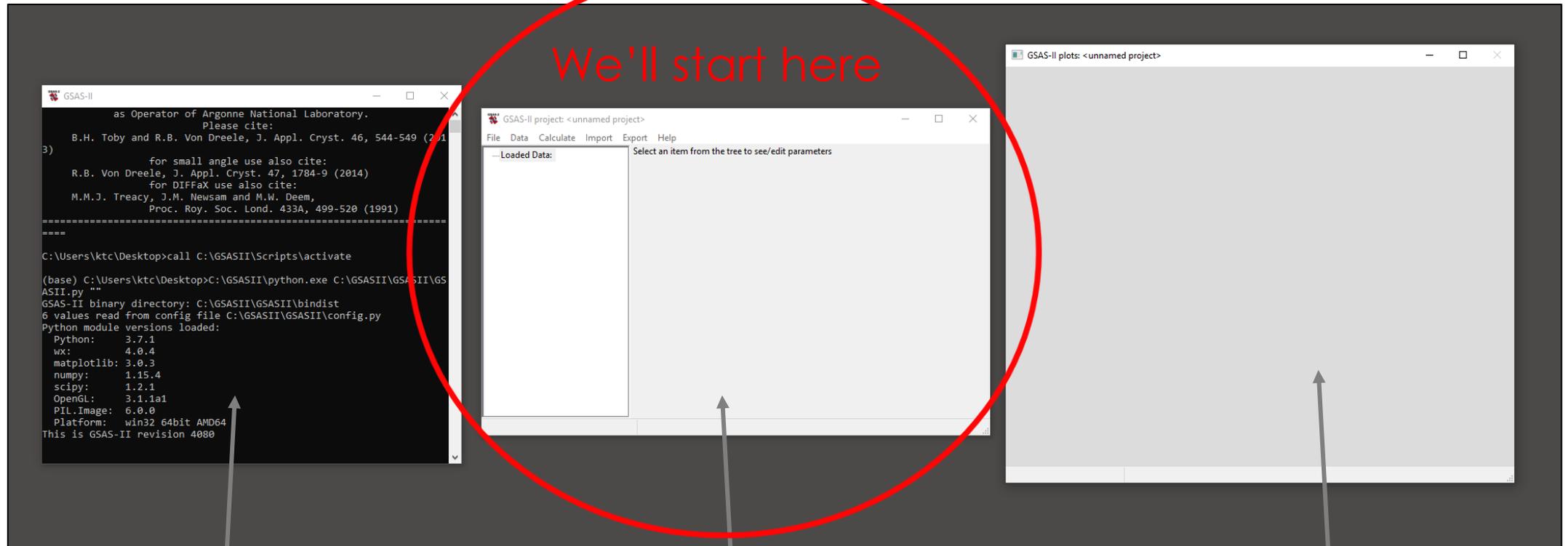


Python window
(won't use here)

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

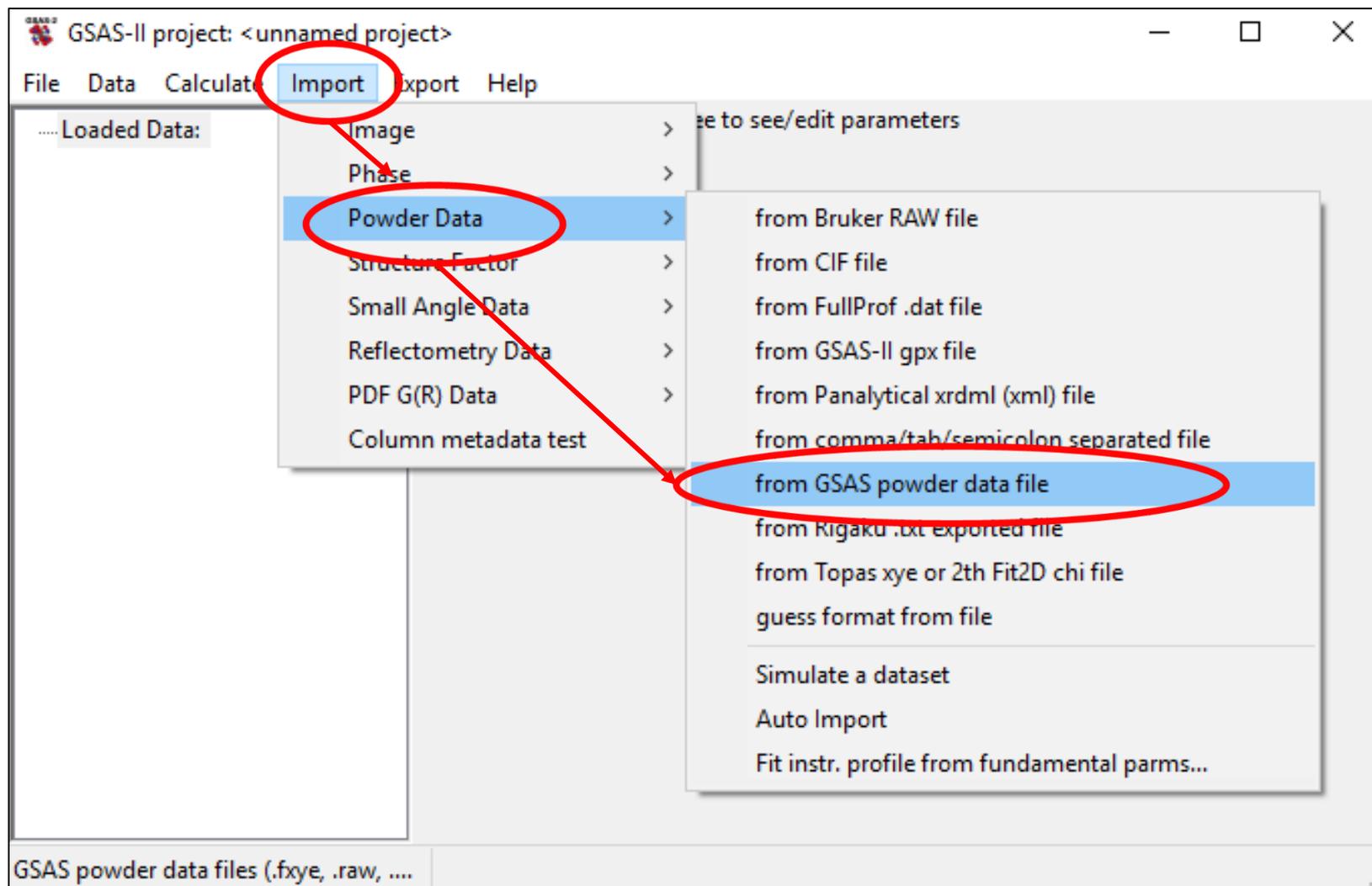


Python window
(won't use here)

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

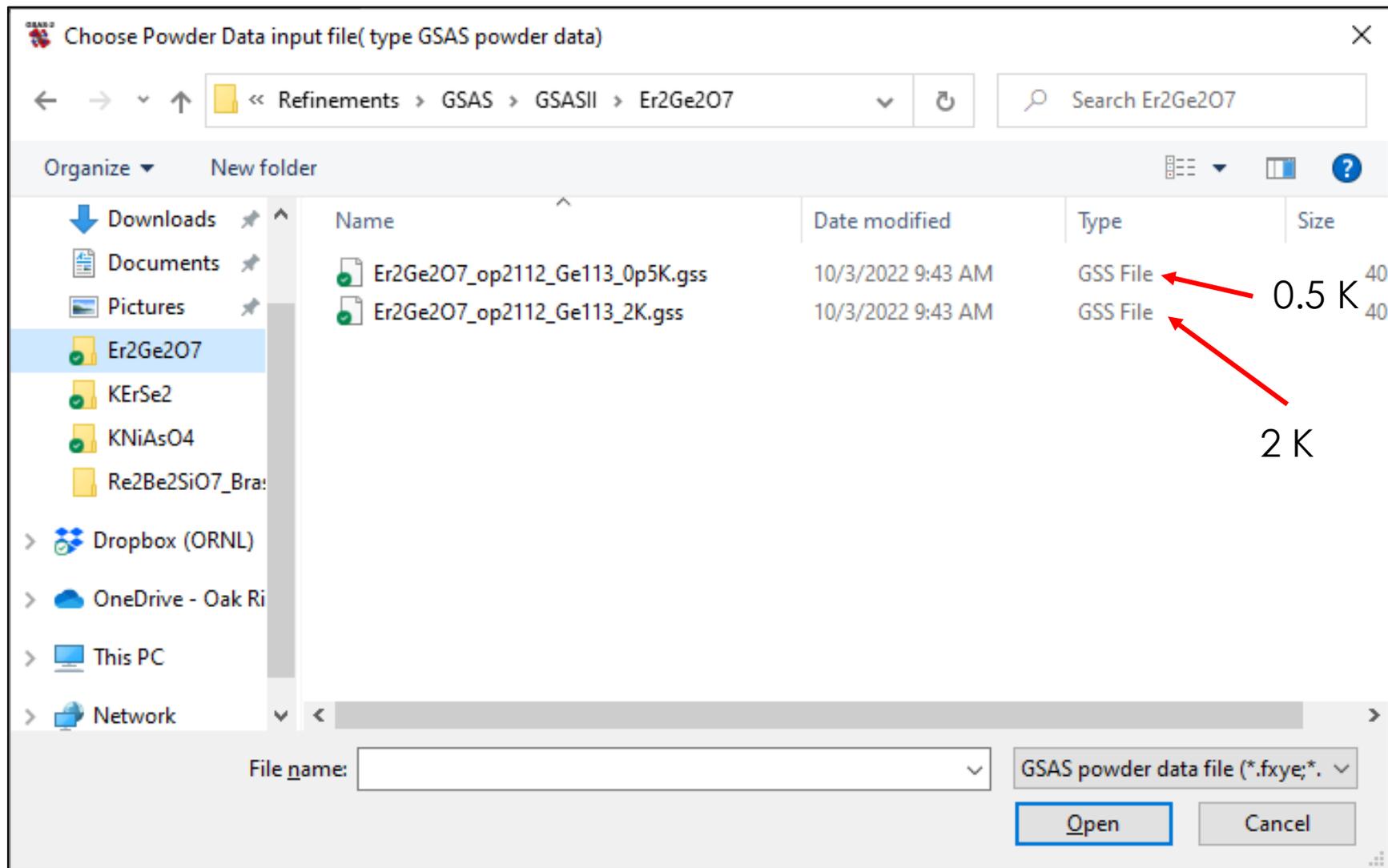
Plotting window
(automatically plots
based on what you
do in the GUI)

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'.

Choose Powder Data input file(type GSAS powder data)

< > << Refinements > GSAS > GSASII > Er2Ge207

Search Er2Ge207

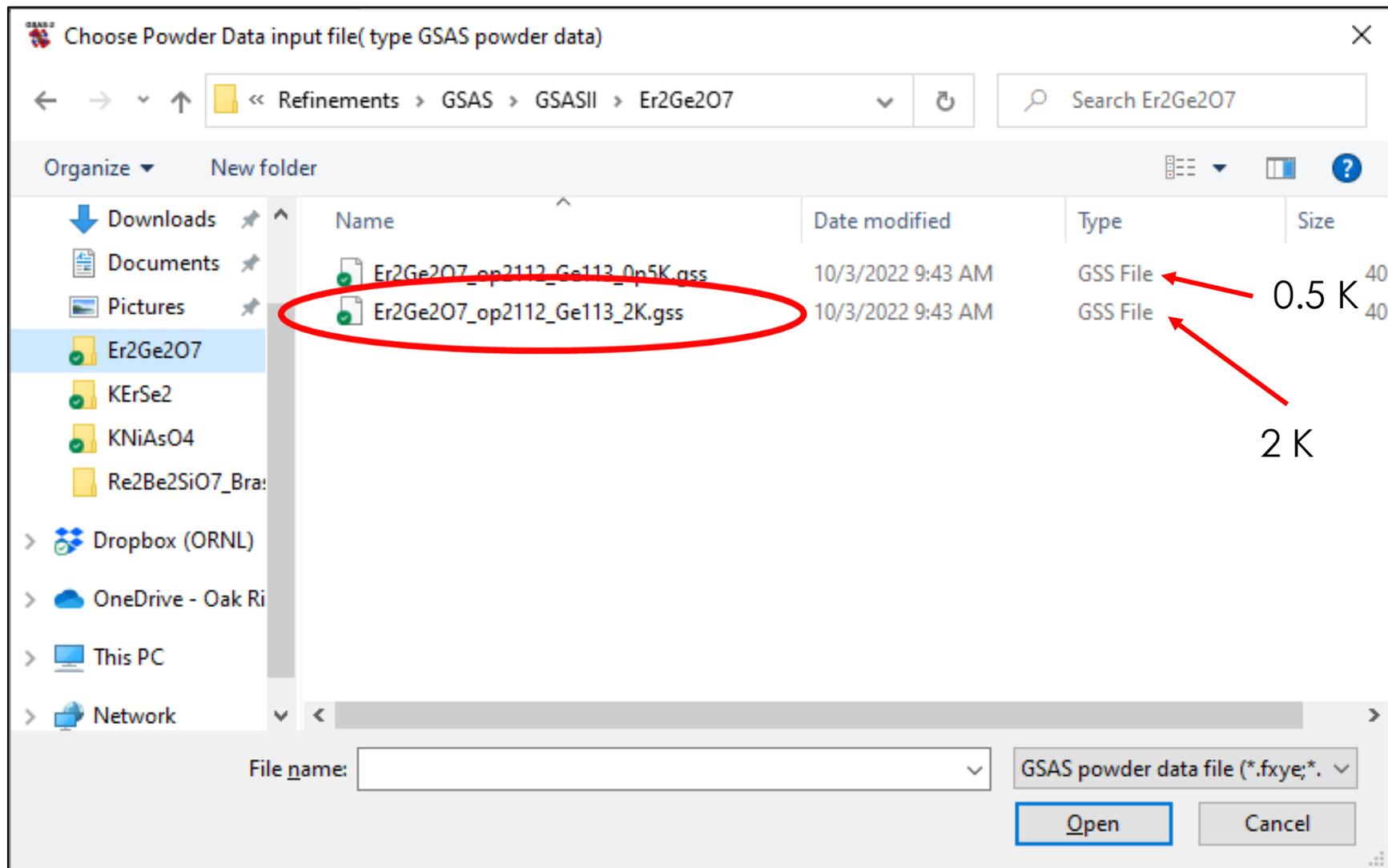
Organize New folder

Name	Date modified	Type	Size
Er2Ge207_op2112_Ge113_0p5K.gss	10/3/2022 9:43 AM	GSS File	0.5 K
Er2Ge207_op2112_Ge113_2K.gss	10/3/2022 9:43 AM	GSS File	2 K

ONCat - /HFIR/HB2A/IPTS-19406

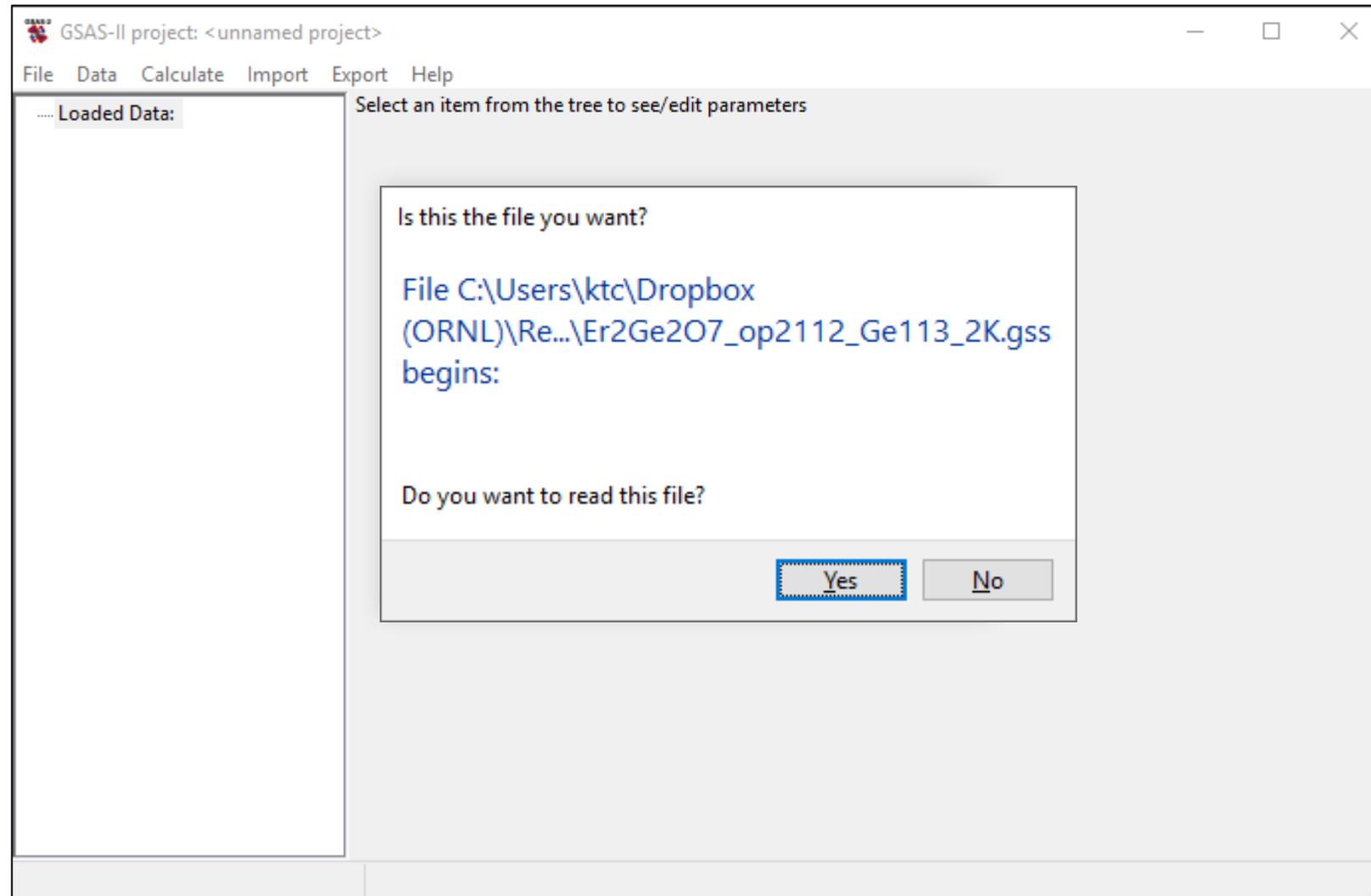
Date	Scan	Sample	File Name
2017/05/11 15:47:06 EDT	scan 2theta 2.6 0.1 preset time 60	ErGeO, 2.41, op-21-12, 0.5K, 1T, 1hr, 500mK	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0033.dat
2017/05/11 14:45:24 EDT	scan 2theta 2.6 0.1 preset time 60	ErGeO, 2.41, op-21-12, 0.5K, 1T, 1hr, 500mK	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0032.dat
2017/05/11 13:51:41 EDT	scan 2theta 2.6 0.1 preset time 60	ErGeO, 2.41, op-21-12, 0.5K, 1T, 1hr, 500mK	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0031.dat
2017/05/11 12:57:52 EDT	scan 2theta 2.6 0.1 preset time 60	ErGeO, 2.41, op-21-12, 0.5K, 1T, 1hr, 500mK	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0030.dat
2017/05/11 12:57:29 EDT	scan 2theta 2.6 0.1 preset time 60	ErGeO, 2.41, op-21-12, 0.5K, 1T, 1hr, 500mK	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0029.dat
2017/05/11 11:43:07 EDT	scan 2theta 2.7 0.05 preset time 60	ErGeO, 2.41, op-21-12, 1.2K 2hr	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0028.dat
2017/05/11 09:44:20 EDT	scan 2theta 2.7 0.05 preset time 60	ErGeO, 2.41, op-21-12, 1.25K 2hr	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0027.dat
2017/05/11 09:07:52 EDT	scan 2theta 2.7 0.05 preset time 60	ErGeO, 2.41, op-21-12, 0.8K 2hr	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0026.dat
2017/05/11 07:11:02 EDT	scan 2theta 2.7 0.05 preset time 60	ErGeO, 1.54, op-21-12, 0.5K 2hr	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0025.dat
2017/05/11 05:06:17 EDT	scan 2theta 2.7 0.05 preset time 60	ErGeO, 1.54, op-21-12, 2K 2hr	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0024.dat
2017/05/11 03:14:14 EDT	scan 2theta 2.9 0.05 preset time 100	ErGeO, 2.41, op-21-12, 2K 4hr	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0023.dat
-	scanon	OP (warming) for mag peak at 11 deg, detector 3	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0022.dat
-	scanon	OP (warming) for mag peak at 11 deg, detector 3	/HFIR/HB2A/IPTS-19406/exp551/Datafiles/HB2A_exp0551_scan0021.dat

Loading your data



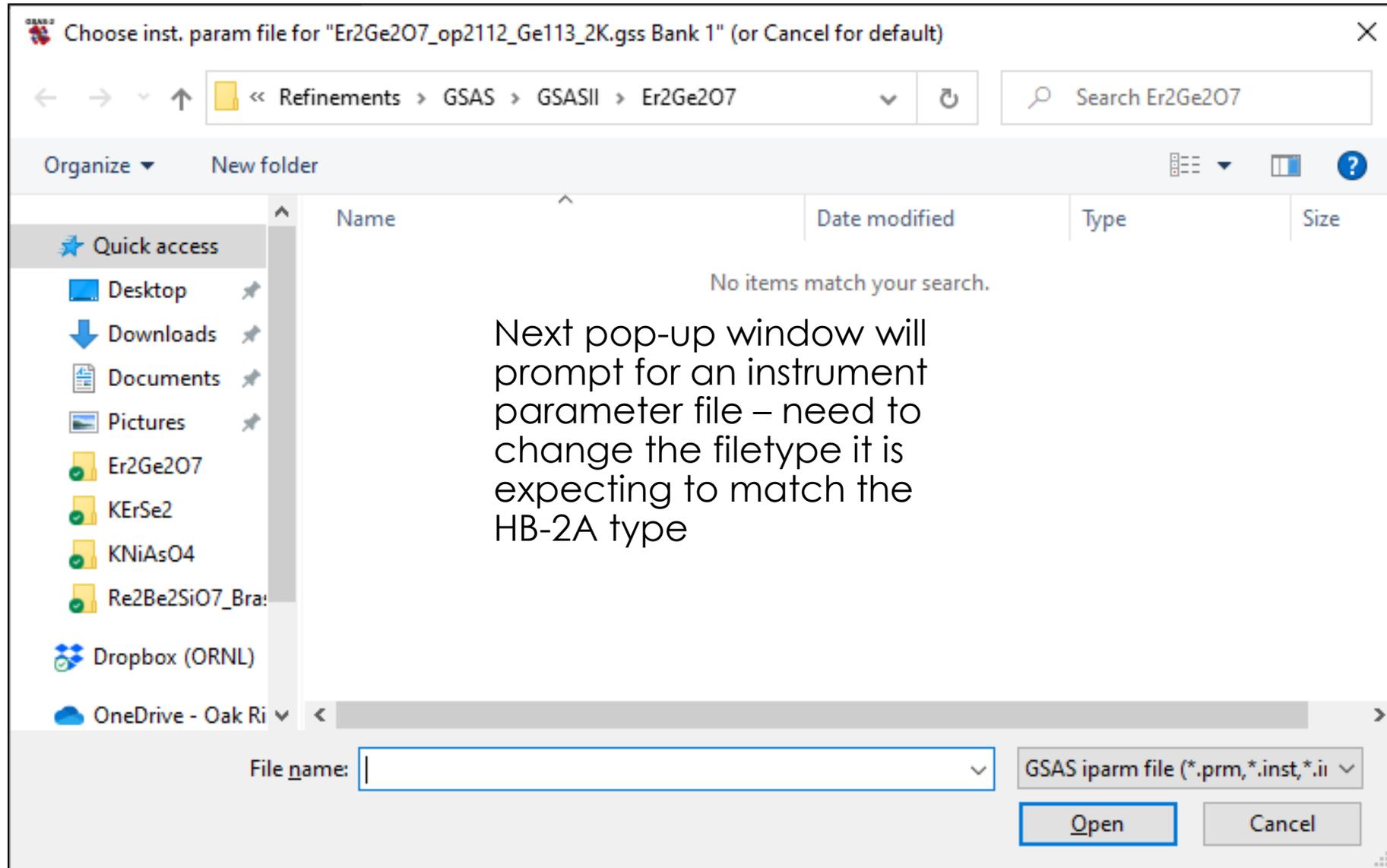
We'll start here

Loading your data

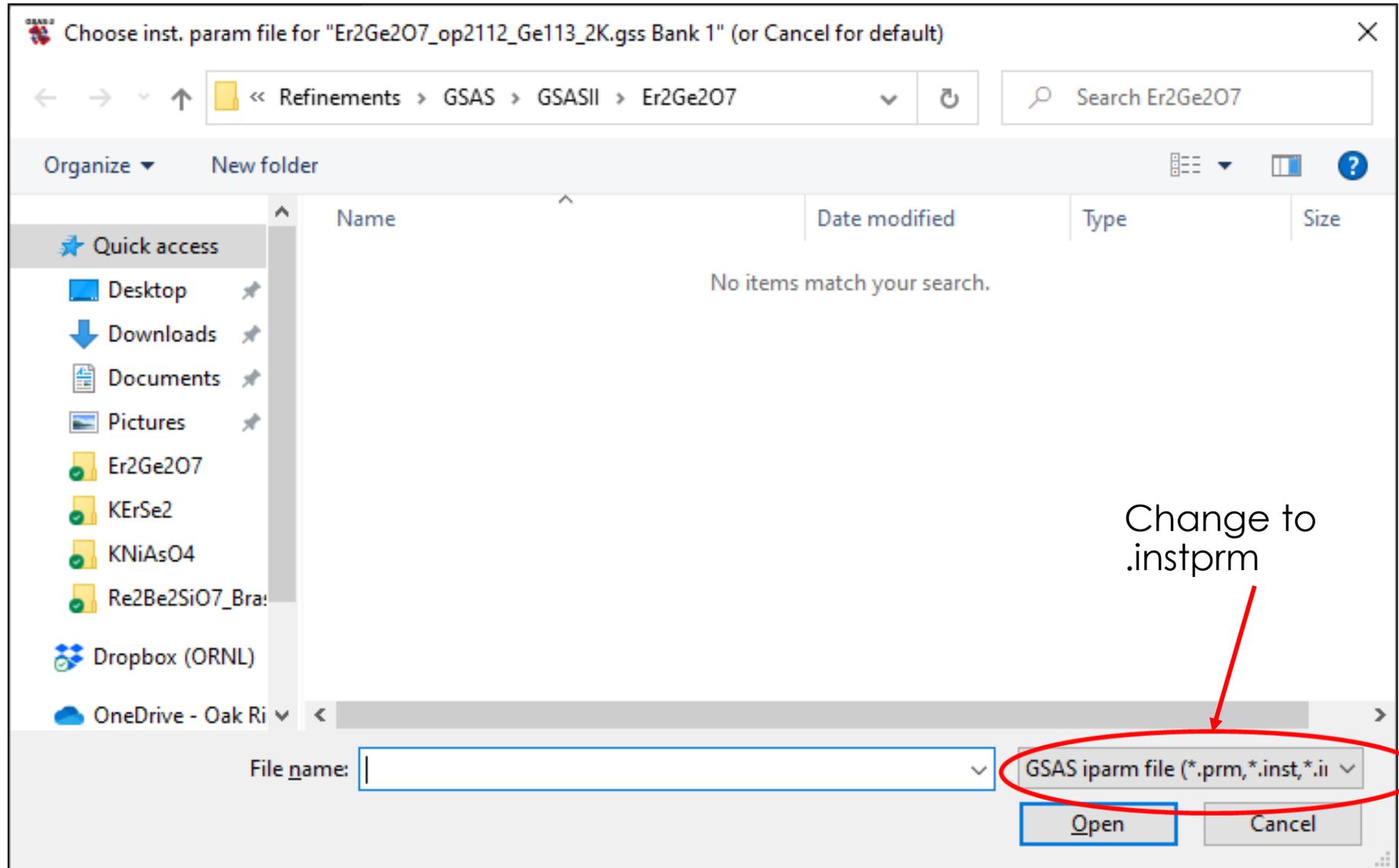


This window should pop-up
Click 'Yes'

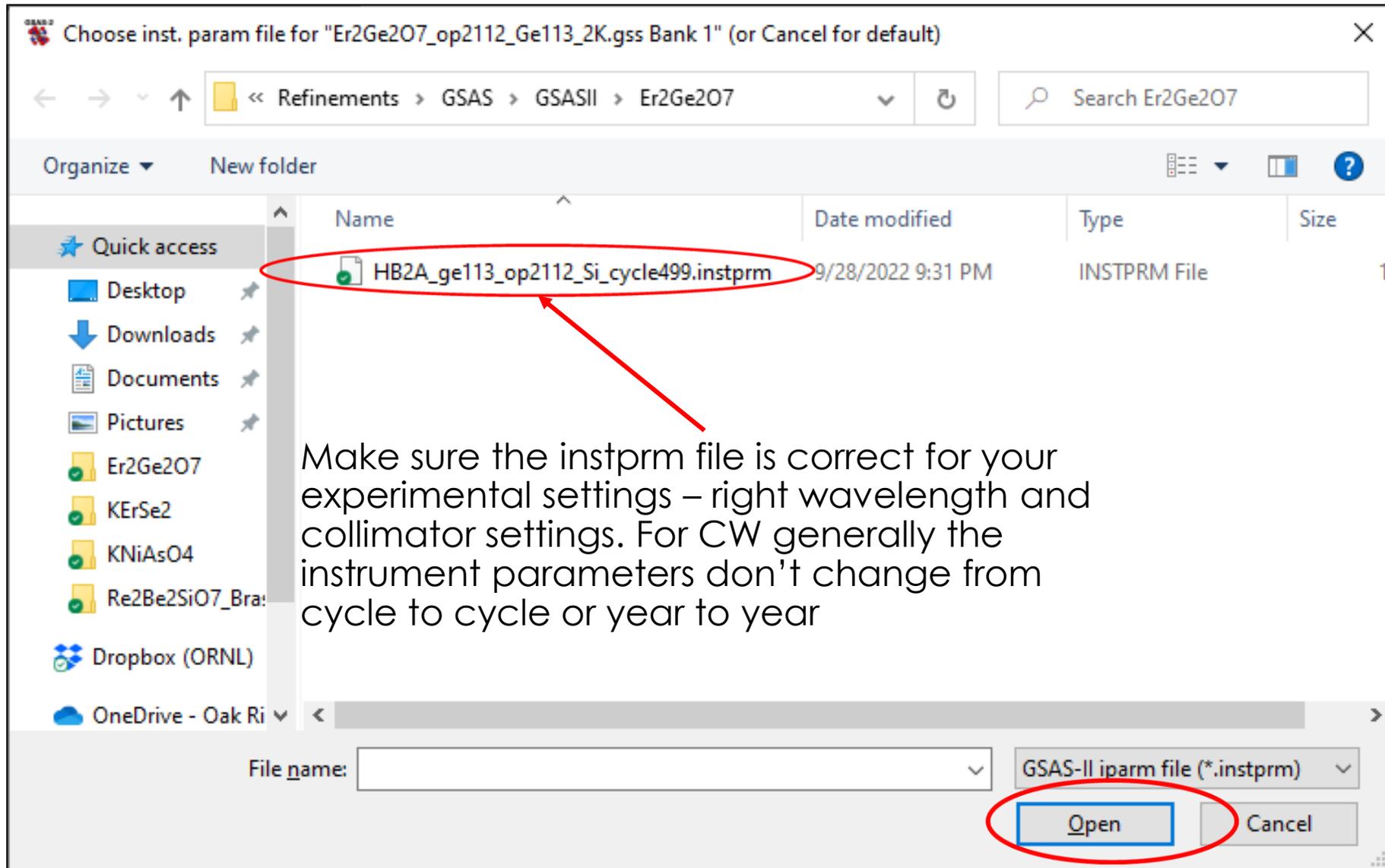
Loading your data



Loading your data



Loading your data



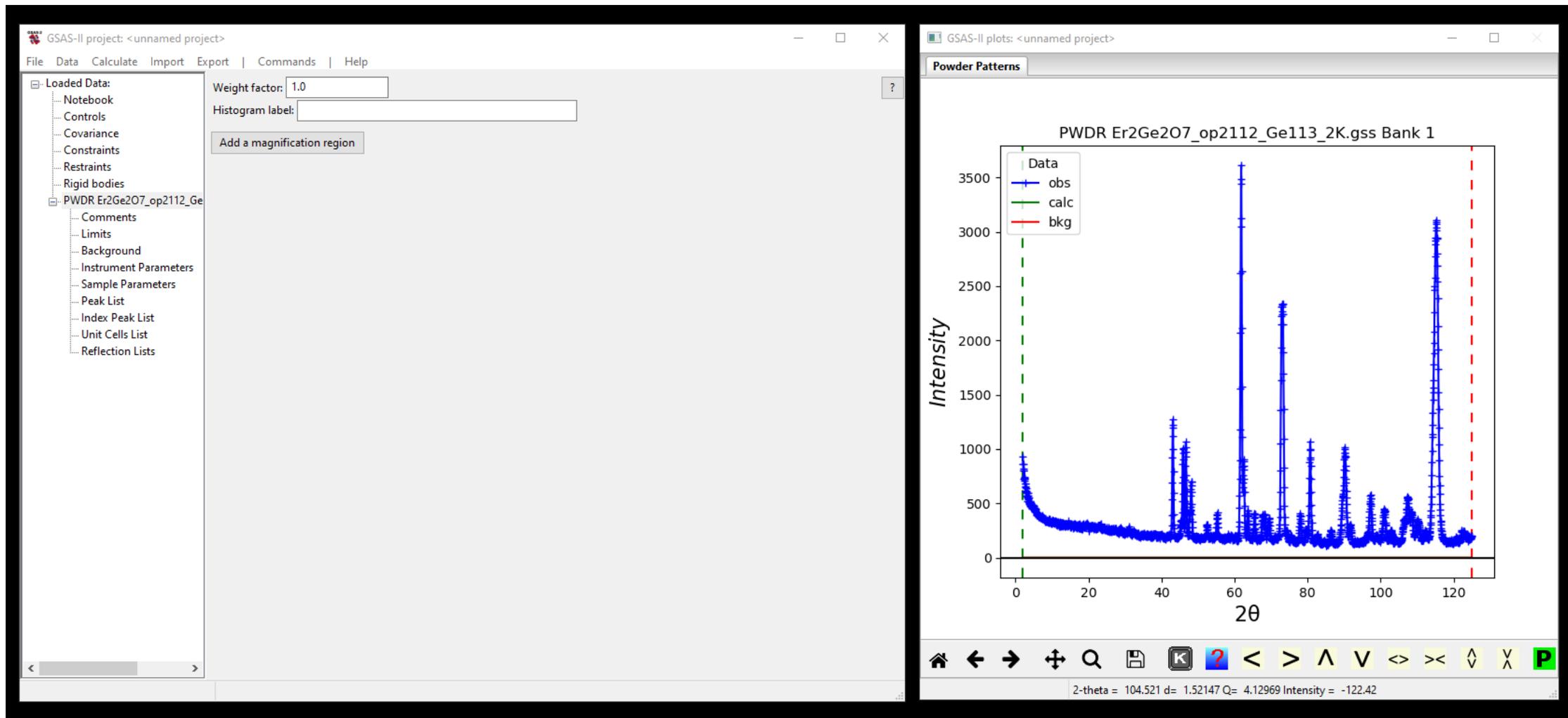
The screenshot shows a Windows File Explorer window titled "Choose inst. param file for 'Er2Ge207_op2112_Ge113_2K.gss Bank 1' (or Cancel for default)". The address bar shows the path: \ll Refinements > GSAS > GSASII > Er2Ge207. The left sidebar shows the "Quick access" pane with folders like Desktop, Downloads, Documents, Pictures, Er2Ge207, KErSe2, KNiAsO4, Re2Be2SiO7_Bra:, Dropbox (ORNL), and OneDrive - Oak Ri. The main pane displays a table of files:

Name	Date modified	Type	Size
HB2A_ge113_op2112_Si_cycle499.instprm	9/28/2022 9:31 PM	INSTPRM File	1

The file "HB2A_ge113_op2112_Si_cycle499.instprm" is circled in red, and a red arrow points to it. The "File name:" field at the bottom is empty, and the file type is set to "GSAS-II iparm file (*.instprm)". The "Open" button is also circled in red.

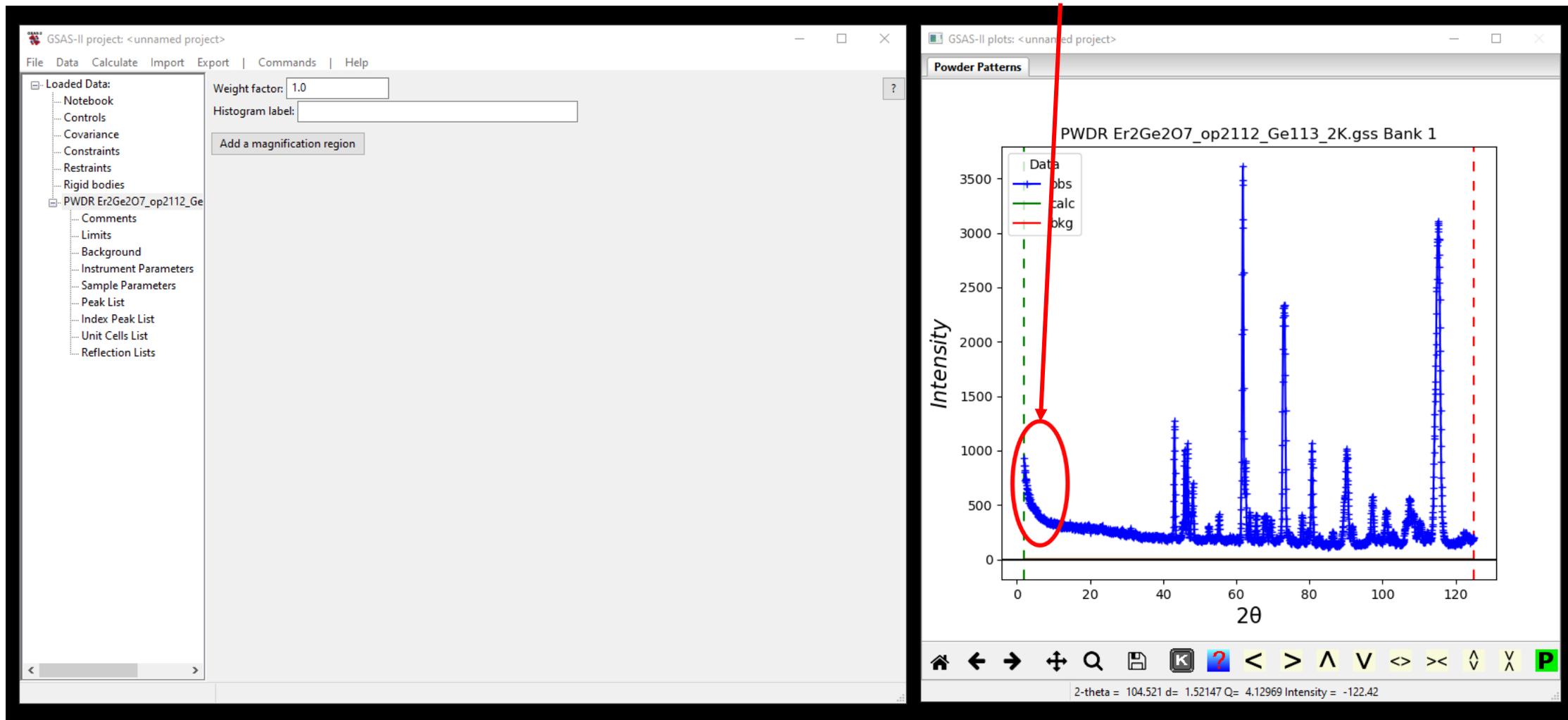
Make sure the instprm file is correct for your experimental settings – right wavelength and collimator settings. For CW generally the instrument parameters don't change from cycle to cycle or year to year

Data loaded



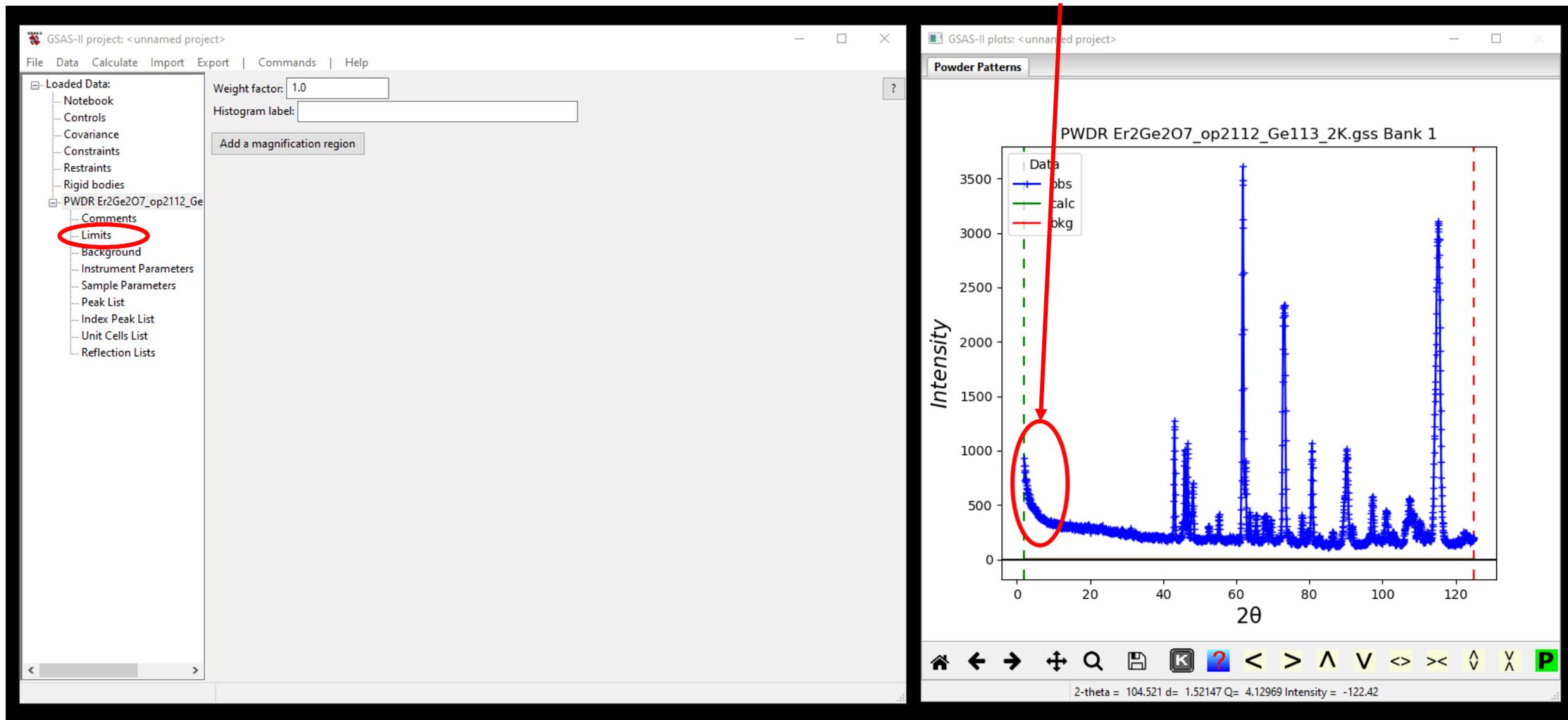
Excluding regions

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



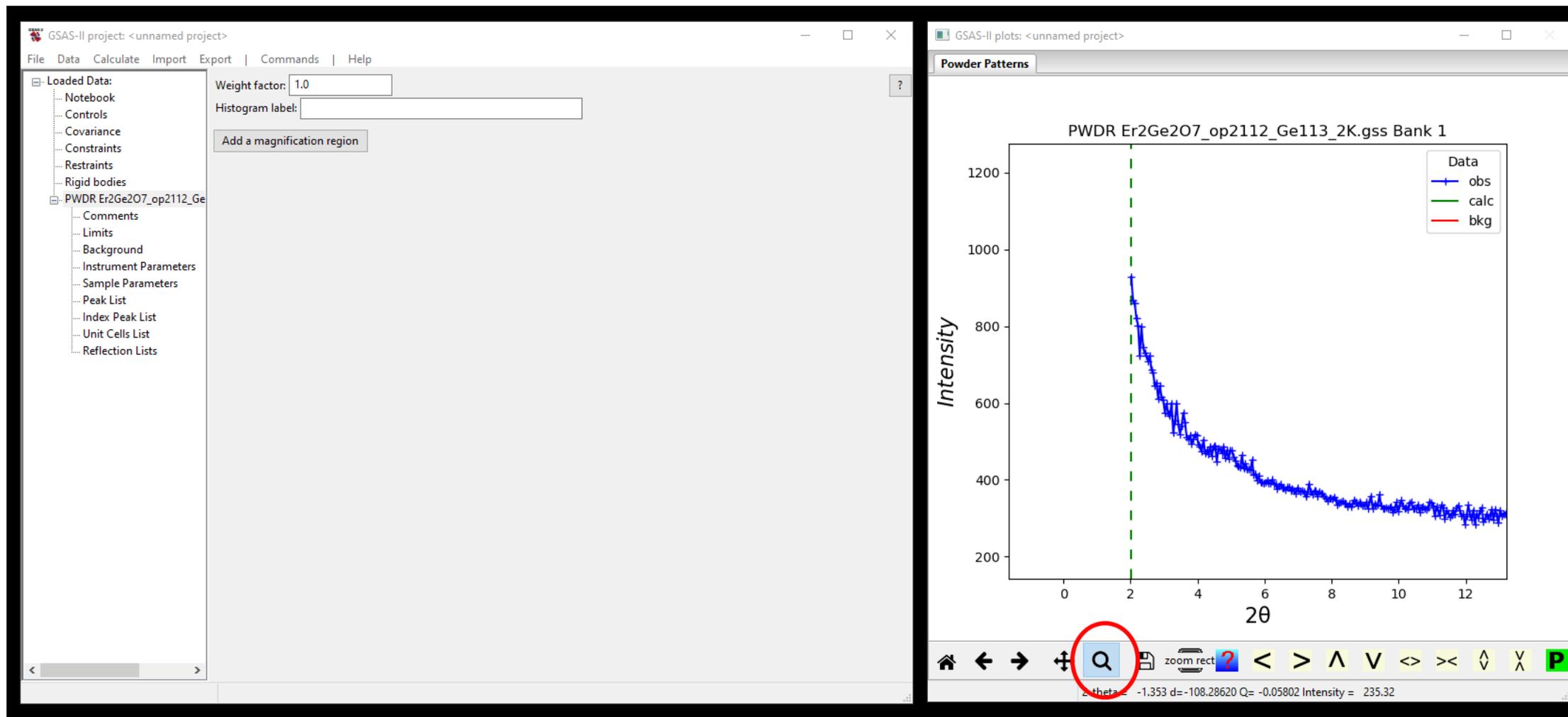
Excluding regions

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



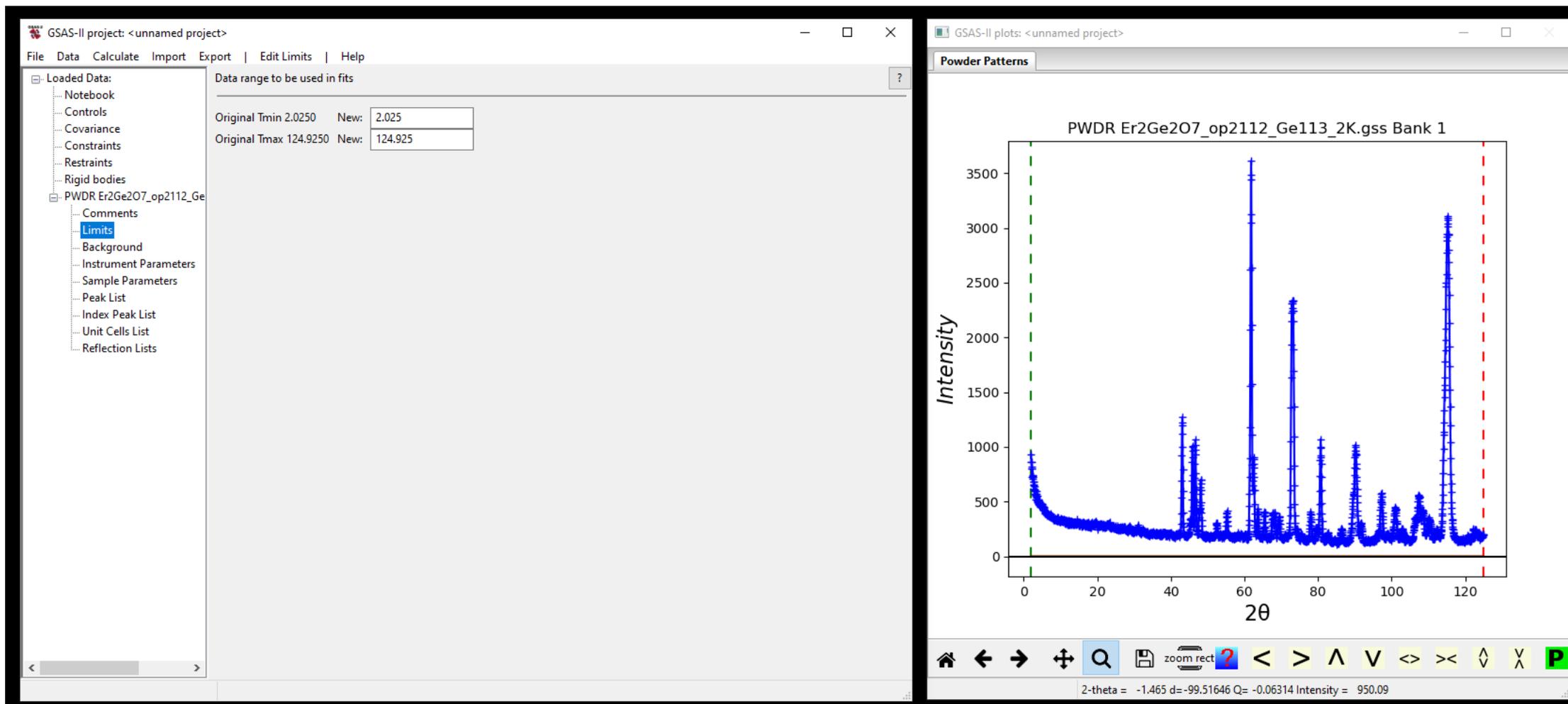
We'll do so using limits

Excluding regions: upper limit



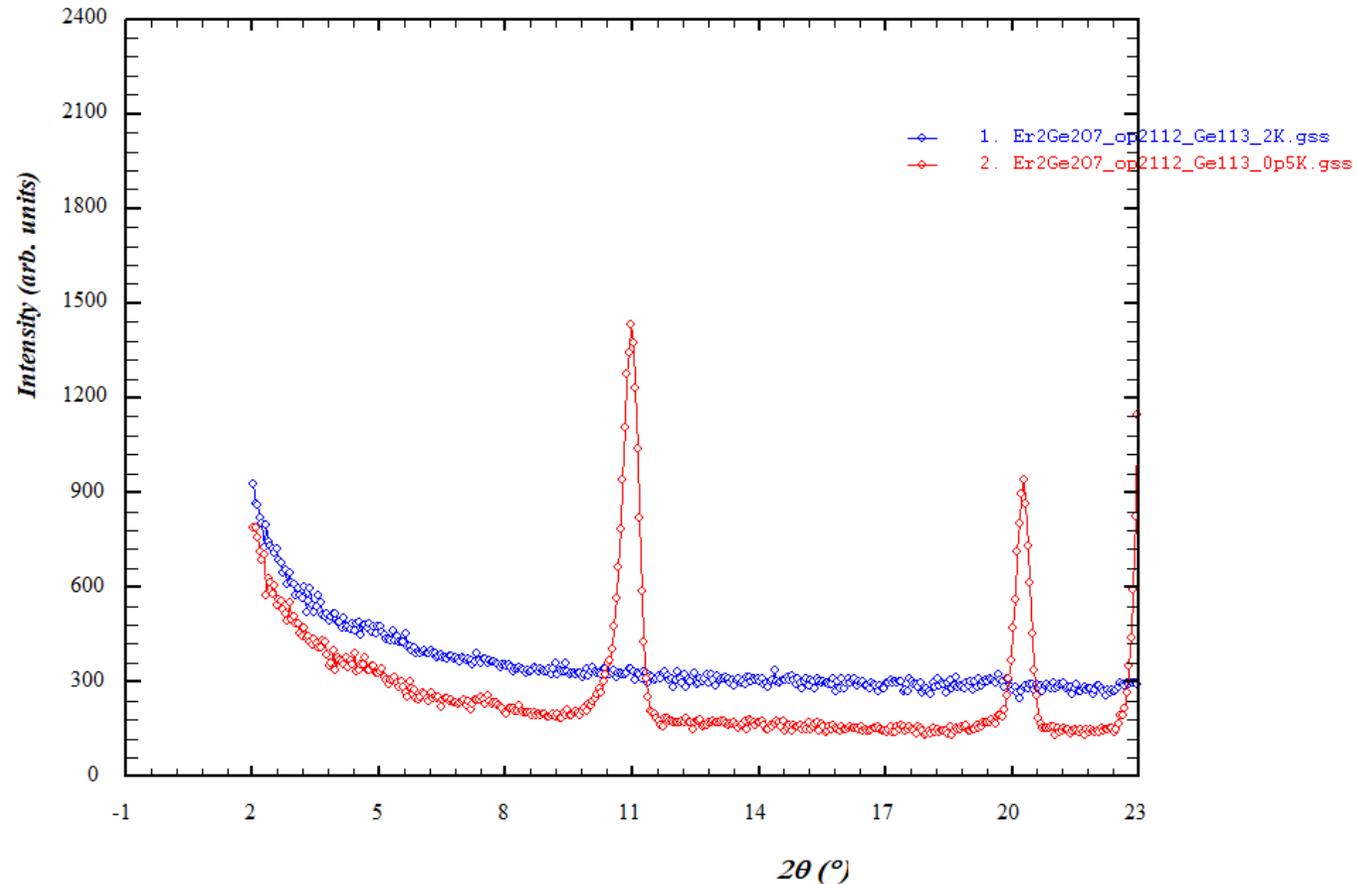
Using the magnifying glass in the plotting window we can zoom in, it has a sharp upturn that is not from our sample but from the direct beam

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 $\sim 11^\circ 2\theta$. Let's cut everything below $\sim 8^\circ 2\theta$ then.

Excluding regions: lower limit

Let's change this to 8°

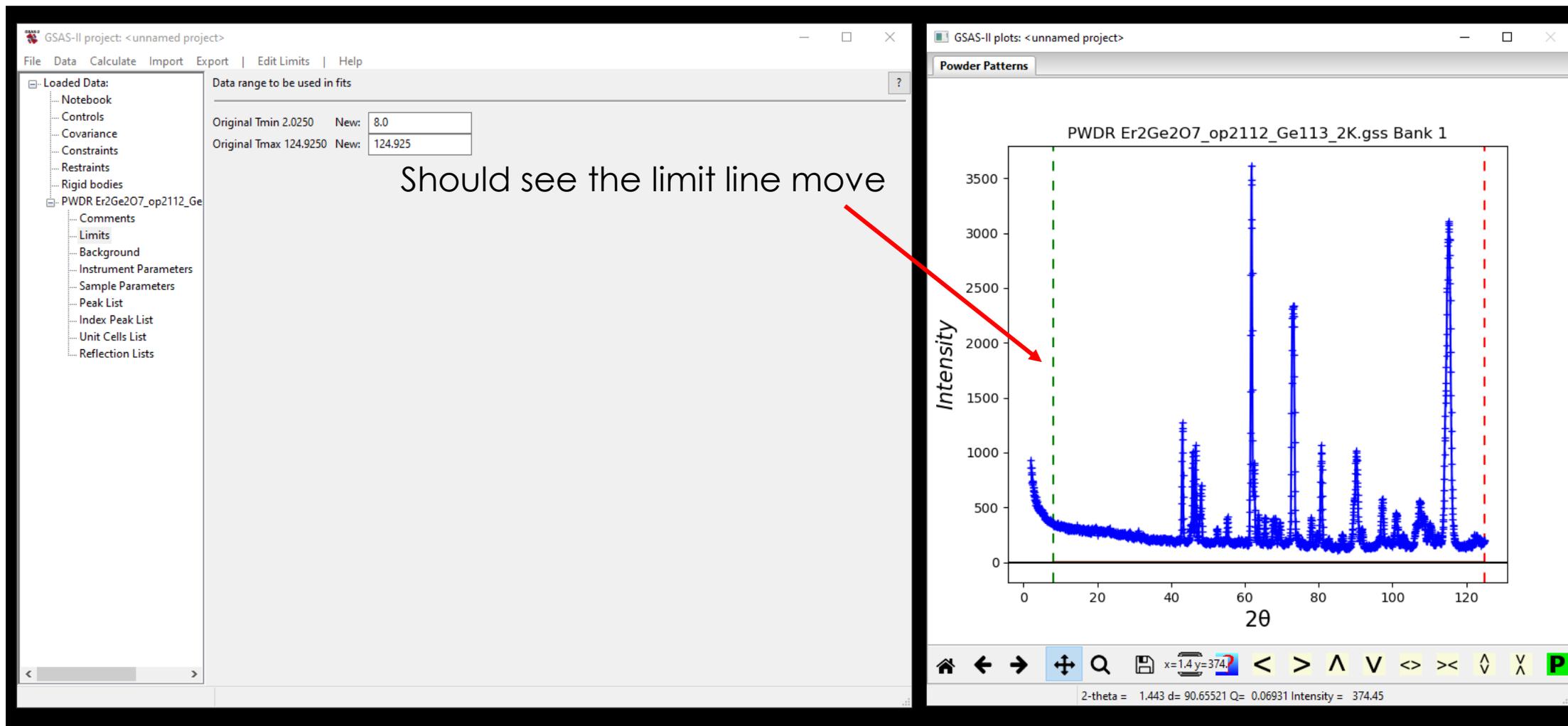
Original Tmin 2.0250 New: 2.025
Original Tmax 124.9250 New: 124.925

Powder Patterns
PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1

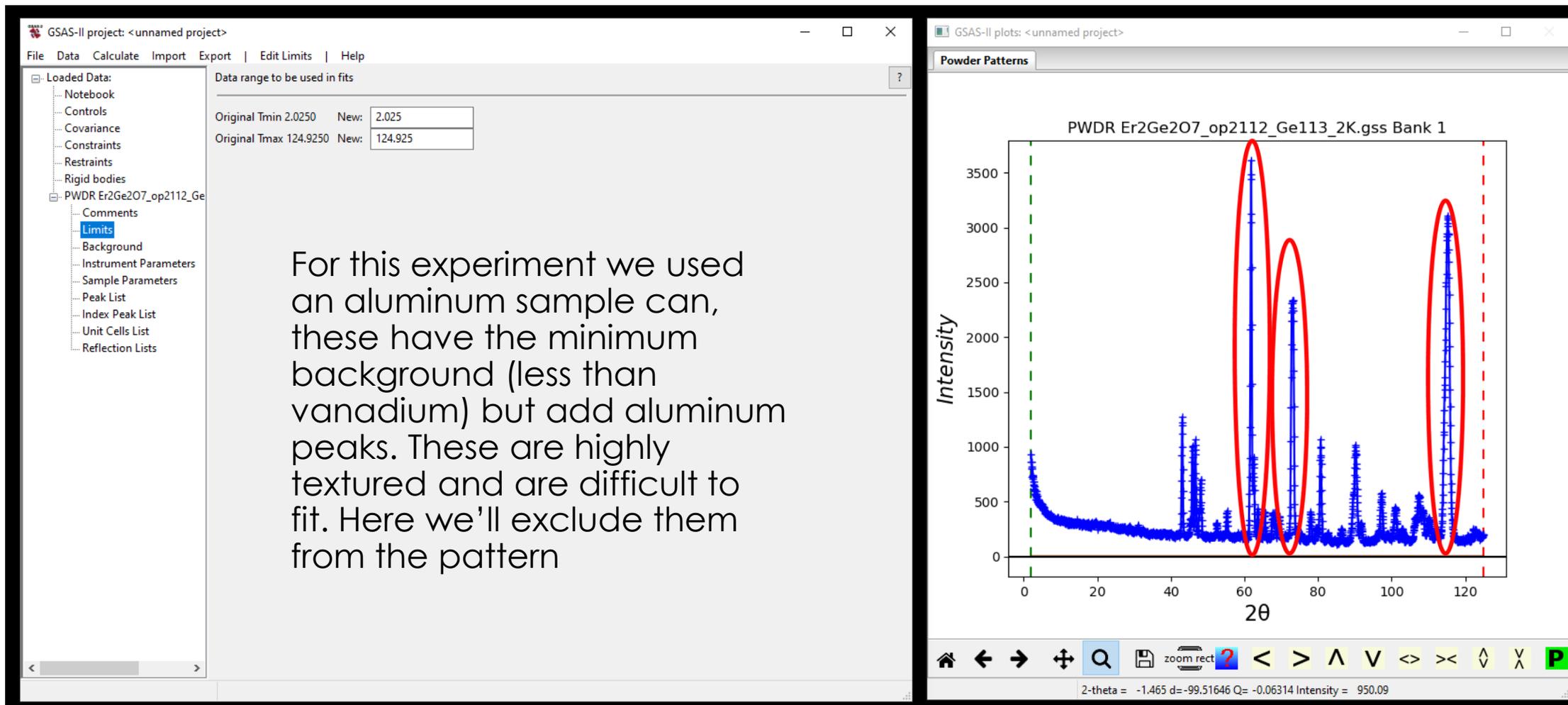
Intensity
2θ

2-theta = -1.465 d=-99.51646 Q=-0.06314 Intensity = 950.09

Excluding regions: lower limit



Excluding regions: Aluminum (can) peaks



The image shows two windows from the GSAS-II software. The left window, titled 'GSAS-II project: <unnamed project>', displays the 'Limits' tab. It shows the 'Data range to be used in fits' with 'Original Tmin 2.0250' and 'New: 2.025', and 'Original Tmax 124.9250' and 'New: 124.925'. The right window, titled 'GSAS-II plots: <unnamed project>', shows a powder pattern plot for 'PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1'. The plot shows Intensity versus 2θ, with several peaks highlighted by red ovals. A green dashed vertical line is at 2θ ≈ 5, and a red dashed vertical line is at 2θ ≈ 125. The status bar at the bottom indicates '2-theta = -1.465 d=-99.51646 Q=-0.06314 Intensity = 950.09'.

For this experiment we used an aluminum sample can, these have the minimum background (less than vanadium) but add aluminum peaks. These are highly textured and are difficult to fit. Here we'll exclude them from the pattern

Excluding regions: Aluminum (can) peaks

The image shows two windows from the GSAS-II software. The left window, titled 'GSAS-II project: <unnamed project>', displays the 'Edit Limits' dialog box. The 'Edit Limits' menu item is circled in red, and the 'Add exclude' button is also circled in red. The dialog box contains a table with the following data:

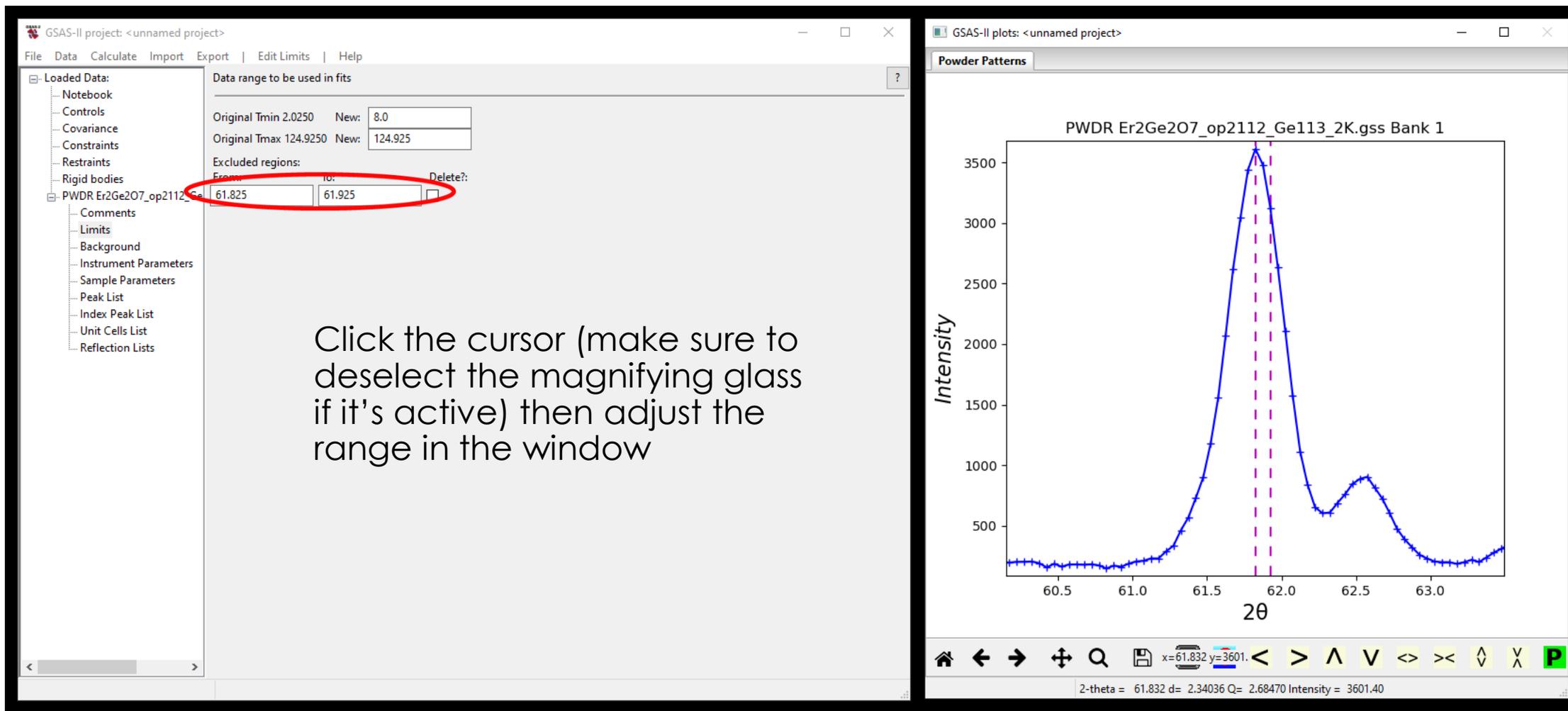
Original T _{min}	New
2.0250	2.025
Original T _{max}	New
124.9250	124.925

Below the table, the text 'Zoom in on the first aluminum peak (around 62°) and go to Edit Limits -> Add exclude' is displayed. The right window, titled 'GSAS-II plots: <unnamed project>', shows a zoomed-in powder pattern plot. The plot is titled 'PWDR Er₂Ge₂O₇_op2112_Ge113_2K.gss Bank 1'. The y-axis is labeled 'Intensity' and ranges from 0 to 3500. The x-axis is labeled '2θ' and ranges from 60.0 to 63.5. A prominent peak is visible at approximately 62.0°, with an intensity of about 3500. The plot shows a blue line with markers representing the data points. At the bottom of the plot window, the following information is displayed: '2-theta = 60.190 d= 2.39789 Q= 2.62030 Intensity = 686.70'.

Excluding regions: Aluminum (can) peaks

The image shows two windows from the GSAS-II software. The left window, titled "GSAS-II project: <unnamed project>", displays the "Data range to be used in fits" settings. The "Original Tmin" is 2.0250 and the "New" value is 8.0. The "Original Tmax" is 124.9250 and the "New" value is 124.925. A large "Click OK" text is overlaid on the window. The right window, titled "GSAS-II plots: <unnamed project>", shows a powder pattern plot for "PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1". The plot shows Intensity versus 2θ. A dialog box titled "Creating excluded region" is overlaid on the plot, with an "OK" button circled in red. The dialog box contains the text: "Click on a point in the pattern to be excluded, then drag or edit limits to adjust range". The plot shows a sharp peak at approximately 61.8 degrees 2θ and a broader peak at approximately 62.5 degrees 2θ. The status bar at the bottom of the plot window shows: "2-theta = 60.267 d= 2.39514 Q= 2.62330 Intensity = 320.52".

Excluding regions: Aluminum (can) peaks



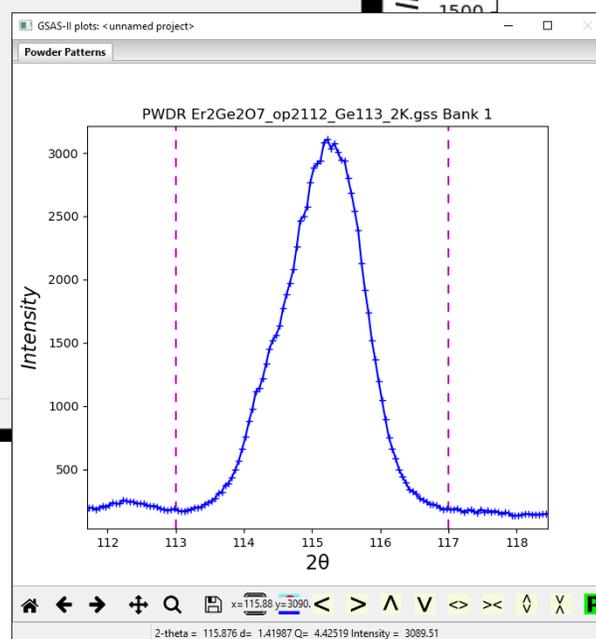
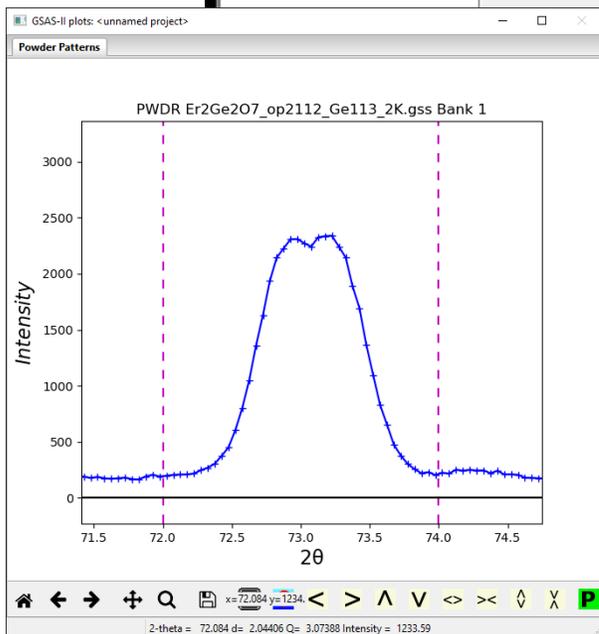
The image displays two windows from the GSAS-II software. The left window, titled 'GSAS-II project: <unnamed project>', shows the 'Data range to be used in fits' section. The 'Excluded regions' table is highlighted with a red circle, showing a region from 61.825 to 61.925. The right window, titled 'GSAS-II plots: <unnamed project>', shows a powder pattern plot for 'PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1'. The plot shows Intensity versus 2θ, with a prominent peak at approximately 61.832 degrees. A vertical dashed purple line is drawn at this peak position. The status bar at the bottom of the plot window displays the coordinates: '2-theta = 61.832 d= 2.34036 Q= 2.68470 Intensity = 3601.40'.

Click the cursor (make sure to deselect the magnifying glass if it's active) then adjust the range in the window

Excluding regions: Aluminum (can) peaks

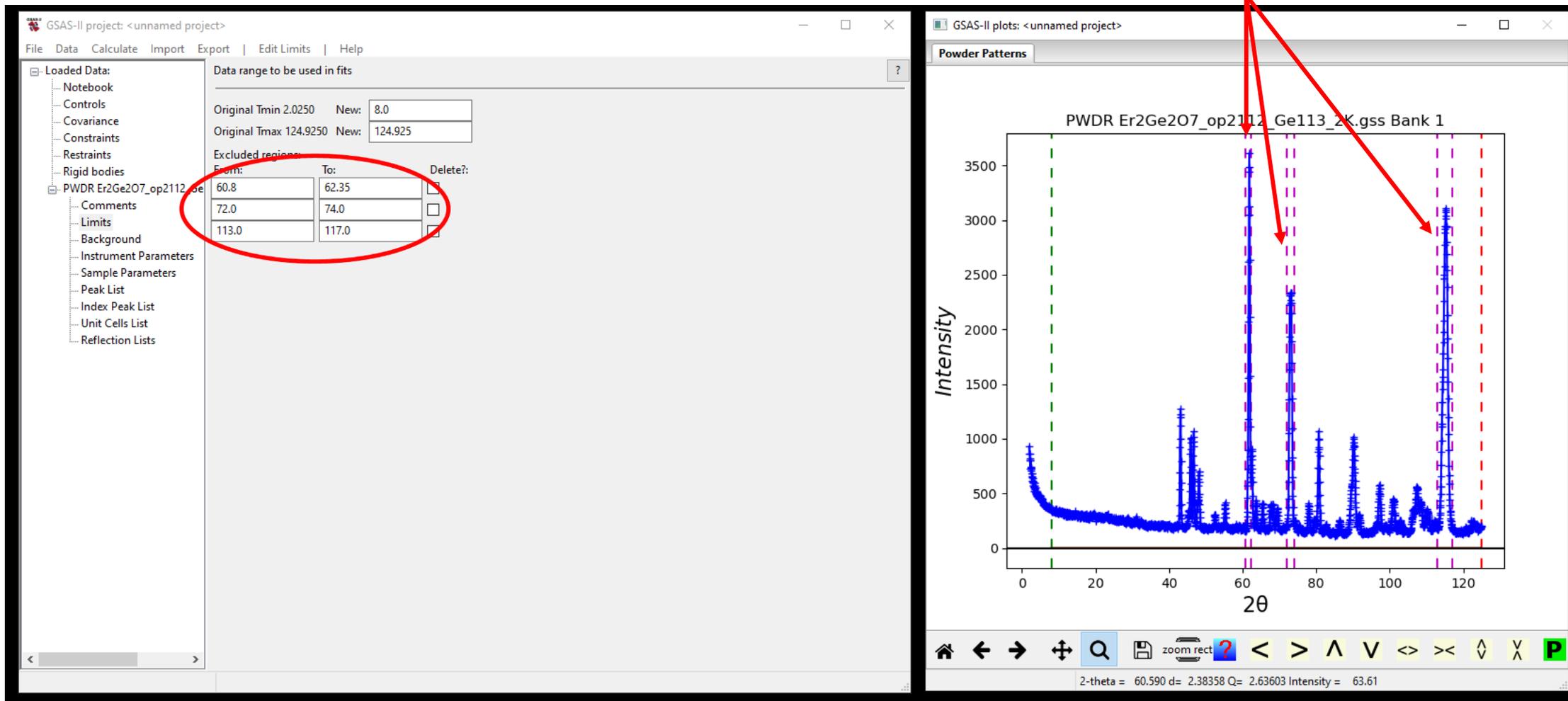
The screenshot shows the GSAS-II software interface. The main window displays the 'Data range to be used in fits' dialog box. The 'Original Tmin' is 2.0250 and the 'New' value is 8.0. The 'Original Tmax' is 124.9250 and the 'New' value is 124.925. The 'Excluded regions' section shows a range from 60.8 to 62.35, with a 'Delete?' checkbox. The 'Powder Patterns' window shows a plot of Intensity vs 2θ for 'PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1'. The plot shows a large peak at approximately $2\theta = 61.8$ and a smaller peak at approximately $2\theta = 62.5$. Two vertical dashed lines are drawn at $2\theta = 60.8$ and $2\theta = 62.35$, indicating the excluded regions.

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)

The image shows two windows from the GSAS-II software. The left window, titled 'GSAS-II project: <unnamed project>', has the 'Import' menu open. Under the 'Phase' sub-menu, the option 'from CIF file' is highlighted. Other options in the 'Phase' sub-menu include 'from GSAS .EXP file', 'from JANA m50 file', 'from PDB file', 'from ICDD .str file', 'from GSAS-II gpx file', 'from SHELX ins, res file', 'from RMCProfile .rnc6f file', 'from XYZ file', and 'guess format from file'. The right window, titled 'GSAS-II plots: <unnamed project>', displays a powder pattern plot. The plot is titled 'PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1'. The y-axis is labeled 'Intensity' and ranges from 0 to 3500. The x-axis is labeled '2θ' and ranges from 0 to 120. The plot shows a blue line representing the experimental data with several sharp peaks. Vertical dashed lines in green, purple, and red are overlaid on the plot, indicating reference peak positions. At the bottom of the plot window, there is a toolbar with various icons for navigation and analysis, and a status bar showing '2-theta = 60.590 d= 2.38358 Q= 2.63603 Intensity = 63.61'.

Go to Import → Phase and click on 'from CIF file'

Now we load our phase information (the cif file)

The screenshot displays the GSAS-II software interface. A file selection dialog titled "Choose phase input file(type CIF)" is open, showing the file "ErGeO_distbase" selected in the "Er2Ge207" folder. The file is a CIF File, dated 7/19/2017 11:18 AM, with a size of 2 KB. The file name "ErGeO_distbase" is entered in the "File name" field, and the file type is set to "CIF file (*.CIF;*.cif;*.mcif)". The "Open" button is highlighted.

In the background, the main GSAS-II window shows a diffraction pattern plot titled "2_Ge113_2K.gss Bank 1". The plot shows intensity versus 2θ (degrees), with a peak at approximately 113 degrees. The x-axis ranges from 80 to 120 degrees. The plot includes a blue line for the experimental data and a red dashed line for the reference pattern. The plot title is "2_Ge113_2K.gss Bank 1".

At the bottom of the plot, the following parameters are displayed: $2\theta = 60.590$, $d = 2.38358$, $Q = 2.63603$, Intensity = 63.61.

Choose 'ErGeO_distbase' (should be the only cif in the folder)

Now we load our phase information (the cif file)

The screenshot shows the GSAS-II software interface. The 'Import' menu is open, and 'Phase' is selected, leading to a sub-menu where 'from CIF file' is highlighted. A dialog box is displayed in the center, asking 'Is this the file you want?' and showing the file path 'File C:\Users\kct\Dropbox (ORNL)\Refinements\GSAS\GSASII\Er2Ge2O7\ErGeO_distbase.cif'. The dialog also shows the beginning of the CIF file content, including '#-----...' and '# CRYST'. A red arrow points to the 'Yes' button. In the background, a powder pattern plot is visible, titled 'PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1', showing intensity versus 2θ. The plot has a blue line for the experimental data and vertical dashed lines for reference peaks. The x-axis is labeled '2θ' and ranges from 0 to 120. The y-axis is labeled 'Intensity' and ranges from 0 to 3500. The status bar at the bottom right shows '2-theta = 60.590 d= 2.38358 Q= 2.63603 Intensity = 63.61'.

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)

The screenshot displays the GSAS-II software interface. On the left, the 'Import' menu is open, with 'Phase' selected. A sub-menu is visible, listing various file formats, with 'from CIF file' highlighted. In the foreground, an 'Edit phase name' dialog box is open, prompting the user to 'Enter the name for the new phase' with an empty text input field. A red arrow points from the text below to this input field. On the right, the 'Powder Patterns' window shows a plot titled 'PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1'. The plot shows a blue experimental data line with several sharp peaks, overlaid with vertical dashed lines representing reference peak positions. The x-axis is labeled '2θ' and ranges from 0 to 120. The y-axis represents intensity, ranging from 0 to 3500. At the bottom of the plot window, technical parameters are displayed: '2-theta = 60.590 d= 2.38358 Q= 2.63603 Intensity = 63.61'.

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

Now we load our phase information (the cif file)

The image displays two windows from the GSAS-II software. The left window, titled "GSAS-II project: <unnamed project>", shows the "General" tab for phase information. The phase name is "Er2Ge2O7", the phase type is "nuclear", and the space group is "P 41 21 2". The unit cell parameters are a = 6.77837, c = 12.32318, and Vol = 566.205. The density is 6.941. The elements are Er, Ge, and O. The isotope and natural abundance fields are empty. The number of atoms per cell is 1. The atom weight is 174.053. The bond radii and angle radii fields are empty. The van der Waals radii field is empty. The default color is blue. The Pawley controls are set to "Apply stride: 1". The Fourier map controls are empty. The map grid step is 10.0. The charge flip controls are empty. The map grid step is 10.0. The test HKLs are 0 0 2, 2 0 0, 1 1 1, 0 2 0, and 1 2 3. The Monte Carlo/Simulated Annealing controls are set to "Reflection set from: None" and "s-min: 2.8". The MC/SA runs are 1, and the MC/SA Refine at is 10.0. The MC/SA schedule is "log" with a slope of 0.9. The annealing schedule is "Start temp: 0.7", "Final temp: 0.1", and "No. trials: 250".

The right window, titled "GSAS-II plots: <unnamed project>", shows a "Powder Patterns" plot. The plot is titled "PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1". The y-axis is "Intensity" ranging from 0 to 3500. The x-axis is "2θ" ranging from 0 to 120. The plot shows a blue line representing the experimental data and a red line representing the calculated pattern. The plot is overlaid with a grid of vertical lines. A red arrow points from the "Add histogram(s)" dialog box in the left window to the "0) PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1" entry in the dialog box.

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)

The image displays two windows from the GSAS-II software. The left window, titled 'GSAS-II project: <unnamed project>', shows the 'General' tab for phase 'Er2Ge2O7'. The 'Add histogram(s)' dialog box is open, with the entry '0) PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1' selected and circled in red. A red arrow points from this entry to the 'OK' button. The right window, titled 'GSAS-II plots: <unnamed project>', shows a 'Powder Patterns' plot for 'PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1'. The plot shows Intensity on the y-axis (0 to 3500) versus 2θ on the x-axis (0 to 120). The plot features a blue experimental data line, a red dashed line representing the fit, and several vertical dashed lines in green, purple, and red representing reference peak positions.

Check the histogram and click 'OK'

Now we're getting somewhere...

GSAS-II project: <unnamed project>

File Data Calculate Import Export | Select tab Compute | Help

General Data Atoms Draw Options Draw Atoms RB Models Map peaks MC/SA RMC ISODISTORT Textur

Phase name: Er₂Ge₂O₇ Phase type: nuclear Space group: P 41 21 2 Modulated?

a = 6.77837 c = 12.32318 Vol = 566.205

Density: 6.941

Elements	Er	Ge	O
Isotope	Nat. Abund.	Nat. Abund.	Nat. Abund.
No. per cell	8.0	8.0	28.0
Atom weight	167.260	72.590	15.999
Bond radii	1.96	1.57	1.09
Angle radii	1.76	1.37	0.89
van der Waals radii	1.70	1.70	1.70
Default color			

Pawley controls: no data

Fourier map controls: Map type: Reflection sets: Select reflection sets

Map grid step: 0.25 Peak cutoff %: 50.0

Charge flip controls: Reflection sets: Select reflection sets Normalizing element: None

Map grid step: 0.25 k-Factor (0.1-1.2): 0.1 k-Max (>=10.0): 20.0

Test HKLs: 0 0 2 2 0 0 1 1 1 0 2 0 1 2 3

Monte Carlo/Simulated Annealing controls: Reflection set from: d-min: 2.8

MC/SA runs: 1 MC/SA Refine at 10.0 % of ranges.

MC/SA schedule: log slope: 0.9

Annealing schedule: Start temp: 0.7 Final temp: 0.1 No. trials: 250

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

Now we're getting somewhere...

GSAS-II project: <unnamed project>

File Data Calculate Import Export | Select tab Compute | Help

General Data Atoms Draw Options Draw Atoms RB Models Map peaks MC/SA RMC ISODISTORT Textur

Phase name: Er₂Ge₂O₇ Phase type: nuclear Space group: P 41 21 2 Modulated?

a = 6.77837 c = 12.32318 Vol = 566.205

Density: 6.941

Elements	Er	Ge	O
Isotope	Nat. Abund.	Nat. Abund.	Nat. Abund.
No. per cell	8.0	8.0	28.0
Atom weight	167.260	72.590	15.999
Bond radii	1.96	1.57	1.09
Angle radii	1.76	1.37	0.89
van der Waals radii	1.70	1.70	1.70
Default color			

Pawley controls: no data

Fourier map controls: Map type: Reflection sets: Select reflection sets

Map grid step: 0.25 Peak cutoff %: 50.0

Charge flip controls: Reflection sets: Select reflection sets Normalizing element: None

Map grid step: 0.25 k-Factor (0.1-1.2): 0.1 k-Max (>=10.0): 20.0

Test HKLs: 0 0 2 2 0 0 1 1 1 0 2 0 1 2 3

Monte Carlo/Simulated Annealing controls: Reflection set from: d-min: 2.8

MC/SA runs: 1 MC/SA Refine at 10.0 % of ranges.

MC/SA schedule: log slope: 0.9

Annealing schedule: Start temp: 0.7 Final temp: 0.1 No. trials: 250

Unit cell information

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

Now we're getting somewhere...

GSAS-II project: <unnamed project>

File Data Calculate Import Export | Select tab Compute | Help

General Data Atoms Draw Options Draw Atoms RB Models Map peaks MC/SA RMC ISODISTORT Textur

Phase name: Er2Ge2O7 Phase type: nuclear Space group: P 41 21 2 Modulated?

a = 6.77837 c = 12.32318 Vol = 566.205

Density: 6.941

Elements	Er	Ge	O
Isotope	Nat. Abund.	Nat. Abund.	Nat. Abund.
No. per cell	8.0	8.0	28.0
Atom weight	167.260	72.590	15.999
Bond radii	1.96	1.57	1.09
Angle radii	1.76	1.37	0.89
van der Waals radii	1.70	1.70	1.70
Default color			

Pawley controls: no data

Fourier map controls: Map type: Reflection sets: Select reflection sets

Map grid step: 0.25 Peak cutoff %: 50.0

Charge flip controls: Reflection sets: Select reflection sets Normalizing element: None

Map grid step: 0.25 k-Factor (0.1-1.0): 0.1 k-Max (>=10.0): 20.0

Test HKLs: 0 0 2 2 0 0 1 1 1 0 2 0 1 2 3

Monte Carlo/Simulated Annealing controls: Reflection set from: d-min: 2.8

MC/SA runs: 1 MC/SA Refine at 10.0 % of ranges.

MC/SA schedule: log slope: 0.9

Annealing schedule: Start temp: 0.7 Final temp: 0.1 No. trials: 250

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

Now we're getting somewhere...

GSAS-II project: <unnamed project>

File Data Calculate Import Export Select tab Compute Help

General Data Atoms Draw Options Draw Atoms RB Models Map peaks MC/SA RMC ISODISTORT Textur

Phase name: Er2Ge2O7 Phase type: nuclear Space group: P 41 21 2 Modulated?

a = 6.77837 c = 12.32318 Vol = 566.205

Density: 6.941

Elements	Er	Ge	O
Isotope	Nat. Abund.	Nat. Abund.	Nat. Abund.
No. per cell	8.0	8.0	28.0
Atom weight	167.260	72.590	15.999
Bond radii	1.96	1.57	1.09
Angle radii	1.76	1.37	0.89
van der Waals radii	1.70	1.70	1.70
Default color	Green	Grey	Red

Pawley controls: no data

Fourier map controls: Map type: Reflection sets: Select reflection sets

Map grid step: 0.25 Peak cutoff %: 50.0

Charge flip controls: Reflection sets: Select reflection sets Normalizing element: None

Map grid step: 0.25 k-Factor (0.1-1.2): 0.1 k-Max (>=10.0): 20.0

Test HKLs: 0 0 2 2 0 0 1 1 1 0 2 0 1 2 3

Monte Carlo/Simulated Annealing controls: Reflection set from: d-min: 2.8

MC/SA runs: 1 MC/SA Refine at: 10.0 % of ranges.

MC/SA schedule: log slope: 0.9

Annealing schedule: Start temp: 0.7 Final temp: 0.1 No. trials: 250

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

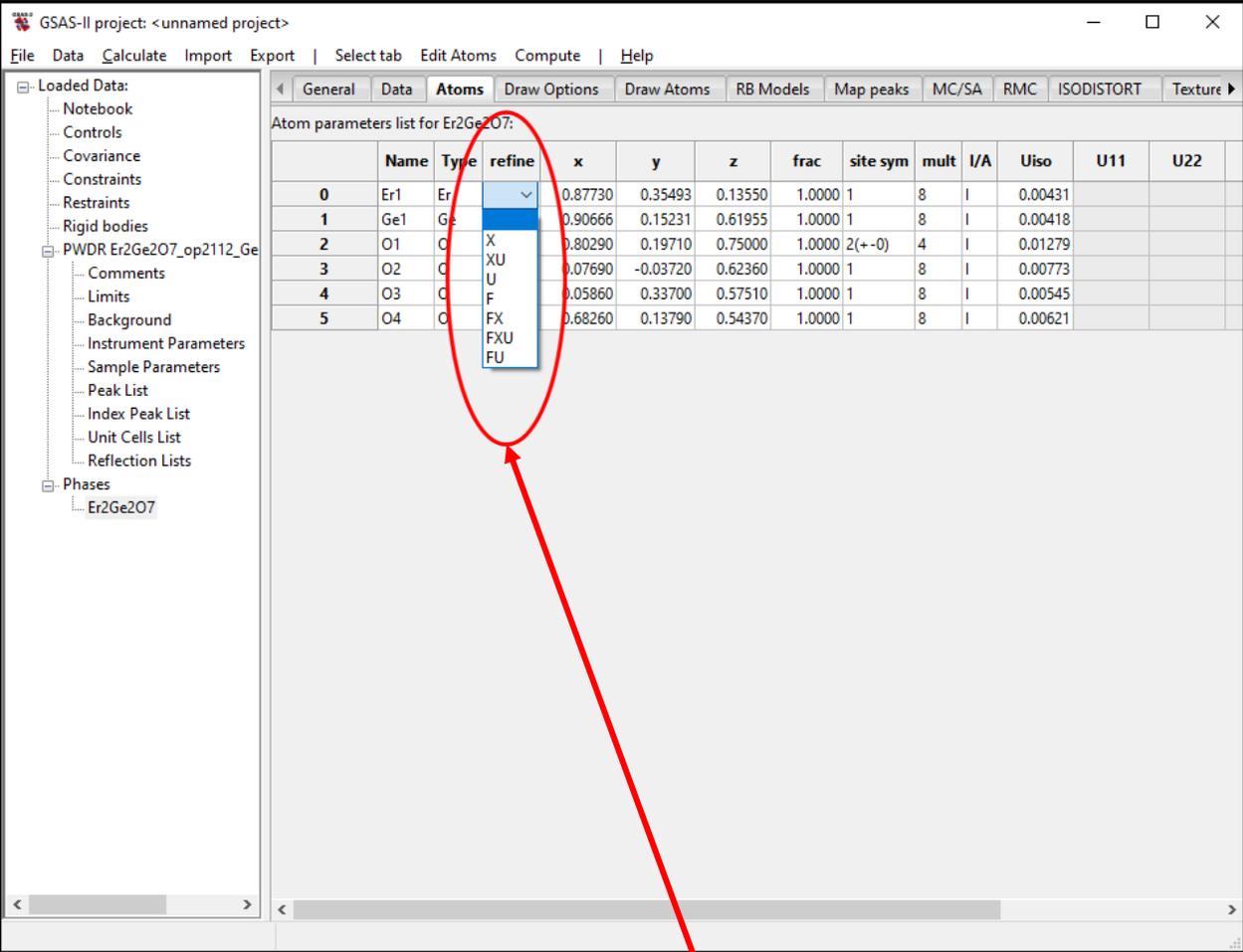
Using the tabs

The screenshot shows the GSAS-II interface with the 'Atoms' tab selected. The table displays the following data:

	Name	Type	refine	x	y	z	frac	site sym	mult	I/A	Uiso	U11	U22
0	Er1	Er		0.87730	0.35493	0.13550	1.0000	1	8	I	0.00431		
1	Ge1	Ge		0.90666	0.15231	0.61955	1.0000	1	8	I	0.00418		
2	O1	O		0.80290	0.19710	0.75000	1.0000	2(+ -0)	4	I	0.01279		
3	O2	O		0.07690	-0.03720	0.62360	1.0000	1	8	I	0.00773		
4	O3	O		0.05860	0.33700	0.57510	1.0000	1	8	I	0.00545		
5	O4	O		0.68260	0.13790	0.54370	1.0000	1	8	I	0.00621		

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



The screenshot shows the GSAS-II interface with the 'Atoms' tab selected. A table of atom parameters is displayed, and a pull-down menu is open over the 'refine' column. A red circle highlights the menu, and a red arrow points to the 'refine' column header.

	Name	Type	refine	x	y	z	frac	site sym	mult	I/A	Uiso	U11	U22
0	Er1	Er	▼	0.87730	0.35493	0.13550	1.0000	1	8	I	0.00431		
1	Ge1	Ge		0.90666	0.15231	0.61955	1.0000	1	8	I	0.00418		
2	O1	O	X	0.80290	0.19710	0.75000	1.0000	2(+ -0)	4	I	0.01279		
3	O2	O	XU	0.07690	-0.03720	0.62360	1.0000	1	8	I	0.00773		
4	O3	O	U	0.05860	0.33700	0.57510	1.0000	1	8	I	0.00545		
5	O4	O	F	0.68260	0.13790	0.54370	1.0000	1	8	I	0.00621		

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

Using the tabs

The image shows two windows from the GSAS-II software. The left window, titled 'GSAS-II project: <unnamed project>', has a menu bar with 'File', 'Data', 'Calculate', 'Import', 'Export', 'Select tab', and 'Help'. Below the menu is a tabbed interface with tabs for 'General', 'Data', 'Atoms', 'Draw Options', 'Draw Atoms', 'RB Models', 'Map peaks', 'MC/SA', 'RMC', 'ISODISTORT', and 'Texture'. The 'Draw Options' tab is currently selected and circled in red. A red arrow points from this tab to the 'Er2Ge207' tab in the right window. The 'Draw Options' panel contains various controls for drawing, including sliders for 'Camera Distance, Å' (50.0), 'Z clipping, Å' (25.0), 'Z step, Å' (0.5), 'van der Waals scale' (0.5), 'Ellipsoid probability, %' (50.0), 'Ball scale' (0.33), 'Bond radius, Å' (0.1), and 'Bond search factor' (0.85). It also has checkboxes for 'Show unit cell?', 'Show hydrogens?', 'Show Rigid Bodies?', 'Fade sym equivs?', and 'Show void map?'. The right window, titled 'GSAS-II plots: <unnamed project>', has a 'Powder Patterns' tab and a 'Er2Ge207' tab, both circled in red. The 'Er2Ge207' tab displays a 3D ball-and-stick model of the crystal structure of Er₂Ge₂O₇ within a unit cell. The atoms are represented by spheres of different colors (red, green, blue, grey) and are connected by lines. A red crosshair is visible in the center of the unit cell. The status bar at the bottom of the right window shows 'View point: 0.5000, 0.5000, 0.5000; density: 0.0000'.

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

The screenshot displays the GSAS-II software interface. The left pane shows the project tree with 'Er2Ge2O7' selected under 'Phases'. The main window is in the 'Draw Atoms' tab, showing a table of atom coordinates and styles. The 'Draw Atom list for Er2Ge2O7:' table is as follows:

	Name	Type	x	y	z	Sym Op	Style	Label	Color	I/A
0	Er1	Er	0.87730	0.35493	0.13550	1	vdW balls			
1	Ge1	Ge	0.90666	0.15231	0.61955	1	vdW balls			
2	O1	O	0.80290	0.19710	0.75000	1	vdW balls			
3	O2	O	0.07690	-0.03720	0.62360	1	vdW balls			
4	O3	O	0.05860	0.33700	0.57510	1	vdW balls			
5	O4	O	0.68260	0.13790	0.54370	1	vdW balls			

The right pane shows a 3D visualization of the structure with atoms represented as green spheres. The status bar at the bottom indicates 'View point: 0.5000, 0.5000, 0.5000; density: 0.0000'.

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

Visualizing the structure

The screenshot displays the GSAS-II interface. The 'Edit Figure' menu is open, with 'Fill unit cell' highlighted. A red arrow points from this menu item to the 3D visualization window on the right, which shows the unit cell of Er₂Ge₂O₇ with atoms represented as green spheres.

Sym Op	Style	Label	Color	I/A
550 1	vdW balls			I
955 1	vdW balls			I
000 1	vdW balls			I
360 1	vdW balls			I
510 1	vdW balls			I
370 1	vdW balls			I

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

GSAS-II project: <unnamed project>

File Data Calculate Import Export | Select tab Edit Figure Compute Restraints Rigid body | 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
Phases
Er2Ge2O7

Draw Atom list for Er2Ge2O7:

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

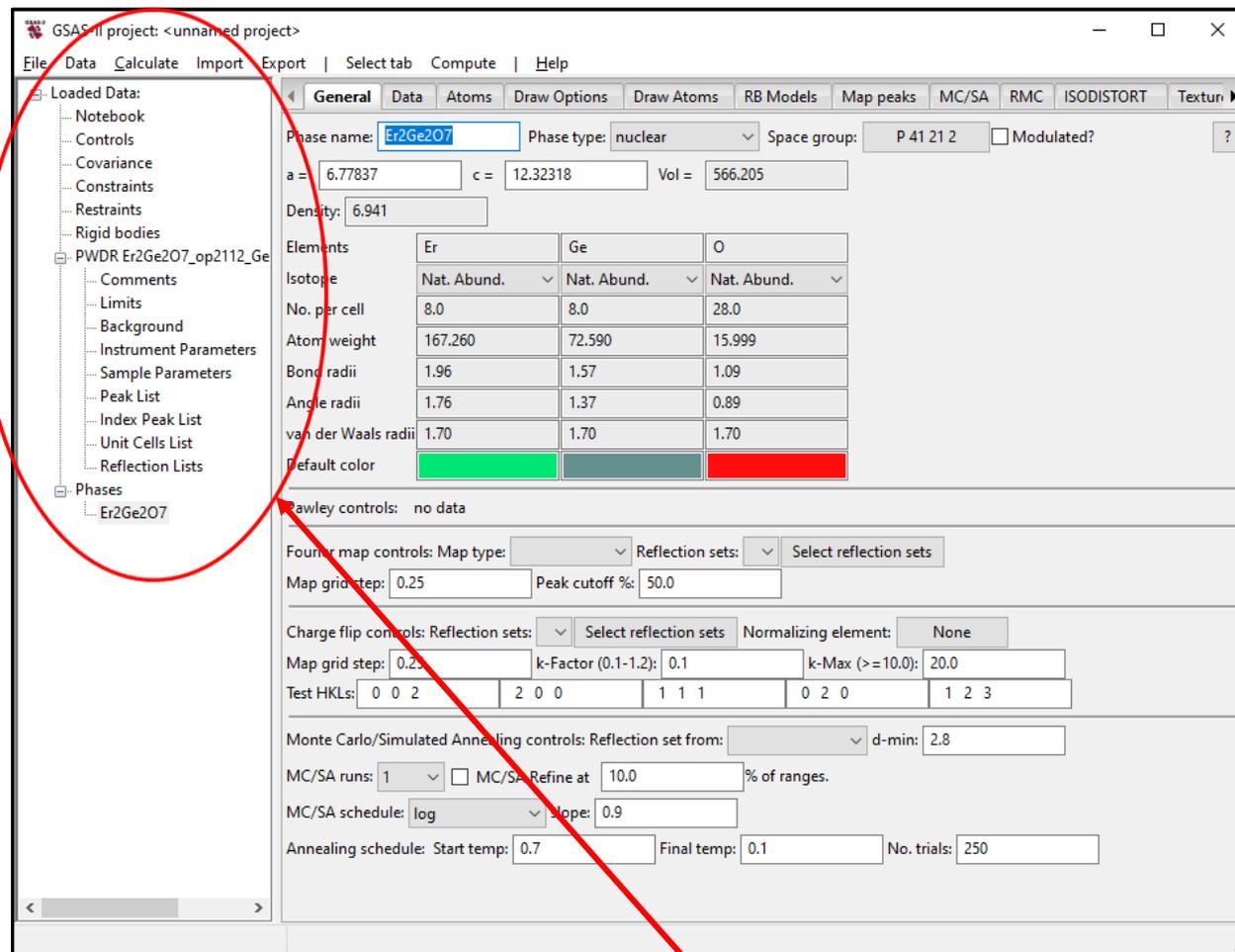
GSAS-II plots: <unnamed project>

Powder Patterns Er2Ge2O7

save as/key: New quaternion: -0.76+, 0.64i+, -0.05j+, -0.05k

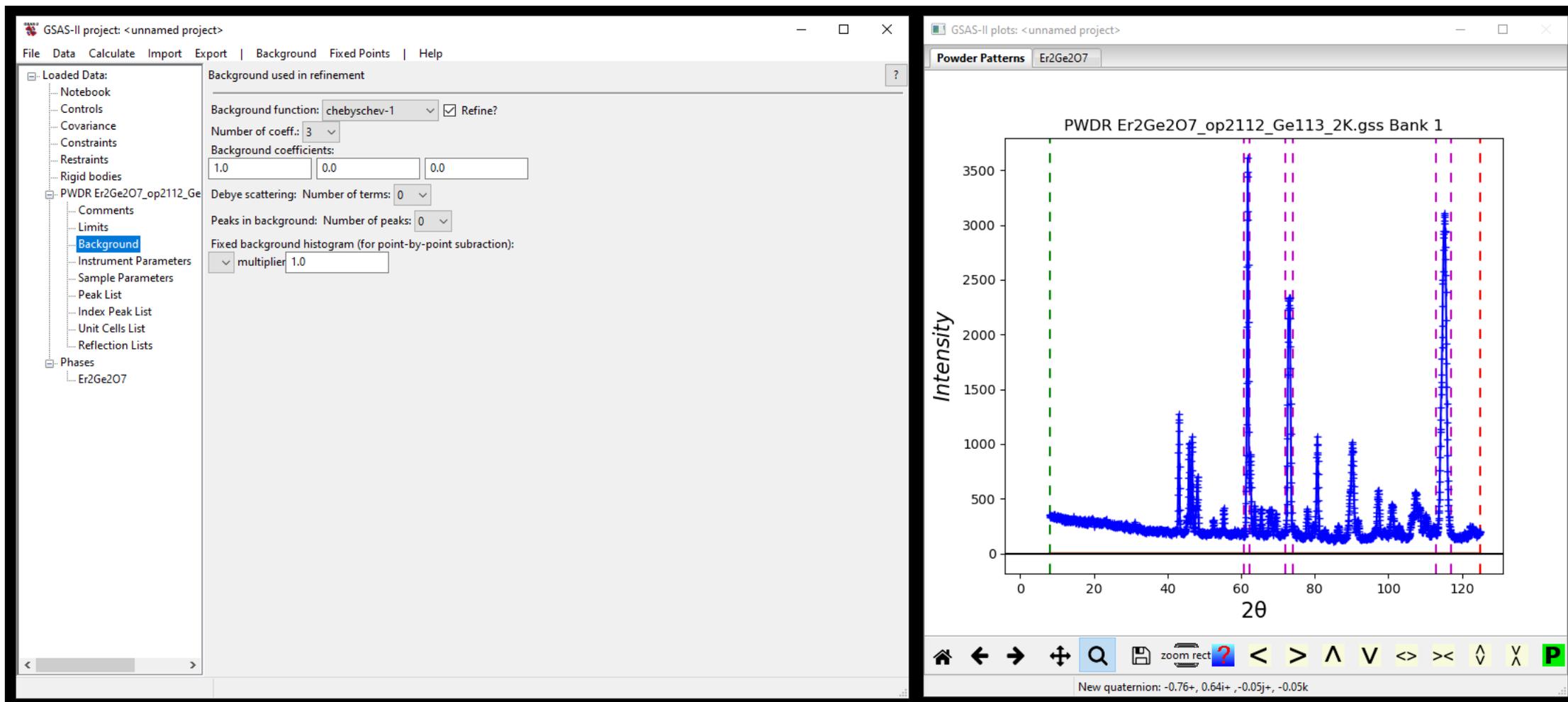
Now all the atoms in the unit cell show
(I've rotated this by clicking and dragging in the window)

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 histogram 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

The image displays two windows from the GSAS-II software. The left window, titled "GSAS-II project: <unnamed project>", shows the "Calculate" menu with "Refine" selected. The "Refinement" panel is visible, showing "chebyshev-1" as the profile function, a checked "Refine?" box, and "Number of terms: 0" and "Number of peaks: 0". The left sidebar shows a tree view with "Phases" > "Er2Ge2O7" selected. The right window, titled "GSAS-II plots: <unnamed project>", shows a "Powder Patterns" plot for "Er2Ge2O7". The plot is titled "PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1" and shows Intensity vs 2θ. The y-axis ranges from 0 to 3500, and the x-axis ranges from 0 to 120. The plot shows a blue line representing the experimental data with several sharp peaks. Vertical dashed lines in green, purple, and red are overlaid on the plot, indicating reference peak positions. The bottom toolbar contains various icons for navigation and analysis, and the status bar at the bottom displays "New quaternion: -0.76+, 0.64i+, -0.05j+, -0.05k".

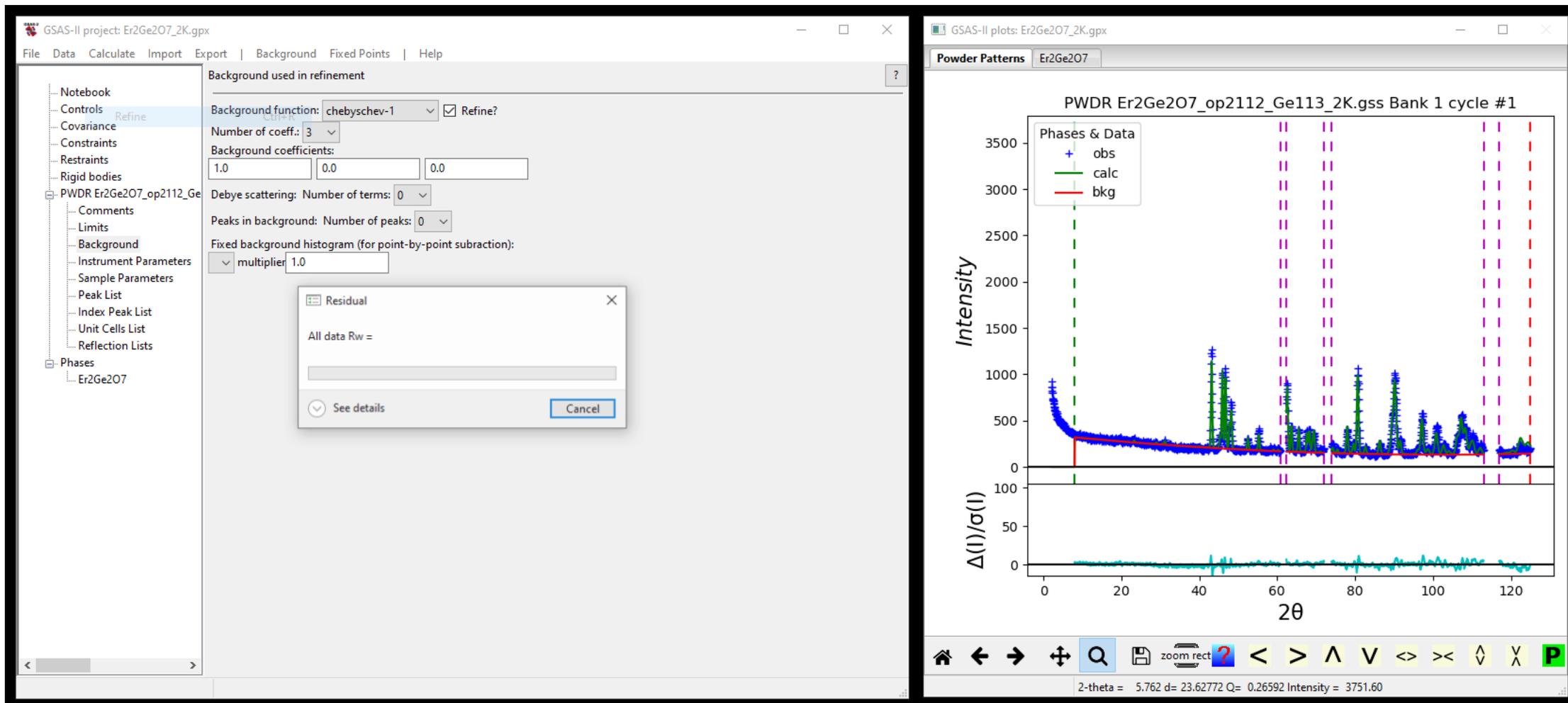
Go to Calculate -> Refine

Let's start refining

The screenshot displays the GSAS-II software interface. On the left, a tree view shows the project structure under 'Loaded Data', with 'Background' selected. The main window is a 'Choose GSAS-II project file name' dialog box. The dialog shows the current location as 'This PC > Desktop'. A search bar contains 'Search Desktop'. Below the search bar, a list of folders and files is shown, including 'Documents', 'Pictures', 'Er2Ge207', 'KerSe2', 'KNiAsO4', 'Re2Be2SiO7_Bra:', 'Dropbox (ORNL)', and 'OneDrive - Oak Ri'. The 'File name' field is empty, and the 'Save as type' is set to 'GSAS-II project file (*.gpx)'. The 'Save' button is highlighted. To the right of the dialog, a plot titled '2_Ge113_2K.gss Bank 1' shows a diffraction pattern with a blue line representing the data and vertical dashed lines representing peak positions. The x-axis is labeled θ and ranges from 80 to 120. At the bottom of the plot, the text 'New quaternion: -0.76+, 0.64i+, -0.05j+, -0.05k' is visible.

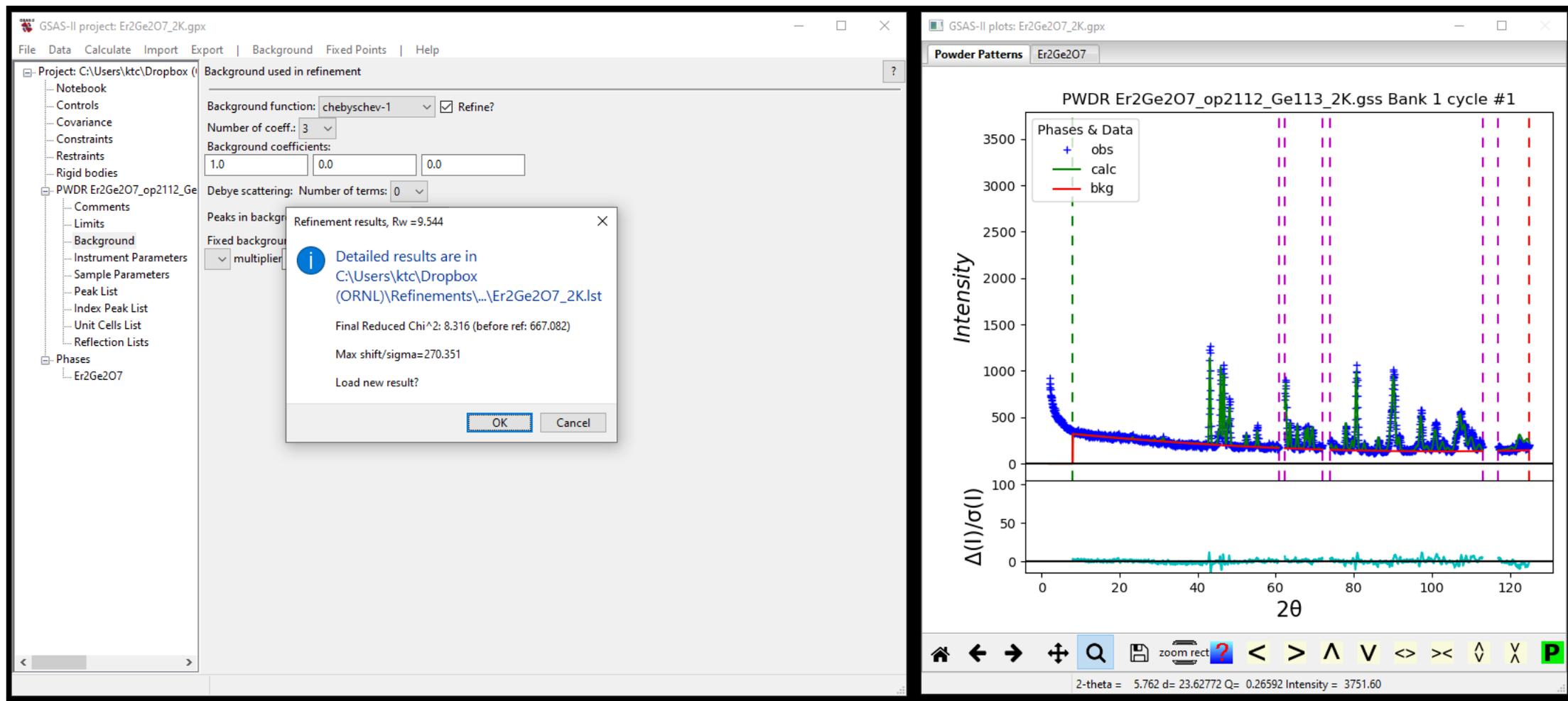
Open, need to save it first (make sure to do so in the same location as the rest of the files). I'll name it Er2Ge207_2K.

Running a refinement



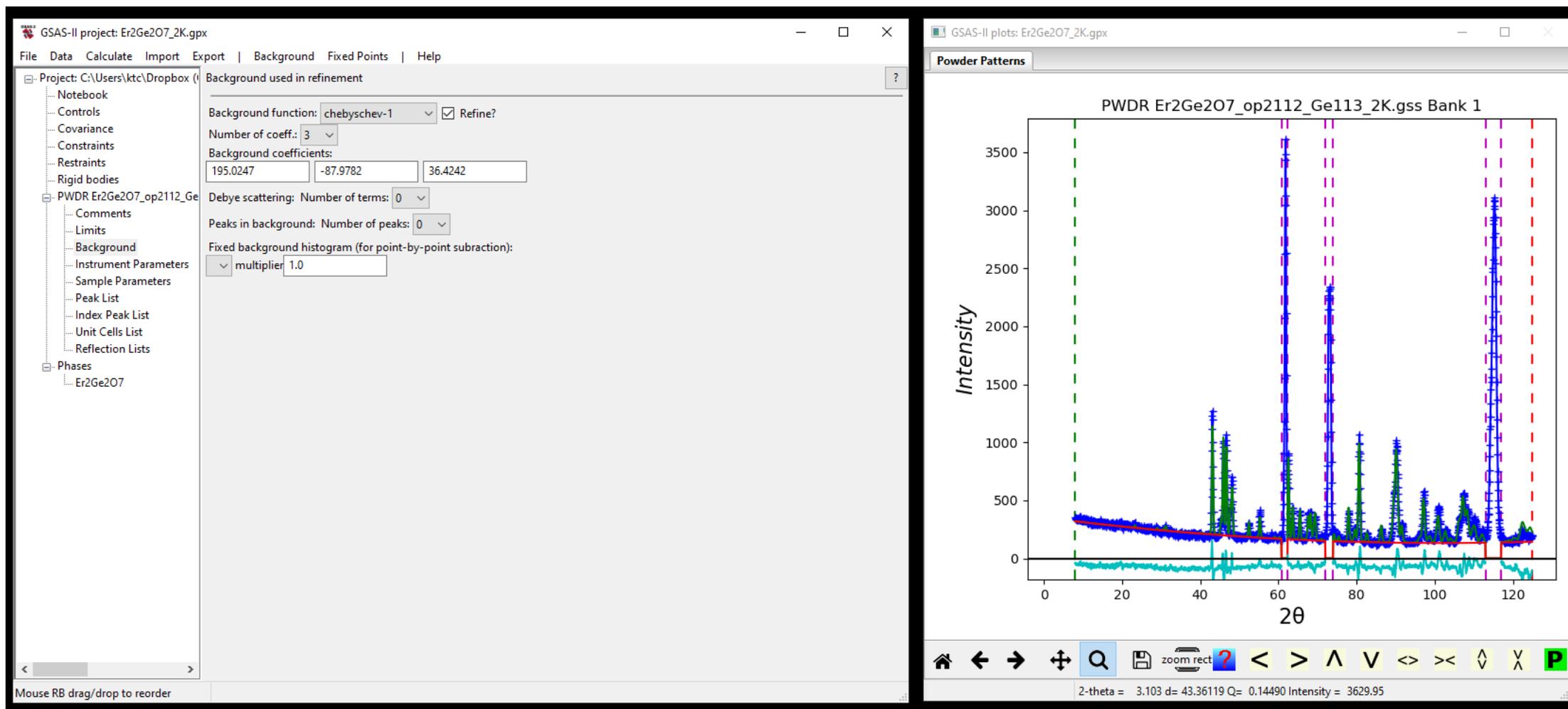
Should get something like this, which will go through a couple cycles...

Running a refinement



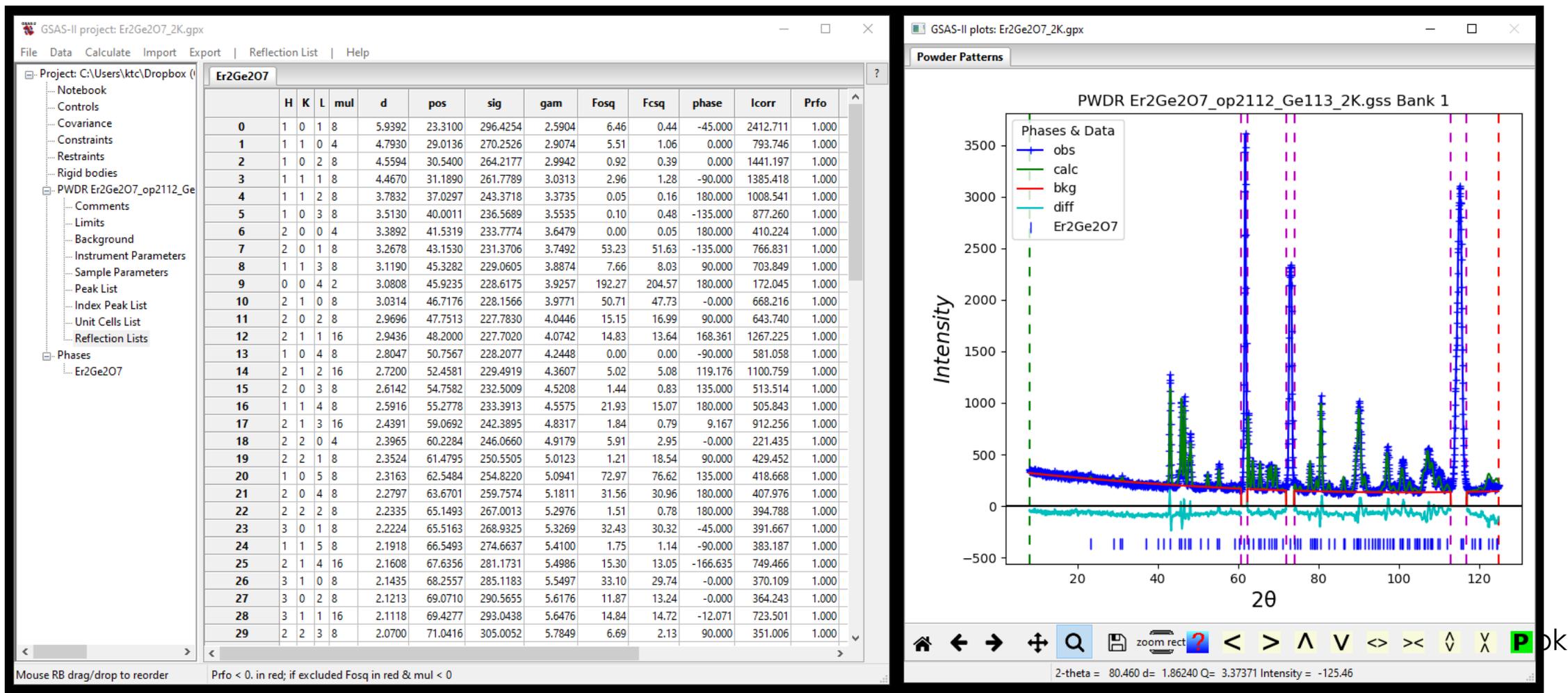
And then it will ask whether you want to load the refinement results
– click 'OK'

Running a refinement



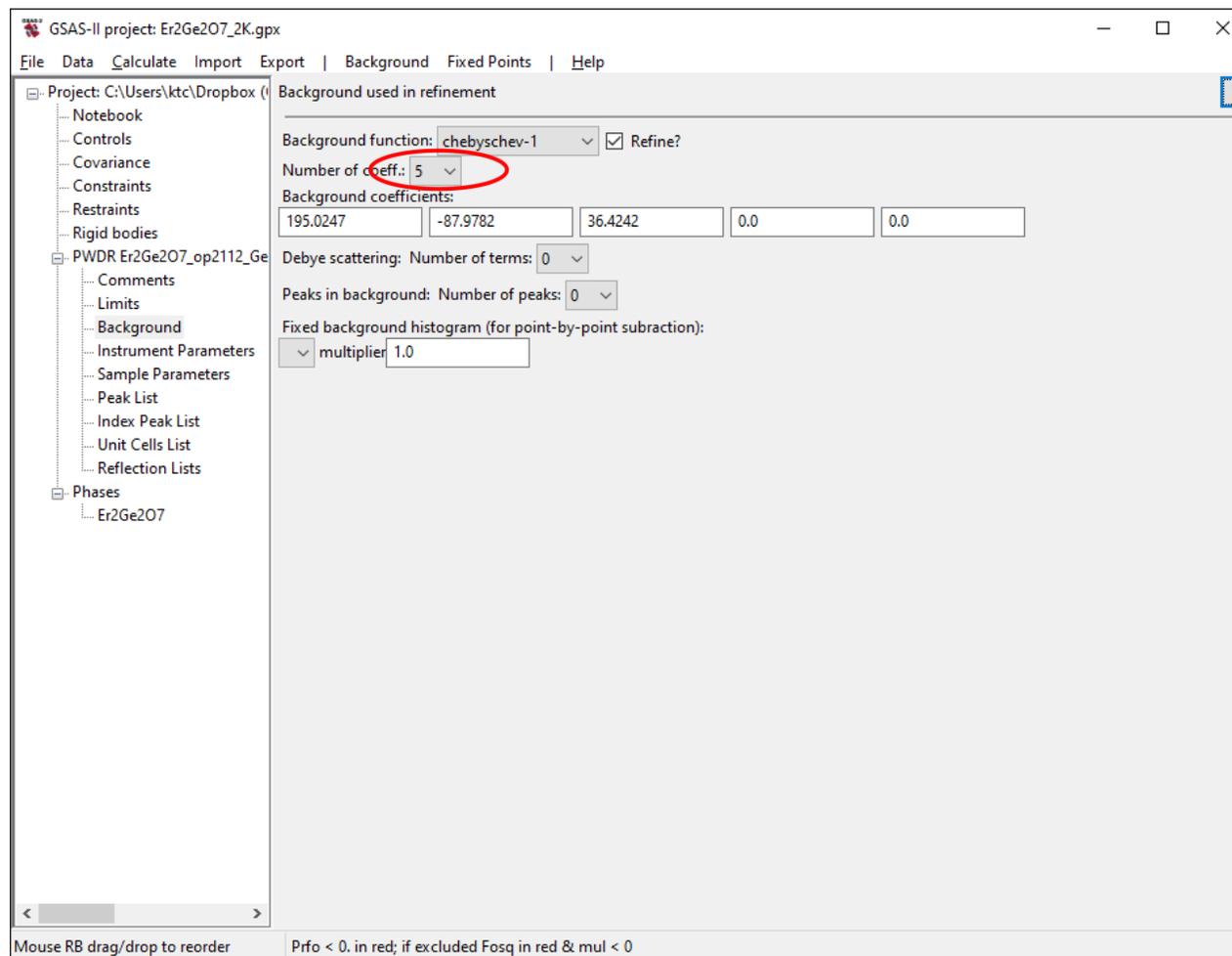
We can see the peaks are roughly in the right positions, but their intensities and the background is off – we need to add more background terms and flag some structural parameters to refine

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

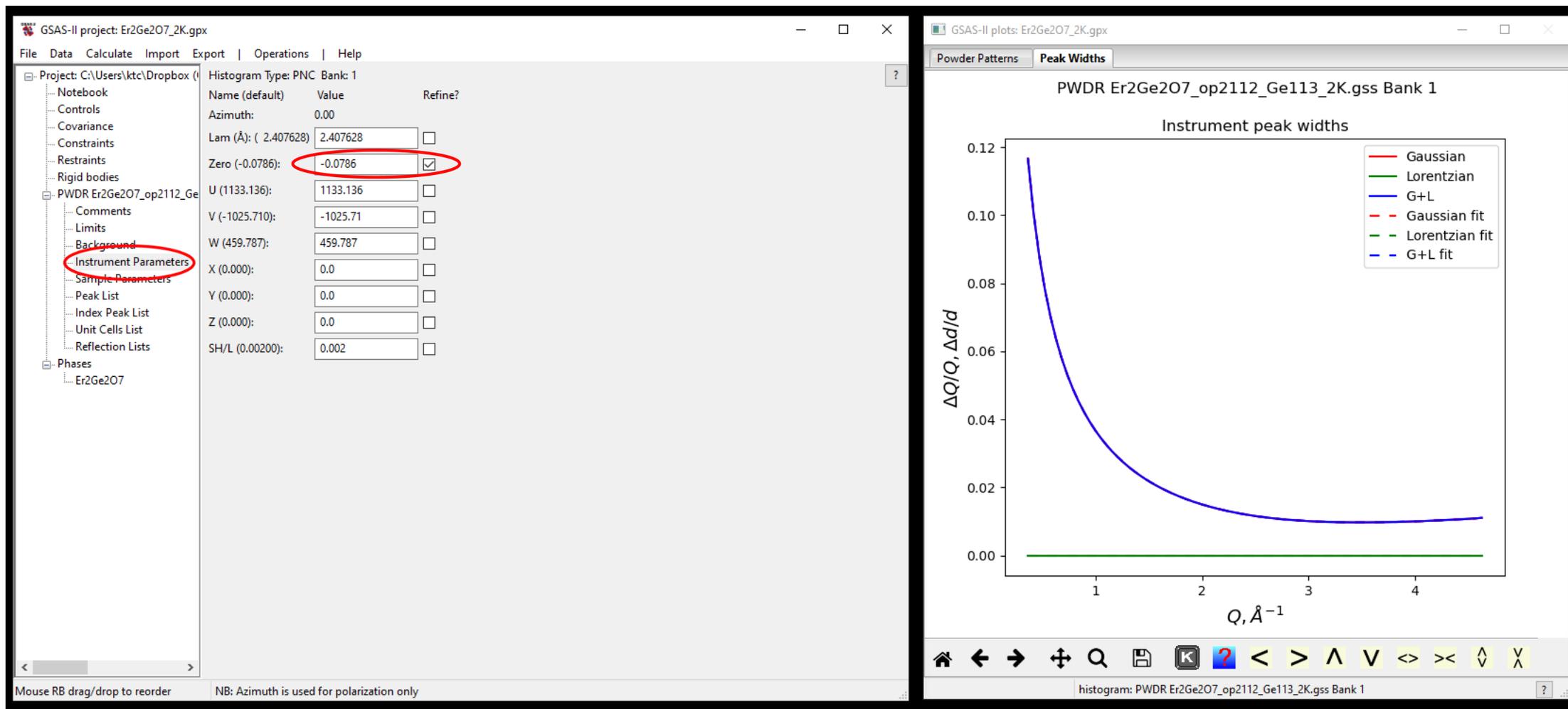
The screenshot displays the GSAS-II interface. On the left, the 'Phases' tree shows 'Er2Ge2O7' selected. The main window shows the 'General' tab for this phase. The 'Refine unit cell' checkbox is checked, and the unit cell parameters are: $a = 6.77837$, $c = 12.32318$, and $\text{Vol} = 566.205$. The 'Elements' table is as follows:

Elements	Er	Ge	O
Isotope	Nat. Abund.	Nat. Abund.	Nat. Abund.
No. per cell	8.0	8.0	28.0
Atom weight	167.260	72.590	15.999
Bond radii	1.96	1.57	1.09
Angle radii	1.76	1.37	0.89
van der Waals radii	1.70	1.70	1.70

The 'Powder Patterns' window on the right shows the plot 'PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1'. The y-axis is 'Intensity' (ranging from -500 to 3500) and the x-axis is '2θ' (ranging from 20 to 120). The plot shows experimental data points (blue) and a fitted curve (red). Vertical dashed lines in green, purple, and red indicate peak positions.

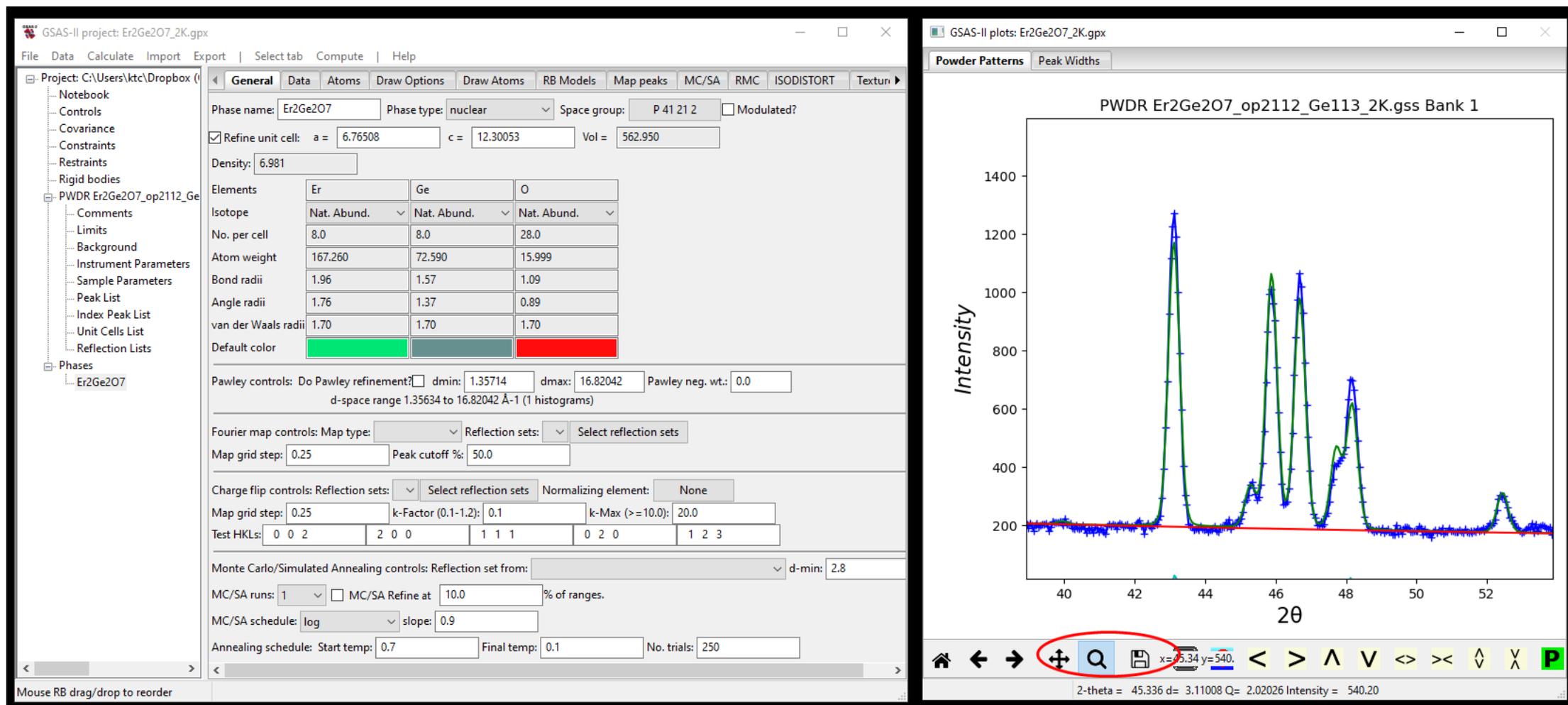
Looking better, now let's add the lattice parameters
Go to the phase node and click 'Refine unit cell'

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

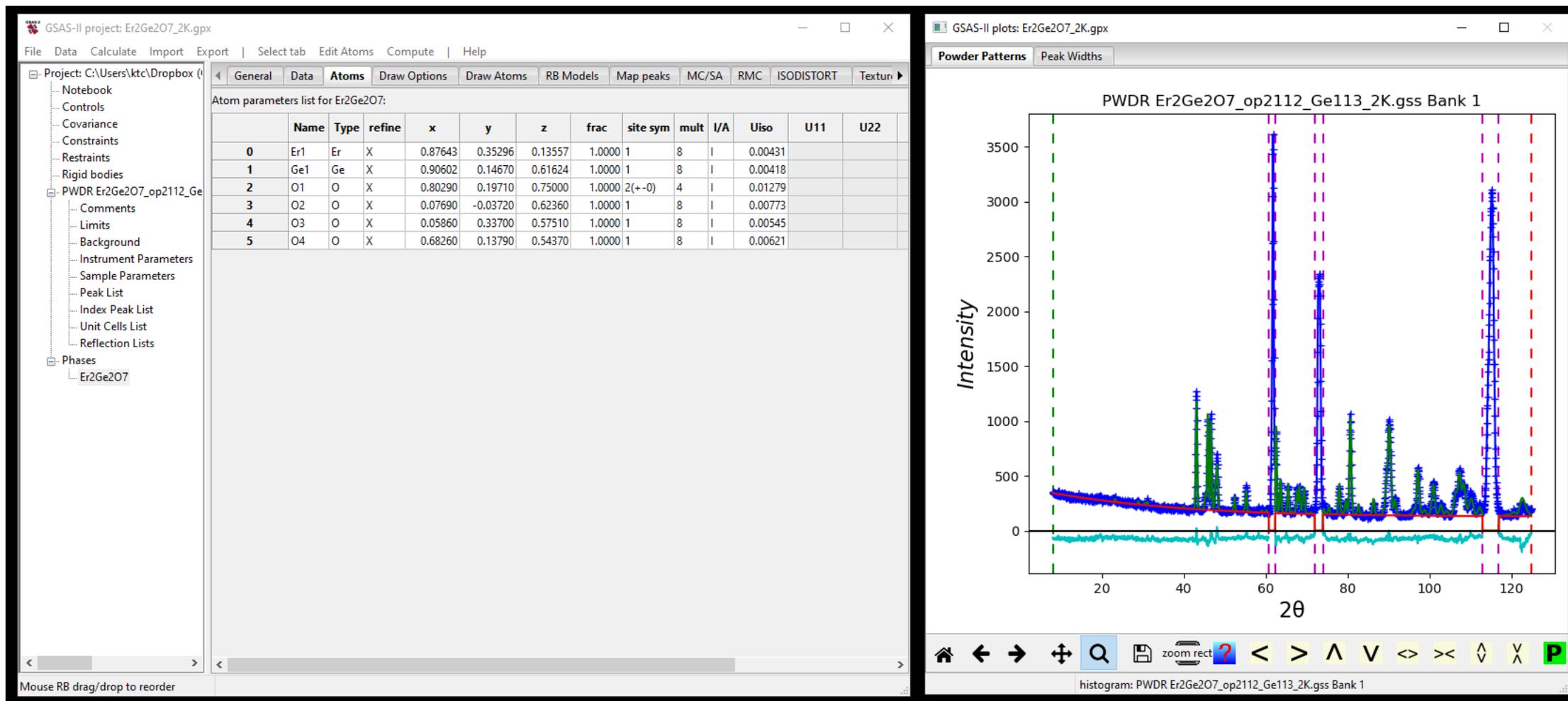
Optimizing the fit – internal parameters

The screenshot displays the GSAS-II interface. The left pane shows the project tree with 'Er2Ge2O7' selected under 'Phases'. The main window is the 'Atoms' tab, showing the 'Atom parameters list for Er2Ge2O7' table. The 'refine' column for the Ge1 site is highlighted. The right pane shows a 3D ball-and-stick model of the crystal structure with atoms colored by element (red for Er, green for Ge, grey for O).

	Name	Type	refine	x	y	z	frac	site sym	mult	I/A	Uiso	U11	U22
0	Er1	Er	X	0.87730	0.35493	0.13550	1.0000	1	8	1	0.00431		
1	Ge1	Ge	X	0.90666	0.15231	0.61955	1.0000	1	8	1	0.00418		
2	O1	O		0.80290	0.19710	0.75000	1.0000	2(+ -0)	4	1	0.01279		
3	O2	O		0.07690	-0.03720	0.62360	1.0000	1	8	1	0.00773		
4	O3	O		0.05860	0.33700	0.57510	1.0000	1	8	1	0.00545		
5	O4	O		0.68260	0.13790	0.54370	1.0000	1	8	1	0.00621		

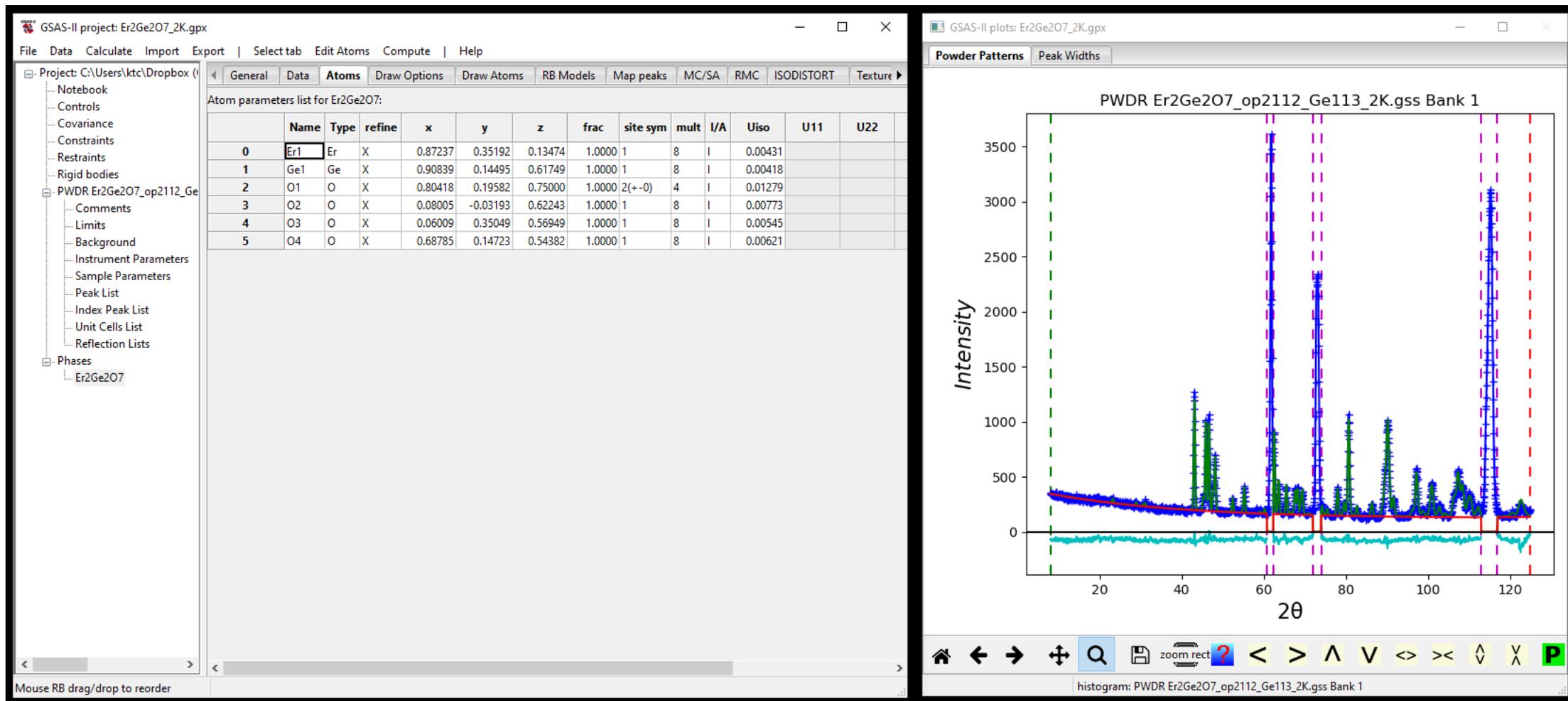
Now let's add the atomic positions and displacement parameters that were in the Atoms tab
NB: This should be done in small groups of atoms usually starting with the highest multiplicity and scattering strength to keep the refinement stable

Optimizing the fit – internal parameters



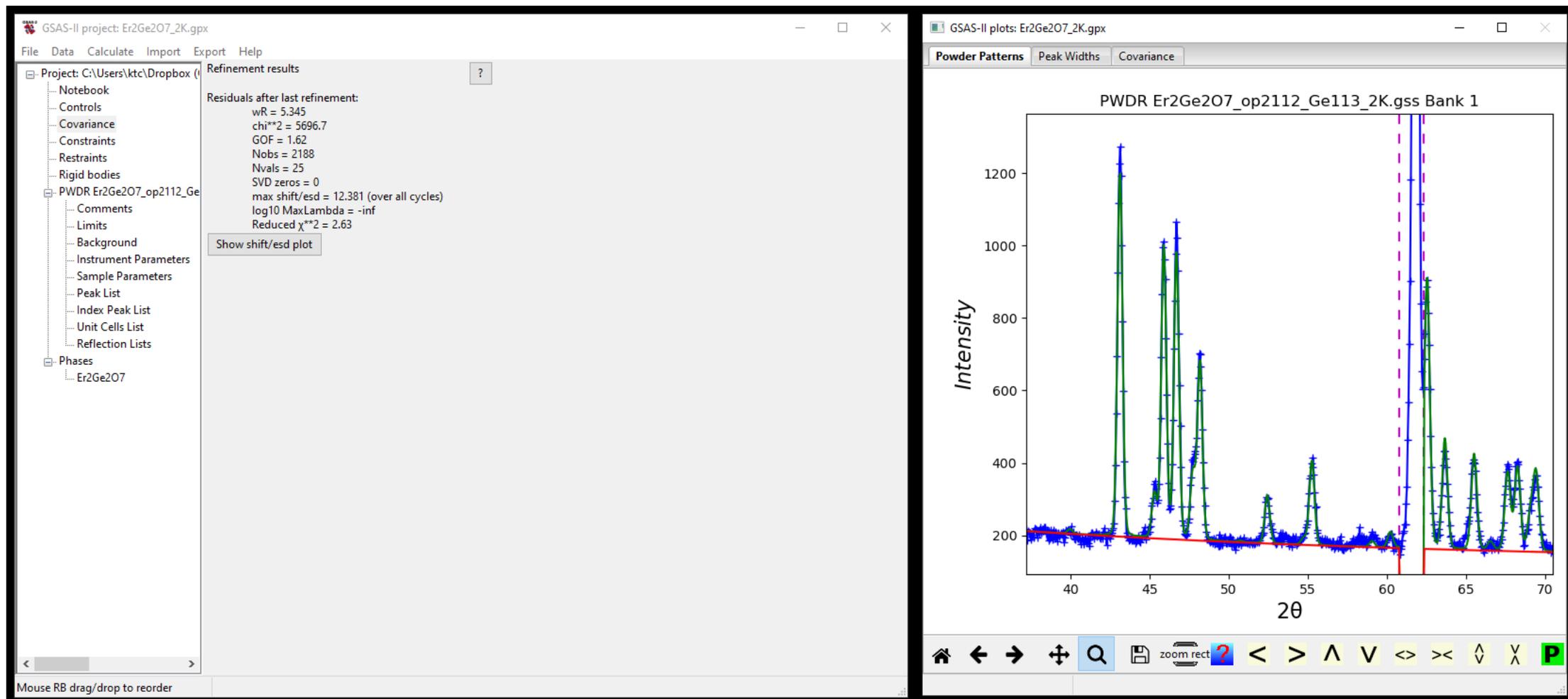
A little better, let's add the rest of the positions (we will not refine the Uiso parameters as this is very low temperature and we don't have very high in Q)

Optimizing the fit



Another modest improvement, lets take a closer look

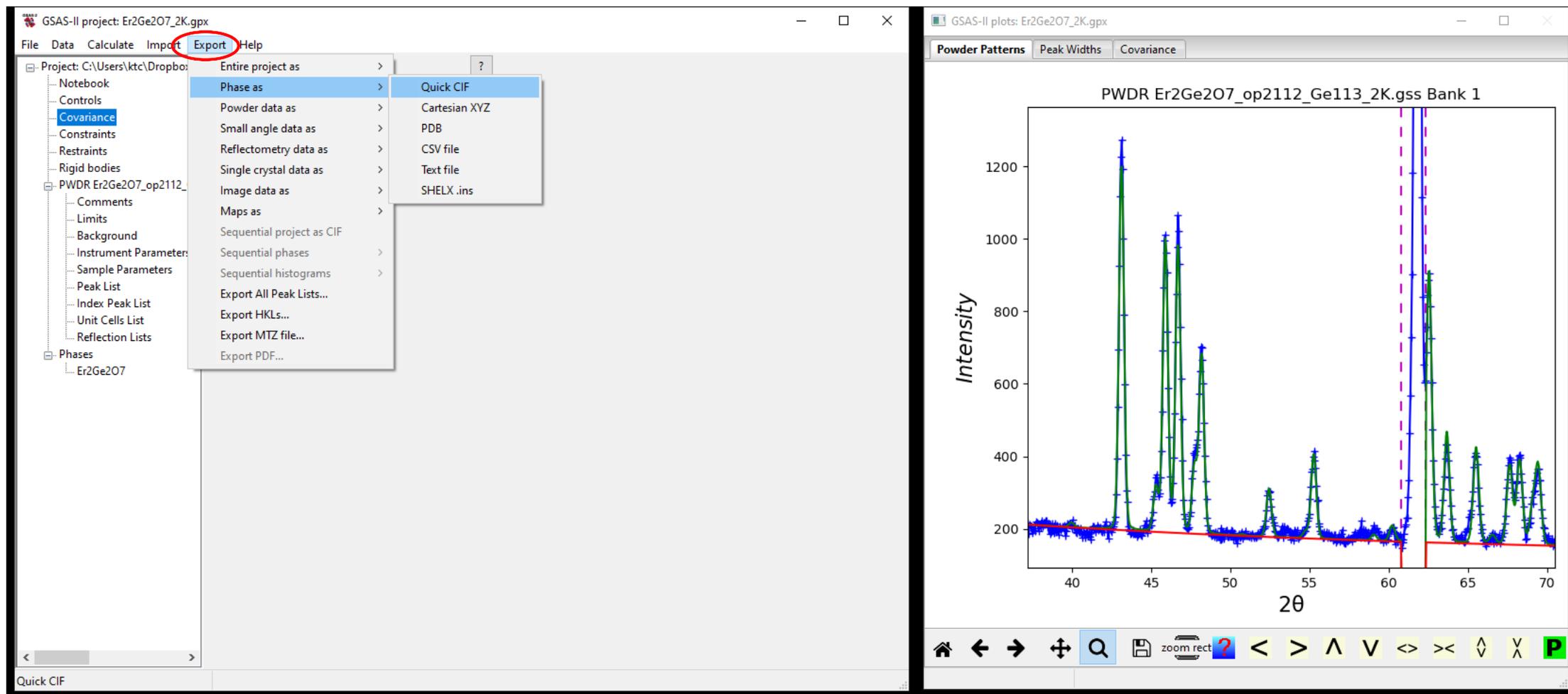
Optimizing the fit



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



The image displays two windows from the GSAS-II software. The left window, titled "GSAS-II project: Er2Ge2O7_2K.gpx", shows the "Export" menu open. The "Phase as" option is selected, and the "Quick CIF" sub-option is highlighted. The right window, titled "GSAS-II plots: Er2Ge2O7_2K.gpx", shows a powder pattern plot for "PWDR Er2Ge2O7_op2112_Ge113_2K.gss Bank 1". The plot shows Intensity versus 2θ, with a red line representing the fit and blue vertical lines indicating peak positions. The x-axis ranges from 40 to 70, and the y-axis ranges from 200 to 1200.

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

The screenshot displays the GSAS-II software interface. On the left, a project tree shows the current project: "GSAS-II project: Er2Ge207_2K.gpx". The main window is divided into several panes. The top-left pane shows "Refinement results" with the following data:

Parameter	Value
wR	5.345
chi**2	5696.7
GOF	1.62
Nobs	2188
Nvals	25
SVD zeros	0
max shift/esd	
log10 MaxLam	
Reduced χ^2	

The top-right pane shows a "Powder Patterns" plot titled "WDR Er2Ge207_op2112_Ge113_2K.gss Bank 1". The x-axis is labeled "2 θ " and ranges from 45 to 70. The plot shows a series of peaks with a red fit line and blue vertical markers. A vertical dashed line is drawn at approximately 61.5 degrees 2 θ .

In the center, a "Save in:" dialog box is open, showing the file name "Er2Ge207_2K" and the save type "Quick CIF (*.cif)". The dialog box also shows the "Save in:" location as "Er2Ge207" and the "Name" as "ErGeO_distbase".

Go ahead and save

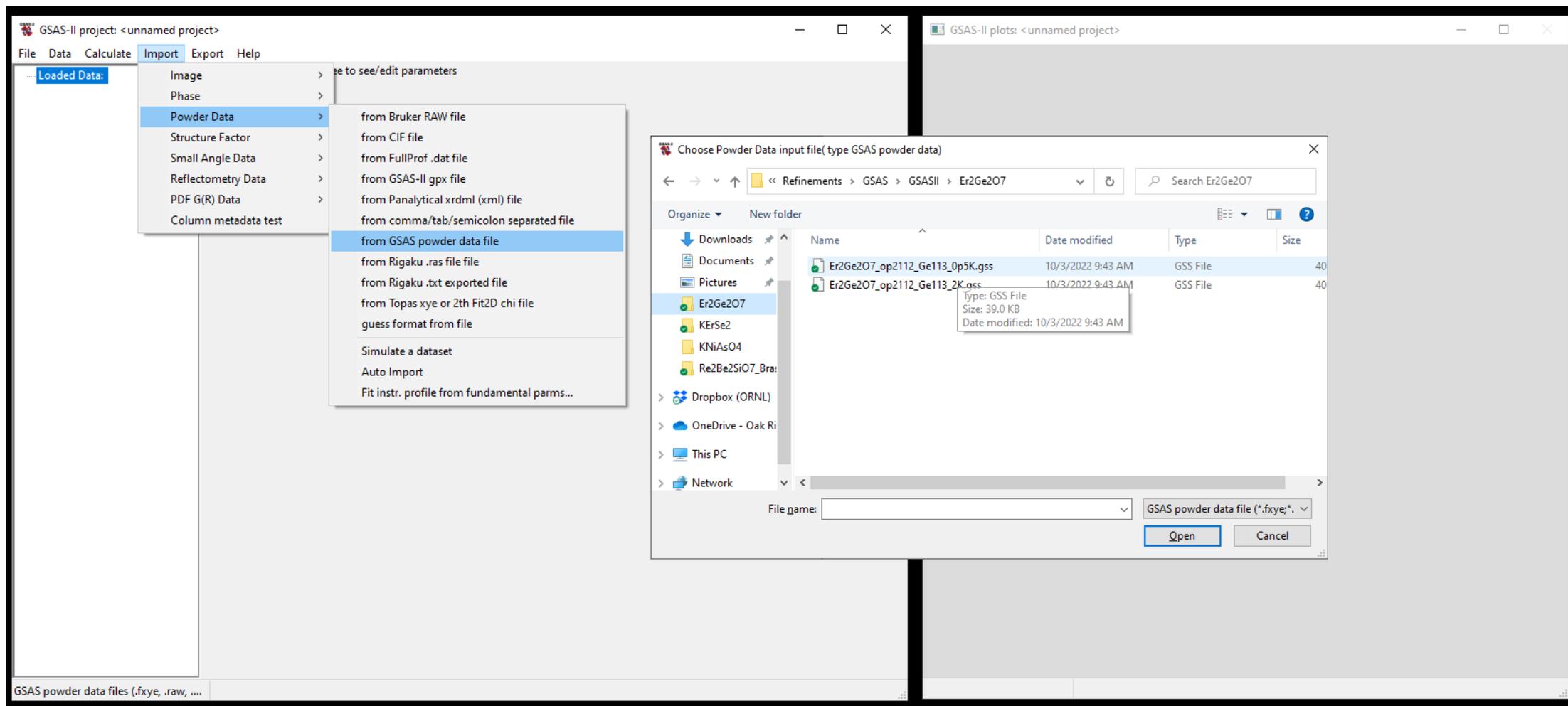
We are ready to move on to the magnetic refinement now!

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

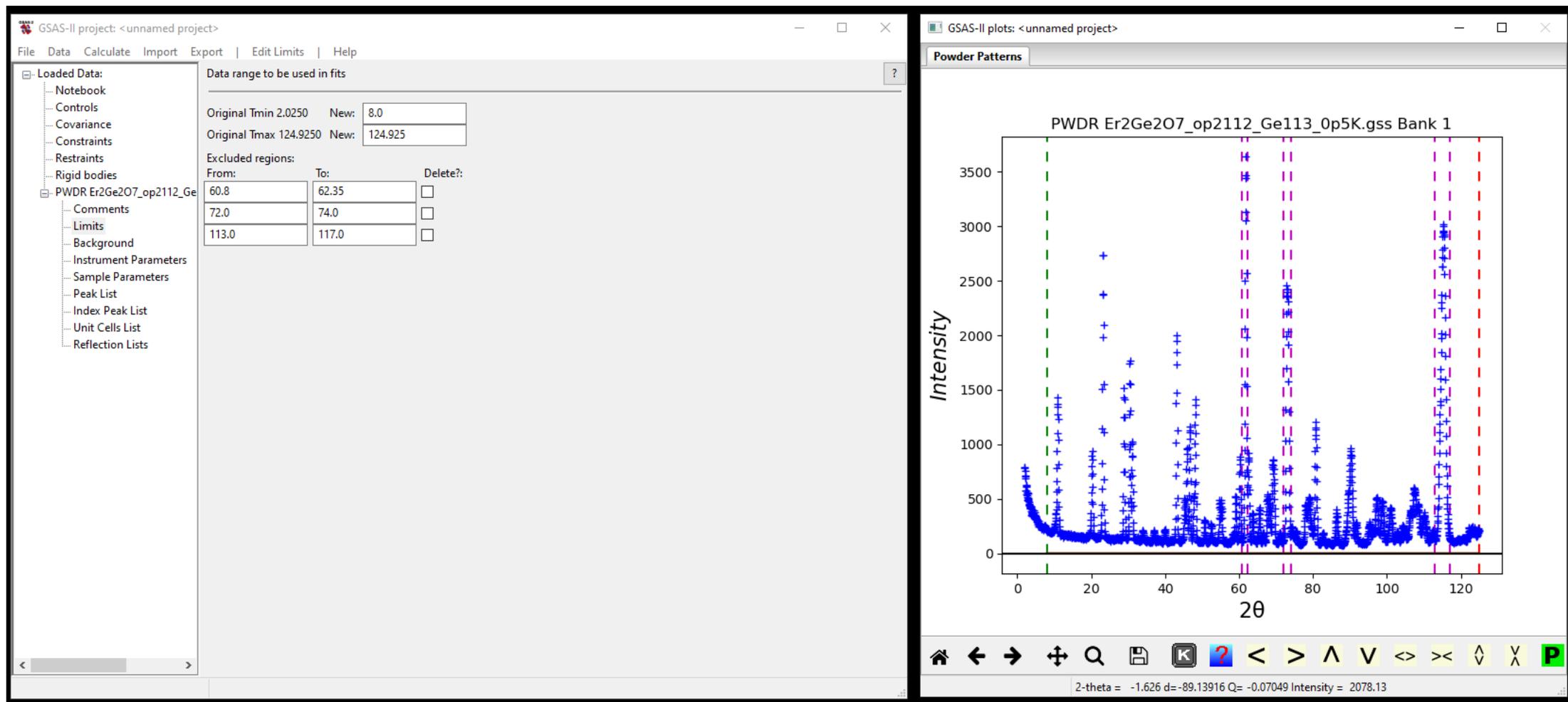


Starting the low temperature refinement



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

The image displays two windows from the GSAS-II software. The left window, titled 'GSAS-II project: <unnamed project>', shows the 'Import' menu open. The 'Phase' option is selected, and the 'from CIF file' option is highlighted. The 'Loaded Data' list on the left includes 'PWDR Er2Ge2O7'. The right window, titled 'GSAS-II plots: <unnamed project>', shows a powder pattern plot for 'PWDR Er2Ge2O7_op2112_Ge113_0p5K.gss Bank 1'. The plot shows Intensity versus 2θ, with a blue line representing the experimental data and vertical dashed lines representing the reference pattern. The x-axis ranges from 0 to 120, and the y-axis ranges from 0 to 3500. The plot title is 'PWDR Er2Ge2O7_op2112_Ge113_0p5K.gss Bank 1'. The status bar at the bottom of the plot window shows '2-theta = -1.626 d=-89.13916 Q=-0.07049 Intensity = 2078.13'.

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

Loading the 20 K structural data

The screenshot displays the GSAS-II interface. A 'Choose phase input file(type CIF)' dialog box is open, showing a file explorer view of the 'Er2Ge2O7' folder. The file 'Er2Ge2O7_2K' is selected. The background shows the main software window with a diffraction pattern plot titled 'Ge113_0p5K.gss Bank 1'. The plot shows intensity versus 2θ with various peaks and markers.

Name	Date modified	Type	Size
Er2Ge2O7_2K	10/3/2022 11:03 PM	CIF File	2
ErGeO_distbase	7/19/2017 11:18 AM	CIF File	2

File name: Er2Ge2O7_2K
File type: CIF file (*.CIF;*.cif;*.mcif)

0.07049 Intensity = 2078.13

Select your 2 K cif file

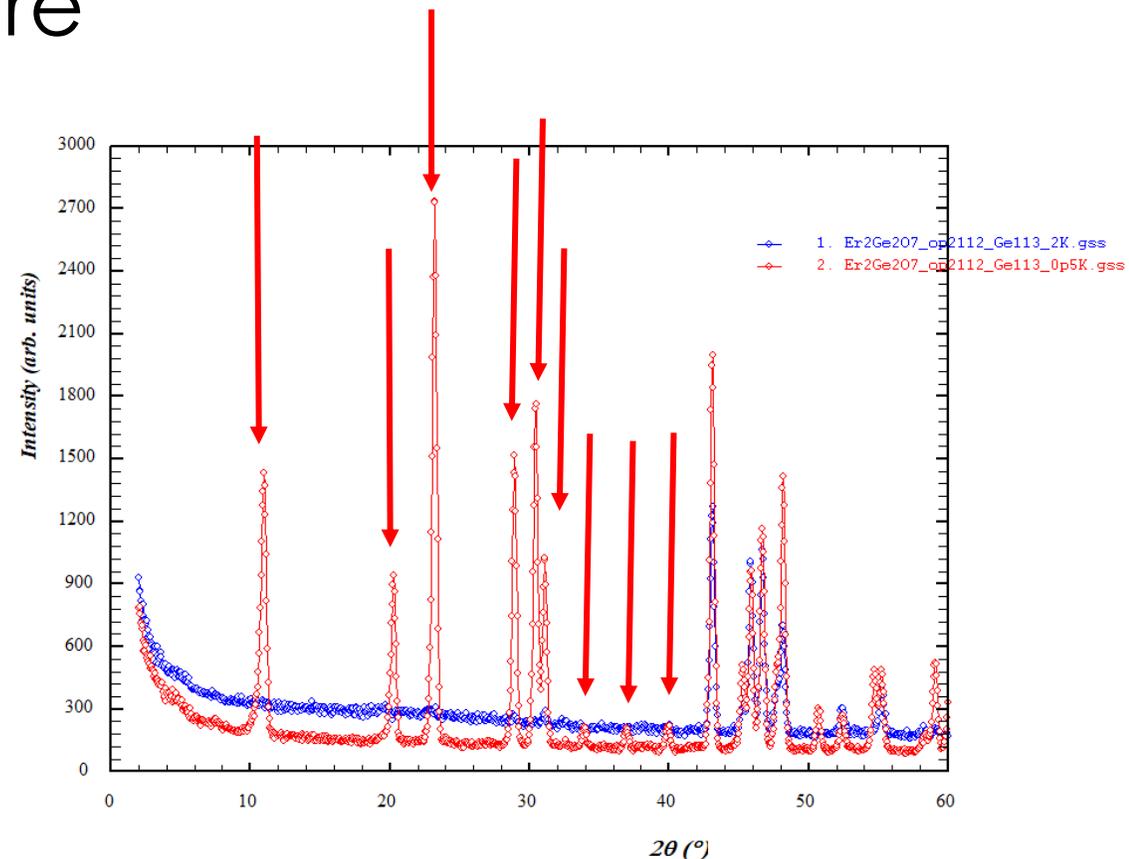
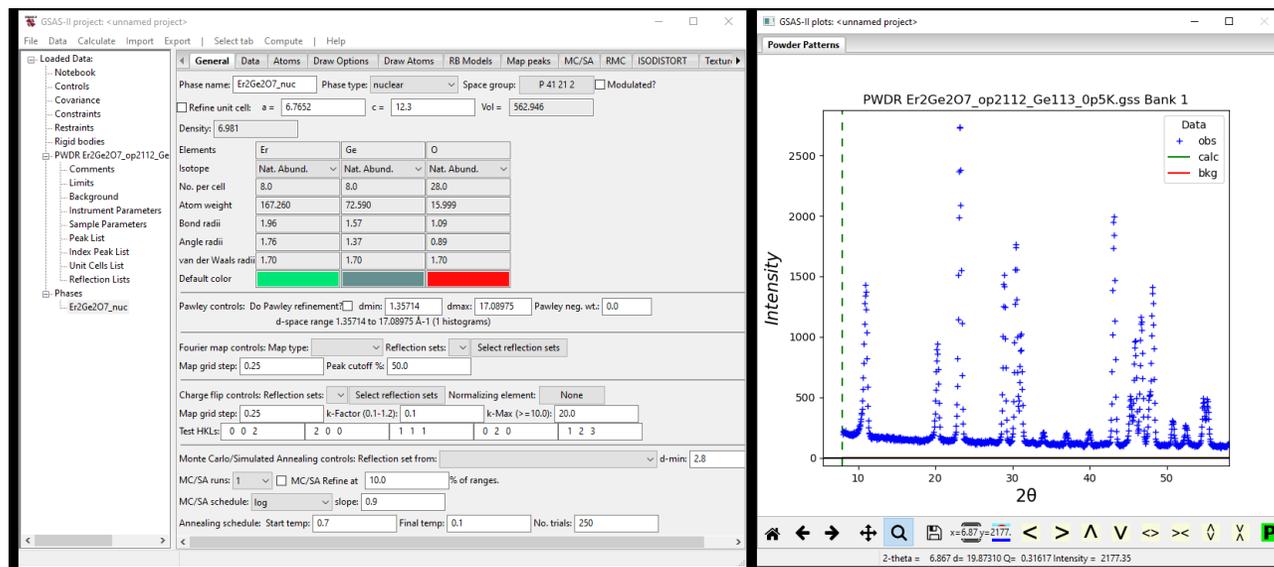
Loading the 100 K structural data

The image shows two windows from the GSAS-II software. The left window, titled "GSAS-II project: <unnamed project>", displays the "Loaded Data" tree on the left with "PWDR Er2Ge2O7_op2112_Ge" selected. The main area shows "Data range to be used in fits" with input fields for "Original Tmin", "New", "Original Tmax", and "New". Below this is a table for "Excluded regions" with columns "From:", "To:", and "Delete?:". A dialog box titled "Edit phase name" is open, with the text "Enter the name for the new phase" and a text box containing "Er2Ge2O7_nuc".

The right window, titled "GSAS-II plots: <unnamed project>", shows a "Powder Patterns" plot. The plot is titled "PWDR Er2Ge2O7_op2112_Ge113_0p5K.gss Bank 1". The y-axis is labeled "Intensity" and ranges from 0 to 3500. The x-axis is labeled "2θ" and ranges from 0 to 120. The plot shows a blue line representing the experimental data with several sharp peaks. Vertical dashed lines in green, purple, and red indicate the reference peak positions for different phases. The status bar at the bottom of the plot window shows "2-theta = -2.649 d=-53.66586 Q=-0.11708 Intensity = 3528.53".

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

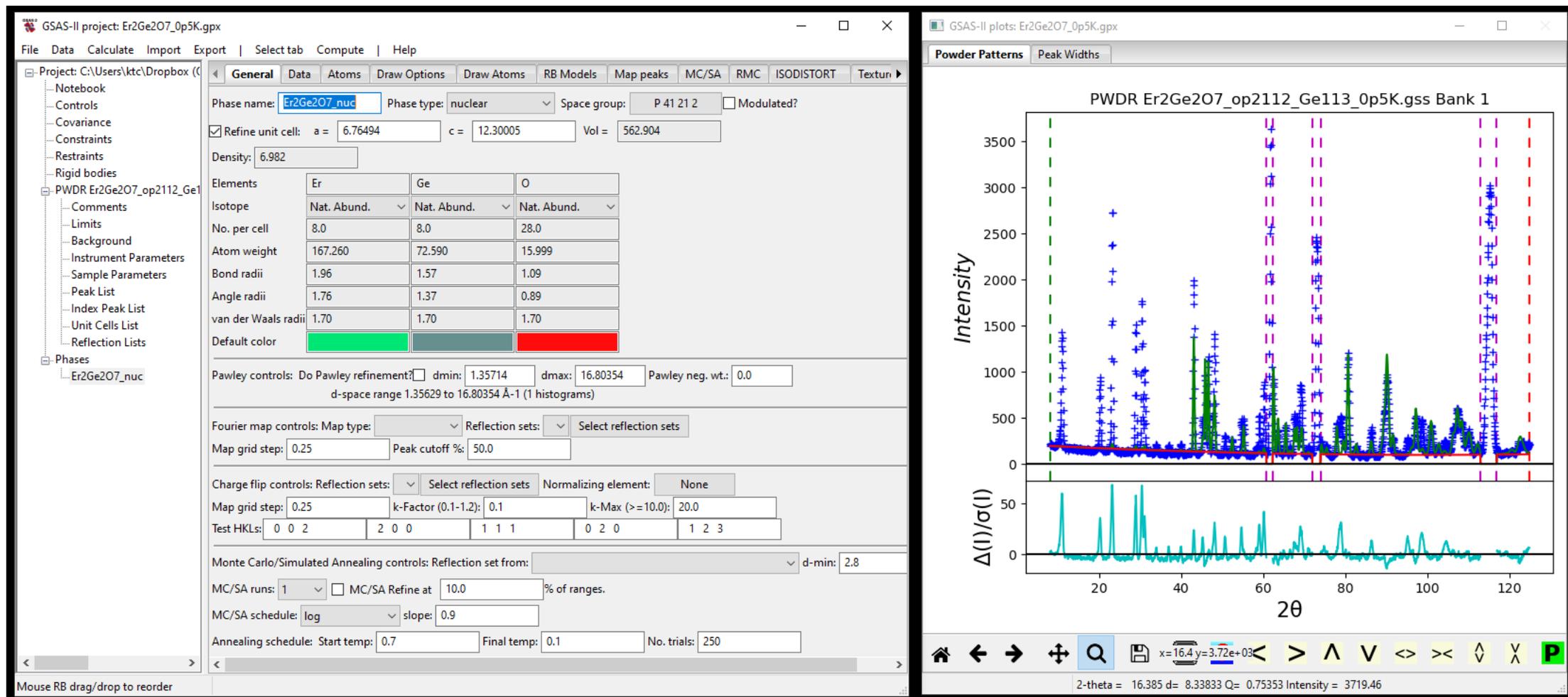
Starting with the nuclear structure



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

The screenshot displays the GSAS-II software interface. The left sidebar shows the project tree with the 'Unit Cells List' node highlighted in red. The main window shows the 'General' tab with various parameters for the phase 'Er2Ge2O7_nuc'. The right window shows a powder pattern plot titled 'PWDR Er2Ge2O7_op2112_Ge113_op5K.gss Bank 1' with intensity versus 2θ. The plot shows a series of peaks with vertical dashed lines indicating peak positions. The bottom status bar shows the current 2θ value: 16.385 d = 8.33833 Q = 0.75353 Intensity = 3719.46.

Phase name	Phase type	Space group	Modulated?
Er2Ge2O7_nuc	nuclear	P 41 21 2	<input type="checkbox"/>

Refine unit cell	a =	c =	Vol =
<input checked="" type="checkbox"/>	6.76494	12.30005	562.904

Elements	Er	Ge	O
Isotope	Nat. Abund.	Nat. Abund.	Nat. Abund.
No. per cell	8.0	8.0	28.0
Atom weight	167.260	72.590	15.999
Bond radii	1.96	1.57	1.09
Angle radii	1.76	1.37	0.89
van der Waals radii	1.70	1.70	1.70
Default color	Green	Grey	Red

Test HKLs	0 0 2	2 0 0	1 1 1	0 2 0	1 2 3

First go to the Unit Cells Node

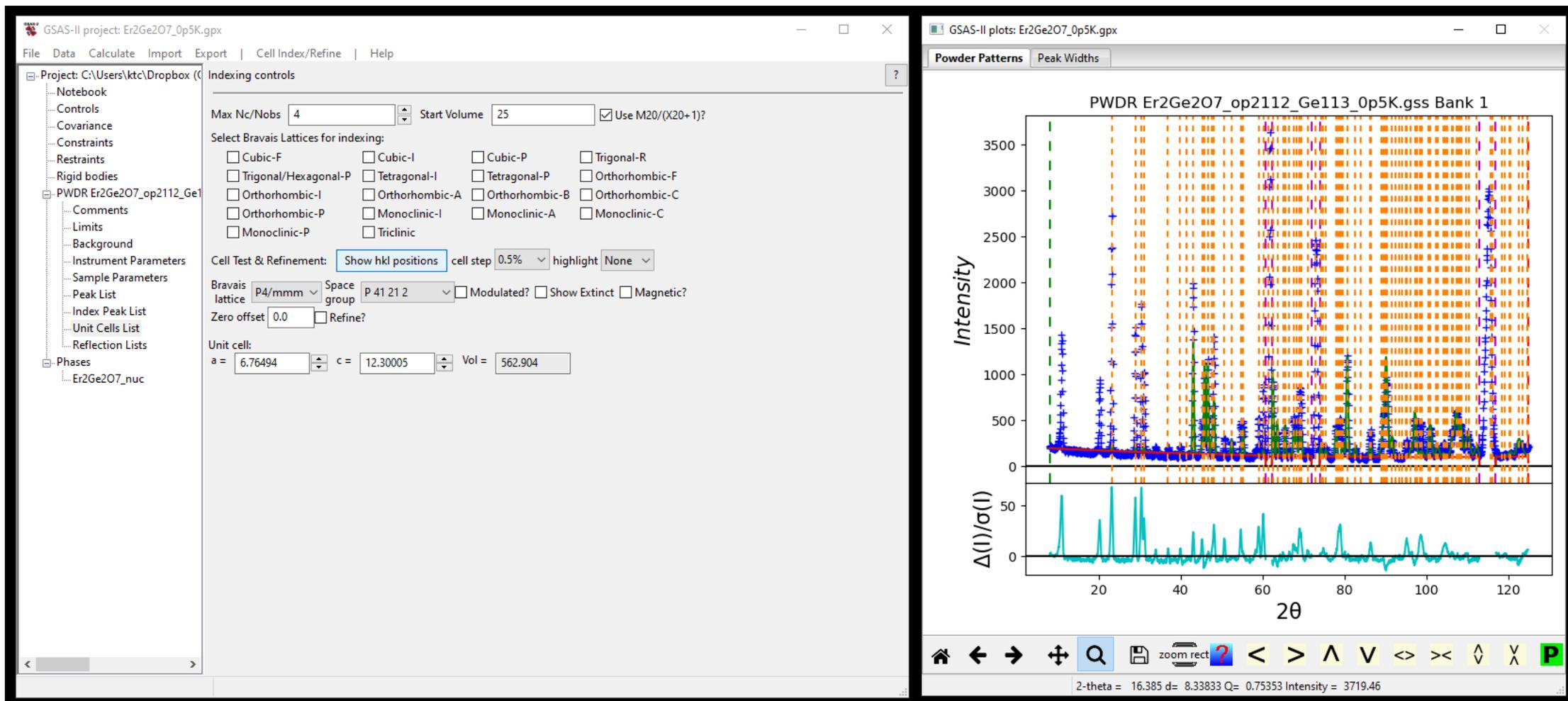
This will bring up a series of controls/options. We could put this all in manually, but you can also load the information from the nuclear phase. Let's do the latter.

Indexing the new peaks

The image displays two windows from the GSAS-II software. The left window, titled 'GSAS-II project: Er2Ge2O7_op5K.gpx', shows the 'Cell Index/Refine' menu with 'Load Phase' highlighted in red. The menu options include: Index Cell, Cell Symmetry Search, Run SUBGROUPS, Run k-SUBGROUPMAG, Copy Cell, Load Phase, Import Cell, Transform Cell, Refine Cell, Make new phase, and Export cell list. Below the menu, various parameters are visible, including 'Volume' (25), 'Use M20/(X20+1)?' (checked), 'Cell Test' (0.5%), 'highlight' (None), 'Bravais lattice' (group), 'Modulated?' (unchecked), 'Show Extinct' (unchecked), 'Magnetic?' (unchecked), 'Zero offset' (0.0), 'Refine?' (unchecked), and 'Unit cell' parameters (a=1.0, b=1.0, c=1.0, alpha=90.0, beta=90.0, gamma=90.0, Vol=1.000). The right window, titled 'GSAS-II plots: Er2Ge2O7_op5K.gpx', shows a powder pattern plot for 'PWDR Er2Ge2O7_op2112_Ge113_op5K.gss Bank 1'. The plot displays 'Intensity' on the y-axis (0 to 3500) versus '2θ' on the x-axis (0 to 120). The plot shows a series of peaks with error bars, overlaid with a red fit line. Below the main plot is a smaller plot showing the difference between the observed and fitted data, labeled $\Delta(I)/\sigma(I)$. The status bar at the bottom of the plot window shows: $2\text{-theta} = 16.385$ $d = 8.33833$ $Q = 0.75353$ $\text{Intensity} = 3719.46$.

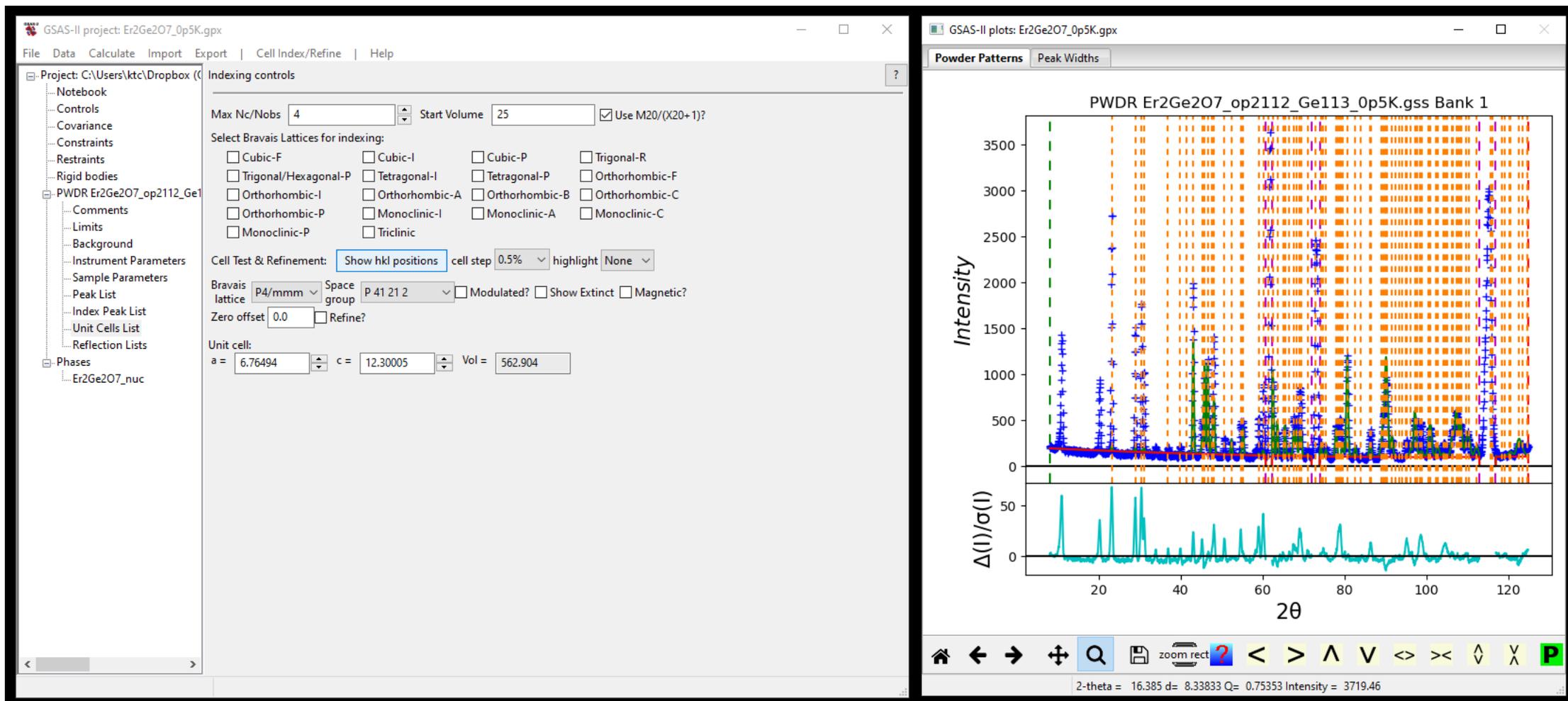
Click on 'Cell Index/Refine' in the toolbar and choose 'Load Phase'

Indexing the new peaks



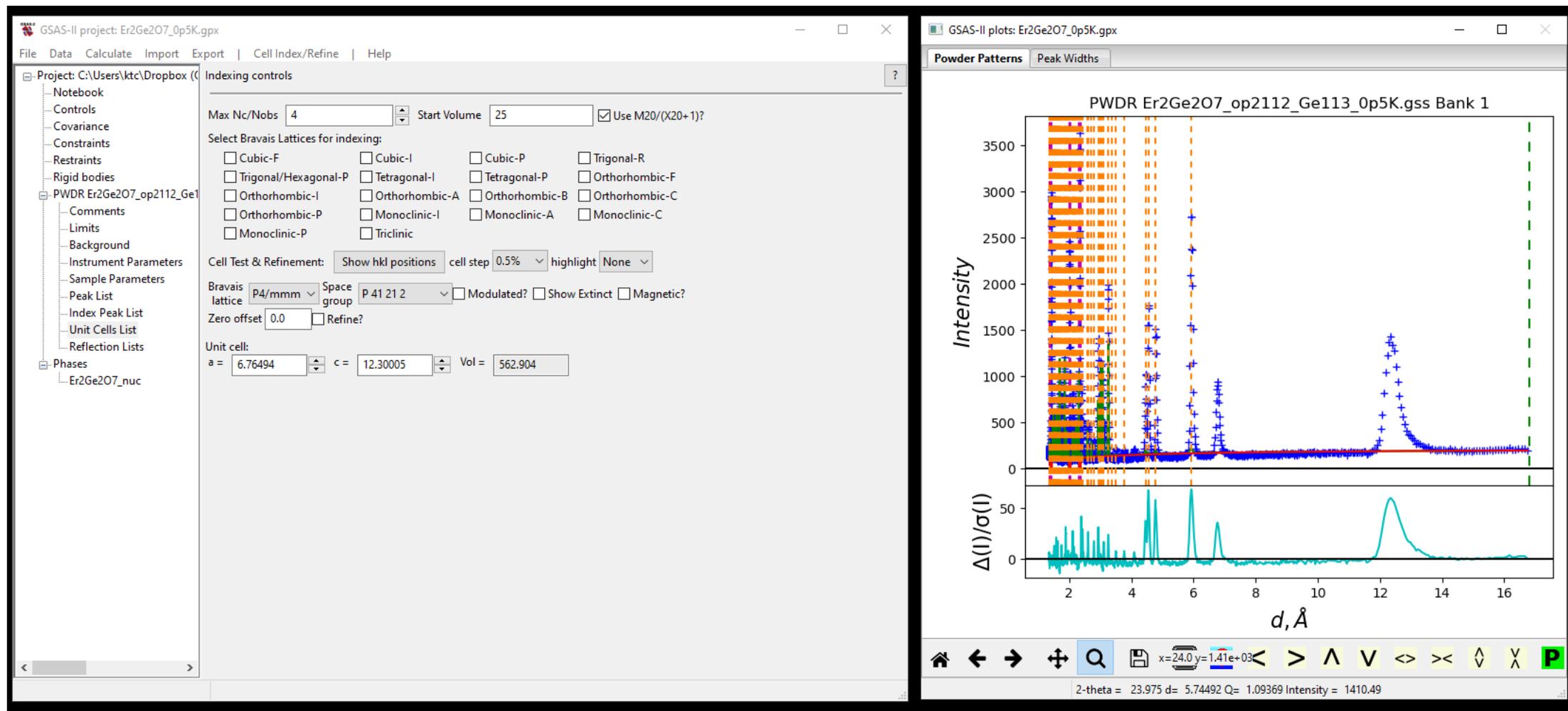
Since we only have one nuclear phase, it will automatically load it. You should see a bunch of vertical lines in the powder pattern which correspond to peak indices.

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

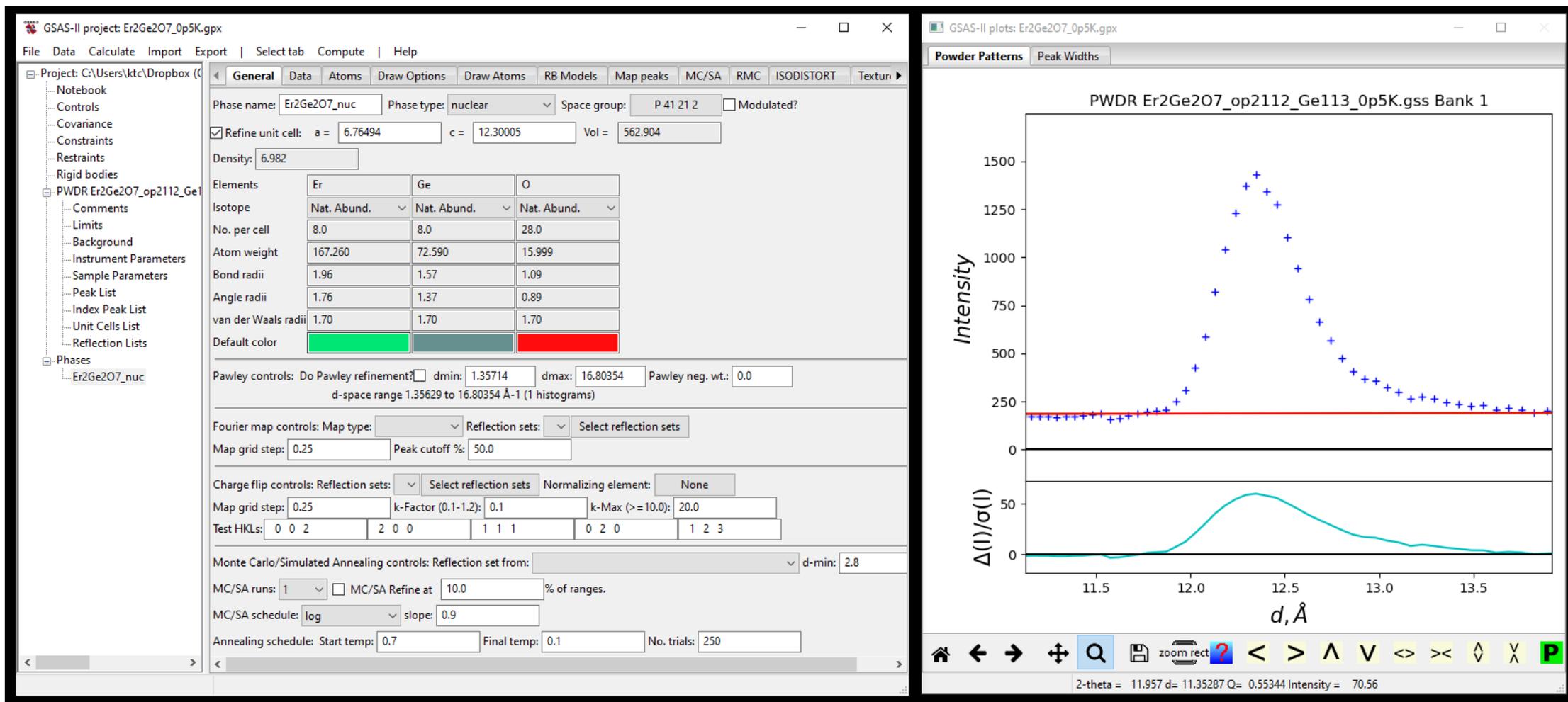


If we are lucky, changing to d-spacing might help us identify this low angle peak – in particular if it can be located at some multiple of a lattice parameter

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.

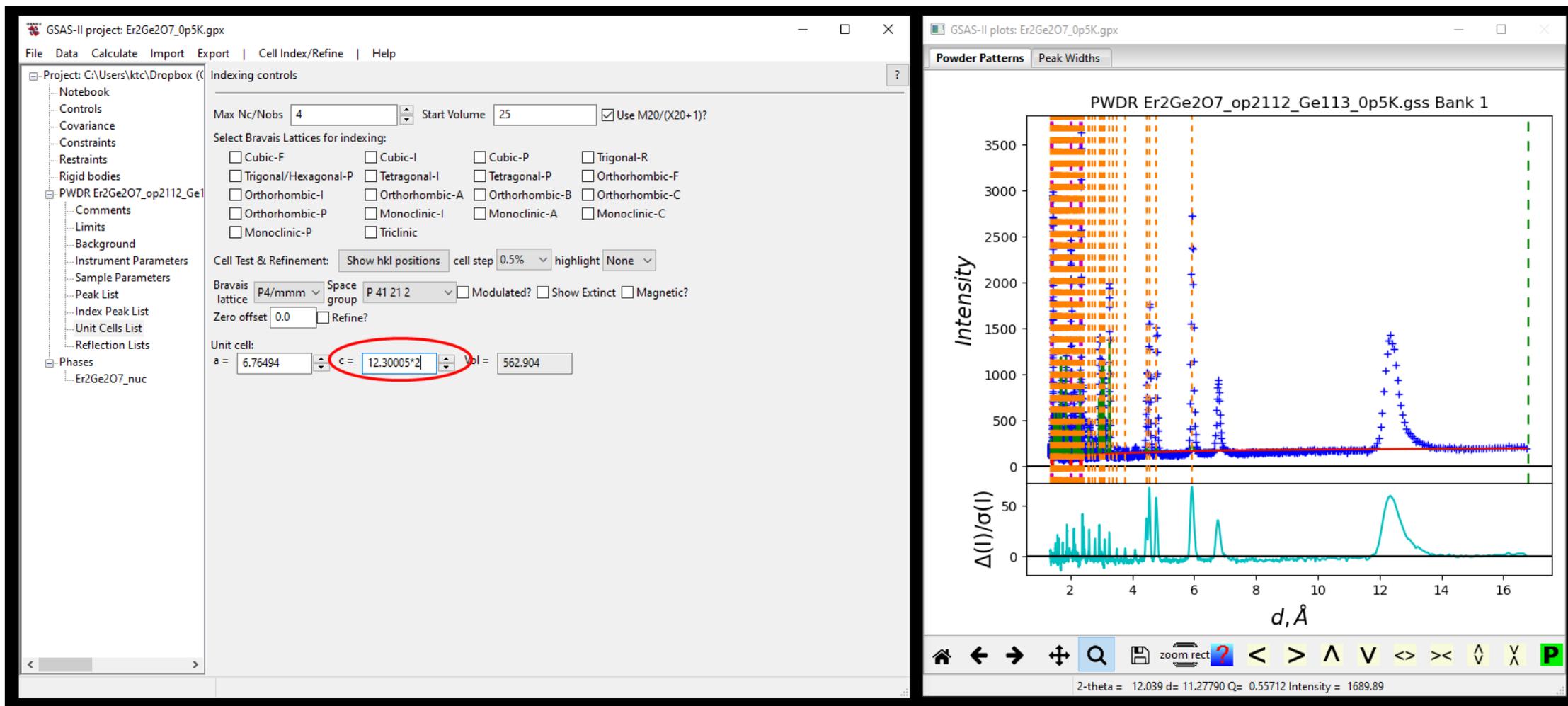
Indexing the new peaks

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



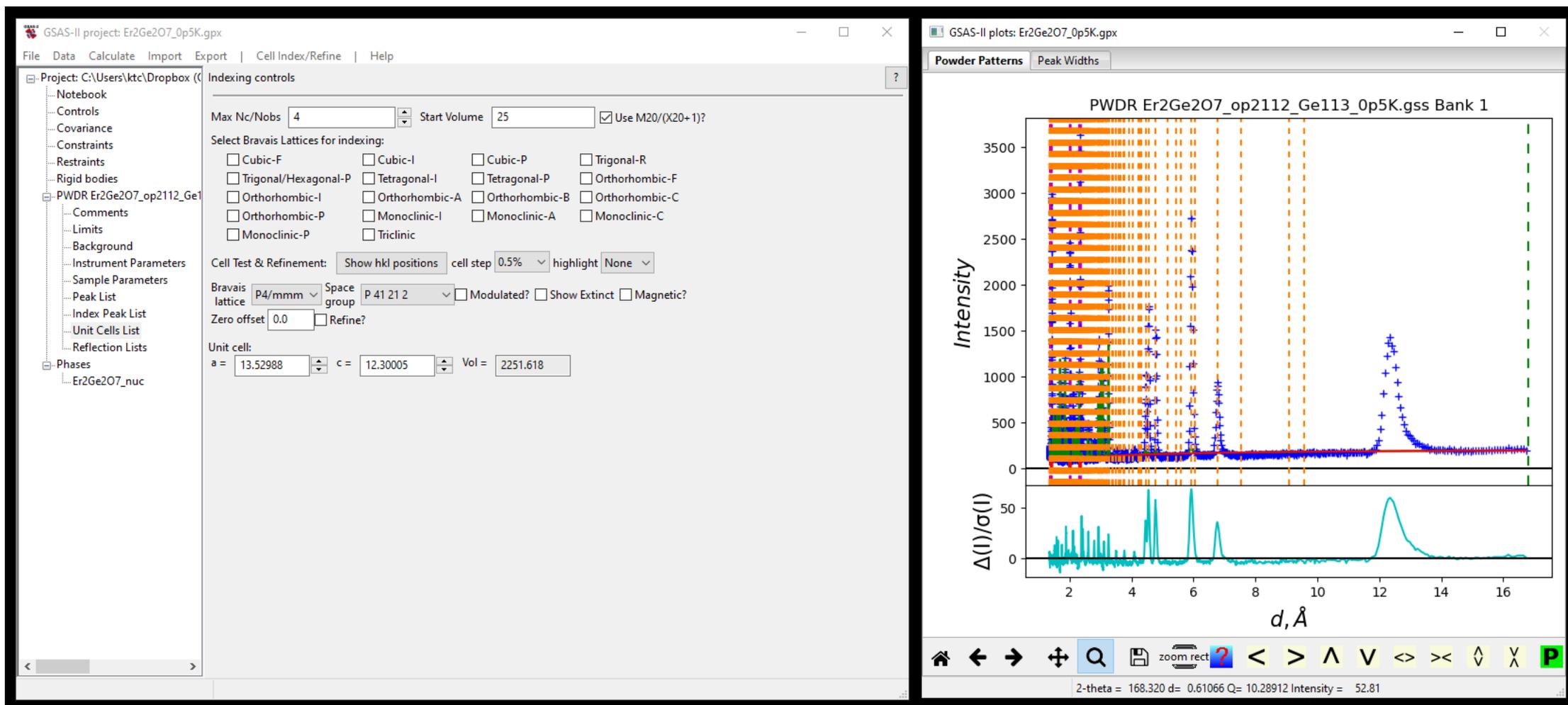
Let's zoom in on that large d-spacing peak. Its at 12.3 Å, very near the c lattice parameter as a 001 reflection. This would indicate a k=(000). We can test this:

Indexing the new peaks



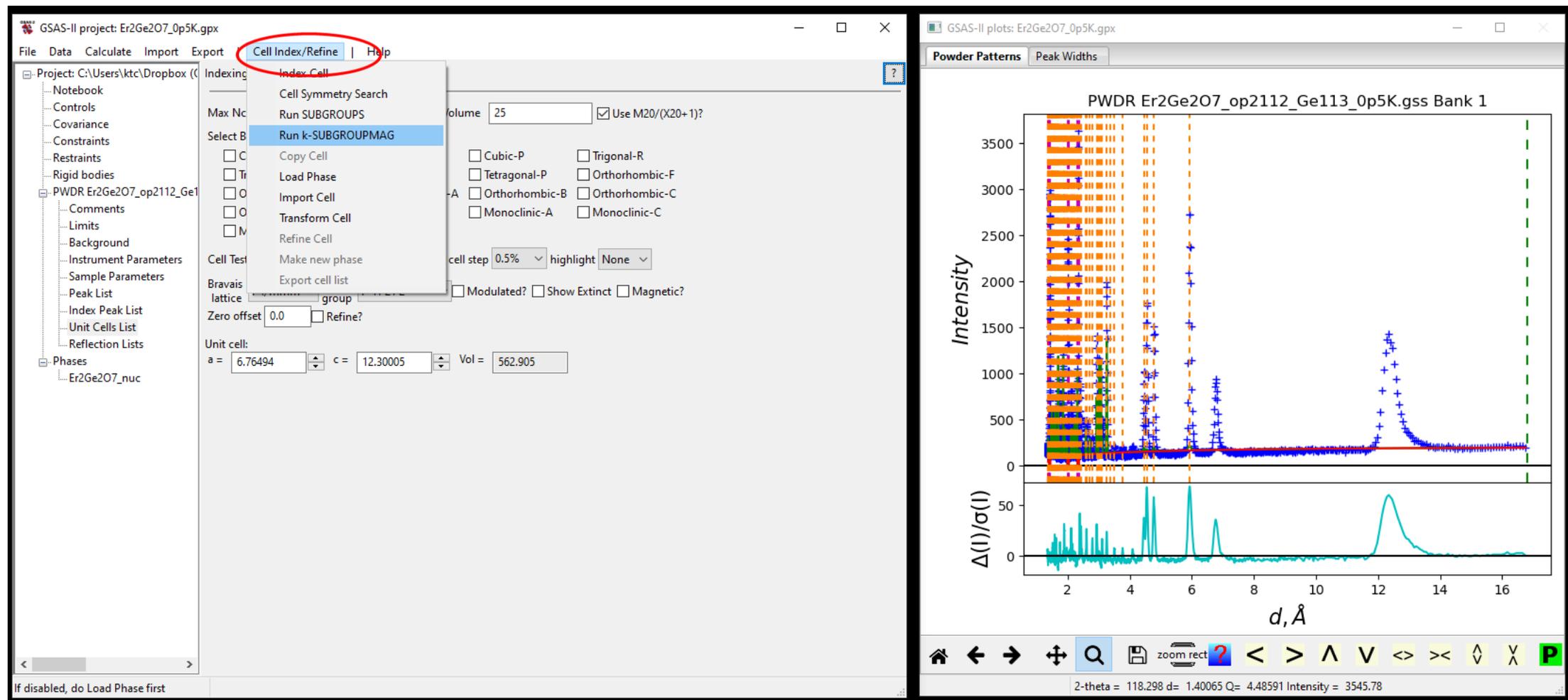
To do so, let's try playing with larger cells type in a doubled lattice parameter into any of the boxes in the Indexing controls window (or type '*2' in the box after the current lattice parameter.) I've tried doubling c above. It doesn't seem to help much – lets try $2*a$.

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 our largest d -spacing peak seems to correspond to our c lattice parameter we have a good lead.

Solving the structure

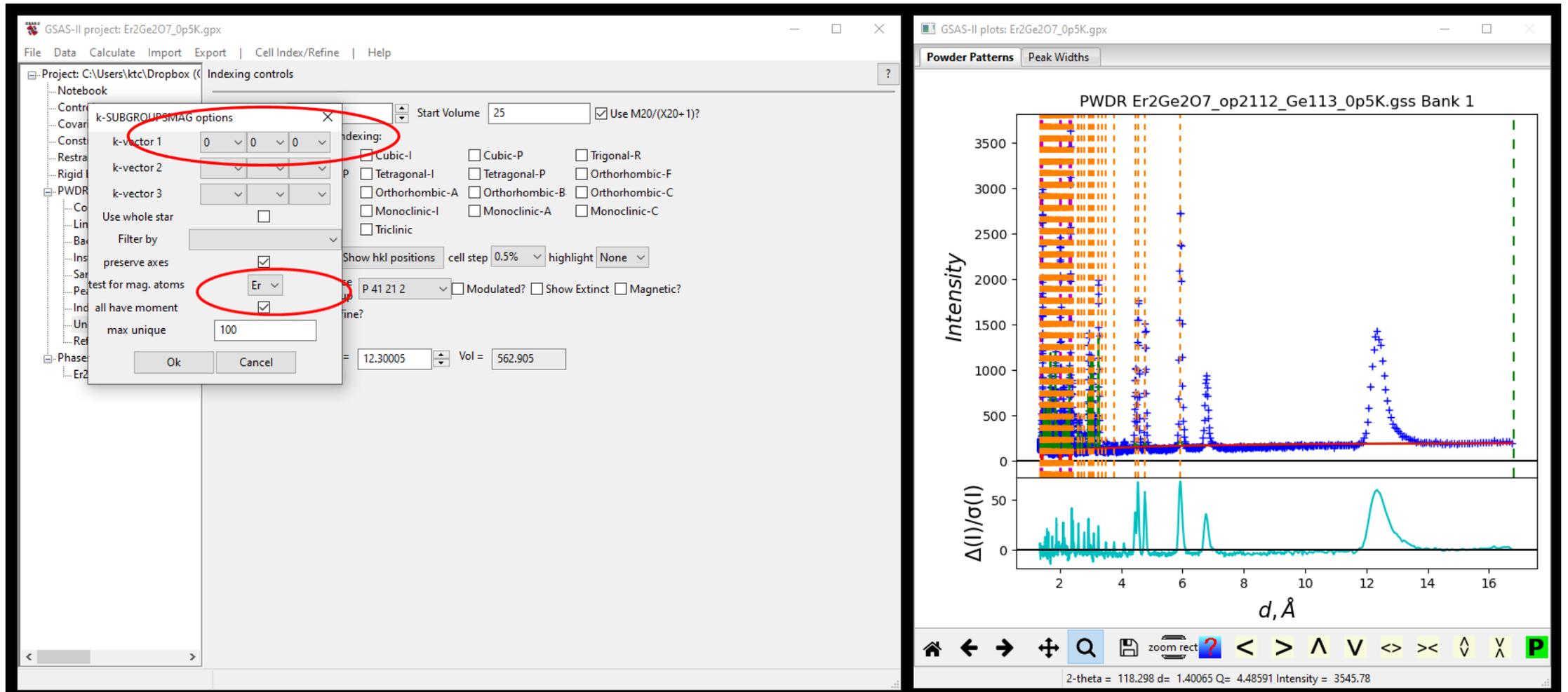


With a guess of the k-vector we can now use GSAS-II to link to the Bilbao server and run k-SUBGROUPMAG. This will provide a list of all the possible magnetic subgroups of our nuclear structure's space group given our found k-vector.

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

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

Solving the structure

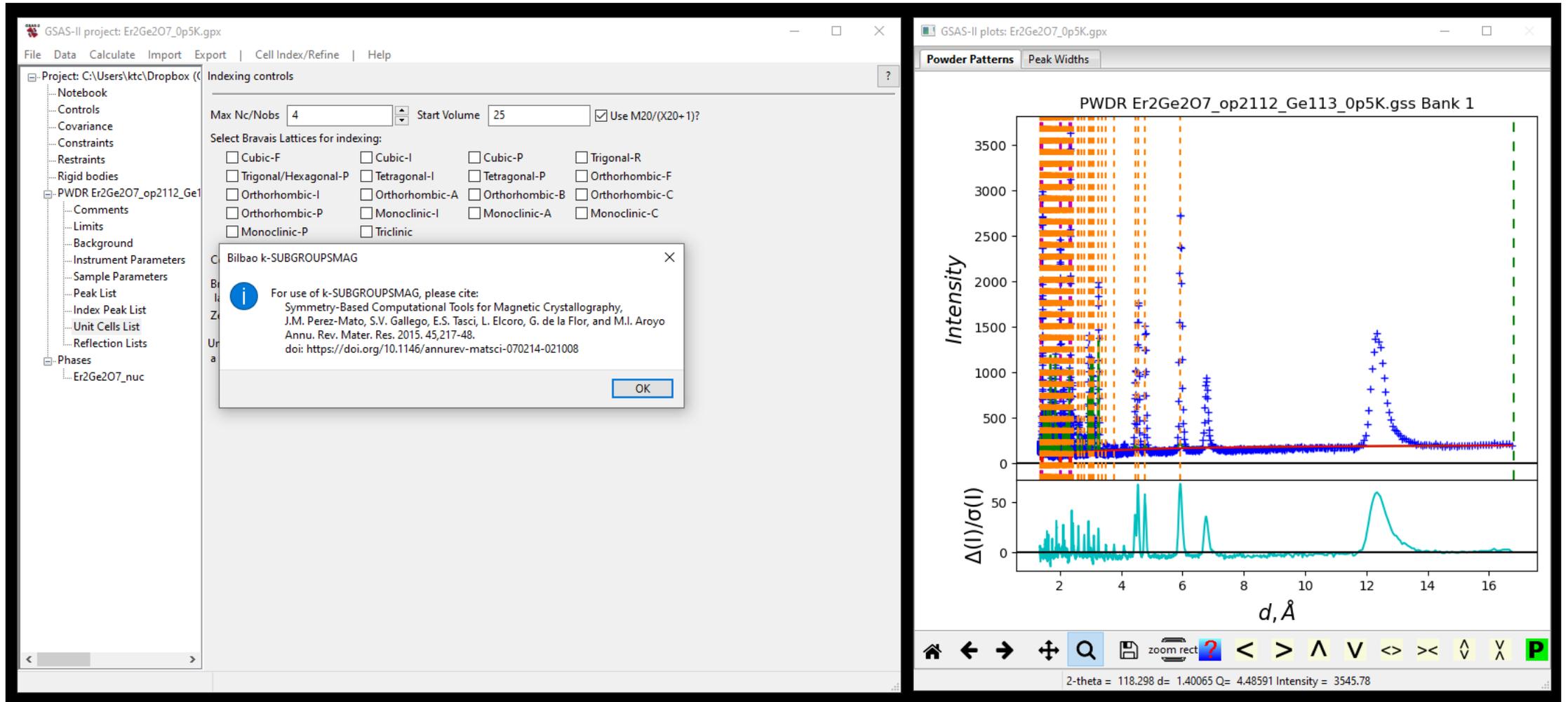


This should open a window allowing you to input one (or several) k-vectors and set some search criteria.

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

GSAS-II project: Er2Ge2O7_0p5K.gpx

File Data Calculate Import Export | Cell Index/Refine | Help

Project: C:\Users\kct\Dropbox (G...)

Start Volume: 25 Use M20/(X20+1)?

indexing:

Cubic-I Cubic-P Trigonal-R

Tetragonal-I Tetragonal-P Orthorhombic-F

Orthorhombic-A Orthorhombic-B Orthorhombic-C

Monoclinic-I Monoclinic-A Monoclinic-C

Triclinic

Show hkl positions cell step 0.5% highlight None

Space group: P 41 21 2 Modulated? Show Extinct Magnetic?

Refinement: $\chi^2 = 12.30005$ Vol = 562.905

From Bilbao k-SUBGROUPSMAG for P 41 21 2; kvec1=(0,0,0):

Try	Keep	Uniq	nConj	nSup	Trans	Vec	a	b	c	alpha	beta	gamma	Volume
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	2	1	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000	90.000	1125.81
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000	90.000	1125.81
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000	90.000	1125.81
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	8	2	1	a,-b,-c	0,1/4,1/8	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	4	2	2	a-b,a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000	90.000	1125.81

Double click Keep to refresh Keep flags; click Space Gp to see sym. ops; Uniq to see unique atoms list; Try to trigger K & J keys on plot

GSAS-II plots: Er2Ge2O7_0p5K.gpx

Powder Patterns Peak Widths

PWDR Er2Ge2O7_op2112_Ge113_0p5K.gss Bank 1

Intensity

$\Delta(I)/\sigma(I)$

$d, \text{\AA}$

2-theta = 118.298 d = 1.40065 Q = 4.48591 Intensity = 3545.78

Note the scroll bar

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

The screenshot displays the GSAS-II software interface. The left pane shows the project hierarchy for 'Er2Ge2O7'. The main window is the 'indexing' panel, where the 'Show Extinct' checkbox is checked and circled in red. Below this, a table lists indexed peaks with columns for 'Try', 'Keep', 'Uniq', 'nConj', 'nSup', 'Trans', 'Vec', and lattice parameters 'a', 'b', 'c', 'alpha', 'beta', 'gamma', and 'Volume'.

Try	Keep	Uniq	nConj	nSup	Trans	Vec	a	b	c	alpha	beta	gamma	Volume
<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	2	1	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000	90.000	1125.81
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000	90.000	1125.81
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000	90.000	1125.81
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90
<input type="checkbox"/>	<input checked="" type="checkbox"/>	8	2	1	a,-b,-c	0,1/4,1/8	6.76494	6.76494	12.30005	90.000	90.000	90.000	562.90

The right pane shows the 'Powder Patterns' plot for 'PWDR Er2Ge2O7_op2112_Ge113_op5K.gss Bank 1'. The plot displays Intensity versus $d, \text{\AA}$. The x-axis ranges from 2 to 16 \AA , and the y-axis ranges from 0 to 3500. The plot shows a series of peaks, with a prominent peak at approximately 12.3 \AA . The plot is overlaid with a red line representing the fit and a blue line representing the observed data. The plot also shows a zoomed-in view of the peak at 12.3 \AA in the bottom panel.

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

The screenshot displays the GSAS-II interface. On the left, the 'Indexing controls' window is open, showing 'Magnetic space group information' for P 41 21' 2'. The lattice is noncentrosymmetric primitive tetragonal with Laue symmetry 4/mmm and magnetic lattice point group 42'2'. The equivalent positions are listed as follows:

- (1) X, Y, Z, (1) (2) 1/2-Y, 1/2+X, 1/4+Z, (4z)
- (3) -X, -Y, 1/2+Z, (2z) (4) 1/2+Y, 1/2-X, 3/4+Z, (4z3)
- (5) 1/2-X, 1/2+Y, 1/4-Z, (2y) (6) -Y, -X, 1/2-Z, (2+-0)
- (7) 1/2+X, 1/2-Y, 3/4-Z, (2x) (8) Y, X, -Z, (2110)

Below this, a table lists symmetry operations for the space group P 41 21' 2'. The table includes columns for Space Gp, Try, Keep, Uniq, nConj, nSup, Trans, Vec, a, b, c, alpha, and beta.

	Space Gp	Try	Keep	Uniq	nConj	nSup	Trans	Vec	a	b	c	alpha	beta
1	P41'21'2	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000
2	P4121'2'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000
3	P41'212'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000
4	P41212	<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000
5	P41'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.000
6	P41	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.000
7	C22'21'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	2	1	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000
8	C2'2'21	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000
9	C2221	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000
10	P21'21'21	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.000
11	P21'2121'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	8	2	1	a,-b,-c	0,1/4,1/8	6.76494	6.76494	12.30005	90.000	90.000

On the right, the 'Powder Patterns' window shows a plot of Intensity vs. d, Å. The plot displays experimental data (blue crosses) and a fit (red line). The x-axis ranges from 2 to 16 Å, and the y-axis ranges from 0 to 3500. The plot is titled 'PWDR Er2Ge2O7_op2112_Ge113_Op5K.gss Bank 1'. The bottom status bar shows: 2-theta = 168.320 d= 1.12719 Q= 5.57419 Intensity = 2626.27.

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

Solving the structure

GSAS-II project: Er2Ge2O7_op5K.gpx

File Data Calculate Import Export Cell Index/Refine Help

Orthorhombic-P
 Monoclinic-P
 Monoclinic-A
 Monoclinic-C
 Monoclinic-P
 Triclinic

Cell Test & Refinement: Show hkl positions cell step 0.5% highlight None

Bravais lattice P4/mmm Space group P 41 21 2 Modulated? Show Extinct Magnetic?

Zero offset 0.0 Refine?

BNS lattice: P 2+ -0: black 2x: black Show spins?

Unit cell:
a = 6.76494 c = 12.30005 Vol = 562.905

Magnetic subgroup cells from Bilbao k-SUBGROUPSMAG for P 41 21 2; kvec1=(0,0,0):

	Space Gp	Try	Keep	Uniq	nConj	nSup	Trans	Vec	a	b	c	alpha	beta
1	P41'21'2'	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000
2	P4121'2'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000
3	P41'212'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000
4	P41212	<input type="checkbox"/>	<input checked="" type="checkbox"/>	1	1	1	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000
5	P41'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.000
6	P41	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	0,1/2,0	6.76494	6.76494	12.30005	90.000	90.000
7	C22'21'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	2	1	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000
8	C2'2'21	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000
9	C2221	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a+b,-a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000
10	P21'21'21	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.000
11	P21'2121'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	8	2	1	a,-b,-c	0,1/4,1/8	6.76494	6.76494	12.30005	90.000	90.000
12	P212121	<input type="checkbox"/>	<input checked="" type="checkbox"/>	2	1	2	a,b,c	1/4,0,-1/8	6.76494	6.76494	12.30005	90.000	90.000
13	C2'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	4	2	2	a-b,a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000
14	C2	<input type="checkbox"/>	<input checked="" type="checkbox"/>	4	2	2	a-b,a+b,c	0,0,0	9.56707	9.56707	12.30005	90.000	90.000
15	P21'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	4	1	4	a,c,-b	0,0,0	6.76494	12.30005	6.76494	90.000	90.000
16	P21'	<input type="checkbox"/>	<input checked="" type="checkbox"/>	4	2	2	a,b,c	1/4,0,1/8	6.76494	6.76494	12.30005	90.000	90.000
17	P21	<input type="checkbox"/>	<input checked="" type="checkbox"/>	4	1	6	a,c,-b	0,0,0	6.76494	12.30005	6.76494	90.000	90.000
18	P21	<input type="checkbox"/>	<input checked="" type="checkbox"/>	4	2	2	a,b,c	1/4,0,1/8	6.76494	6.76494	12.30005	90.000	90.000
19	P1	<input type="checkbox"/>	<input checked="" type="checkbox"/>	8	1	10	a,b,c	0,0,0	6.76494	6.76494	12.30005	90.000	90.000

Double click Keep to refresh Keep flags; click Space Gp to see sym. ops., Uniq to see unique atoms list; Try to trigger K & J keys on plot

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

The image displays the GSAS-II software interface. On the left, the 'Project' tree shows 'Er2Ge2O7_nuc' selected under 'Phases'. The 'Compute' menu is open, with 'Select magnetic/subgroup phase' highlighted. The main window shows the 'General' tab with various refinement parameters. On the right, the 'Powder Patterns' window displays a plot of 'PWDR Er2Ge2O7_op2112_Ge113_op5K.gss Bank 1'. The plot shows Intensity vs. $d, \text{\AA}$ with a red line for the observed data and a blue line for the fit. The x-axis ranges from 2 to 16 \AA , and the y-axis ranges from 0 to 3500. The plot also shows $\Delta(I)/\sigma(I)$ vs. $d, \text{\AA}$ at the bottom.

GSAS-II project: Er2Ge2O7_op5K.gpx

File Data Calculate Import Export | Select **Compute** Help

Project: C:\Users\kct\Dropbox (G...)

- Notebook
- Controls
- Covariance
- Constraints
- Restrains
- Rigid bodies
- PWDR Er2Ge2O7_op2112_Ge1...
 - Comments
 - Limits
 - Background
 - Instrument Parameters
 - Sample Parameters
 - Peak List
 - Index Peak List
 - Unit Cells List
 - Reflection Lists
 - Phases
 - Er2Ge2O7_nuc

General Data

Phase name: Er2Ge2O7

Refine unit cell:

Density: 6.982

Elements: []

Isotope: []

No. per cell: []

Atom weight: []

Bond radii: []

Angle radii: 1.76 1.37 0.89

van der Waals radii: 1.70 1.70 1.70

Default color: [Green] [Grey] [Red]

Space group: P 41 21 2 Modulated?

Vol = 562.904

Fourier map

Search map

Charge flipping

4D Charge flipping

Clear map

MC/SA

Multi MC/SA

Transform

Compare

Select magnetic/subgroup phase

Protein quality

Pawley controls: Do Pawley refinement? dmin: 1.35714 dmax: 16.80354 Pawley neg. wt.: 0.0

d-space range 1.35629 to 16.80354 \AA^{-1} (1 histograms)

Fourier map controls: Map type: [] Reflection sets: [] Select reflection sets

Map grid step: 0.25 Peak cutoff %: 50.0

Charge flip controls: Reflection sets: [] Select reflection sets Normalizing element: None

Map grid step: 0.25 k-Factor (0.1-1.2): 0.1 k-Max (≥ 10.0): 20.0

Test HKLs: 0 0 2 2 0 0 1 1 1 0 2 0 1 2 3

Monte Carlo/Simulated Annealing controls: Reflection set from: [] d-min: 2.8

MC/SA runs: 1 MC/SA Refine at 10.0 % of ranges.

MC/SA schedule: log slope: 0.9

Annealing schedule: Start temp: 0.7 Final temp: 0.1 No. trials: 250

If disabled, make in PWDR/Unit Cells

GSAS-II plots: Er2Ge2O7_op5K.gpx

Powder Patterns Peak Widths

PWDR Er2Ge2O7_op2112_Ge113_op5K.gss Bank 1

Intensity

$d, \text{\AA}$

$\Delta(I)/\sigma(I)$

2-theta = 168.320 d= 1.12719 Q= 5.57419 Intensity = 2626.27

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!)

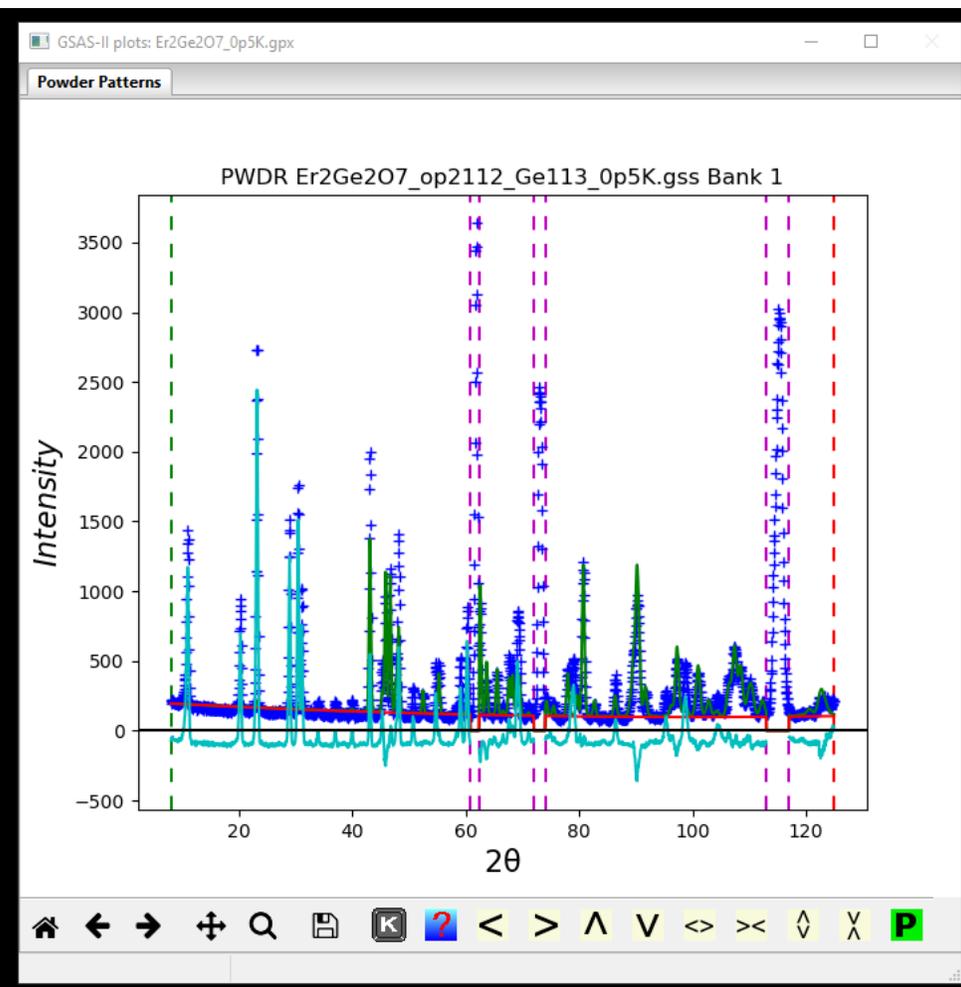
Solving the structure

The screenshot shows the GSAS-II software interface. The main window is titled 'GSAS-II project: Er2Ge2O7_op5K.gpx'. The 'General' tab is active, showing the following parameters:

- Phase name: Er2Ge2O7_nuc
- Phase type: nuclear
- Space group: P 41 21 2
- Modulated?
- Refine unit cell: a = 6.76494, c = 12.30005, Vol = 562.904
- Density: 6.982
- Elements: Er, Ge, O
- Isotope: Nat. Abund. (dropdowns)
- No. per cell: 8.0
- Atom weight: 167.2
- Bond radii: 1.96
- Angle radii: 1.76
- van der Waals radii: 1.70
- Default color: Green
- Pawley controls: Do Pawley (checkbox), Pawley neg. wt.: 0.0
- Fourier map controls: Map grid step: 0.25
- Charge flip controls: Refl. (checkbox), Refining element: None
- Map grid step: 0.25, k-Factor (0.1-1.2): 0.1, k-Max (>=10.0): 20.0
- Test HKLs: 0 0 2, 2 0 0, 1 1 1, 0 2 0, 1 2 3
- Monte Carlo/Simulated Annealing controls: Reflection set from: (dropdown), d-min: 2.8
- MC/SA runs: 1, MC/SA Refine at: 10.0 % of ranges.
- MC/SA schedule: log, slope: 0.9
- Annealing schedule: Start temp: 0.7, Final temp: 0.1, No. trials: 250

A dialog box titled 'Make new magnetic phase' is open, showing a list of magnetic space groups:

- (1) P41'21'2; (a,b,c) + (0,0,0)
- (2) P4121'2; (a,b,c) + (0,0,0)
- (3) P41'212; (a,b,c) + (0,0,0)
- (4) P41212; (a,b,c) + (0,0,0)
- (5) P41; (a,b,c) + (0,1/2,0)
- (6) P41; (a,b,c) + (0,1/2,0)
- (7) C22'21; (a+b,-a+b,c) + (0,0,0)
- (8) C2'2'21; (a+b,-a+b,c) + (0,0,0)
- (9) C2221; (a+b,-a+b,c) + (0,0,0)
- (10) P21'21'21; (a,b,c) + (1/4,0,-1/8)



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

Solving the structure

GSAS-II project: Er2Ge207_op5K.gpx

Project: Er2Ge207_op5K.gpx

Magnetic atom selection

For: (1) P41'21'2; (a,b,c) + (0,0,0)

Name	x, y, z	allowed moments, mag. site sym:
Er1_0	0.87240 0.35190 0.13470	(Mx, My, Mz) 1

Use? Yes No Delete

Space group: P 41 21 2 Modulated?

Vol = 562.904

Pawley controls: Do Pawley refinement? dmin: 1.35714 dmax: 16.80354 Pawley neg. wt.: 0.0
d-space range 1.35629 to 16.80354 Å⁻¹ (1 histograms)

Fourier map controls: Map type: Reflection sets: Select reflection sets

Map grid step: 0.25 Peak cutoff %: 50.0

Charge flip controls: Reflection sets: Select reflection sets Normalizing element: None

Map grid step: 0.25 k-Factor (0.1-1.2): 0.1 k-Max (>=10.0): 20.0

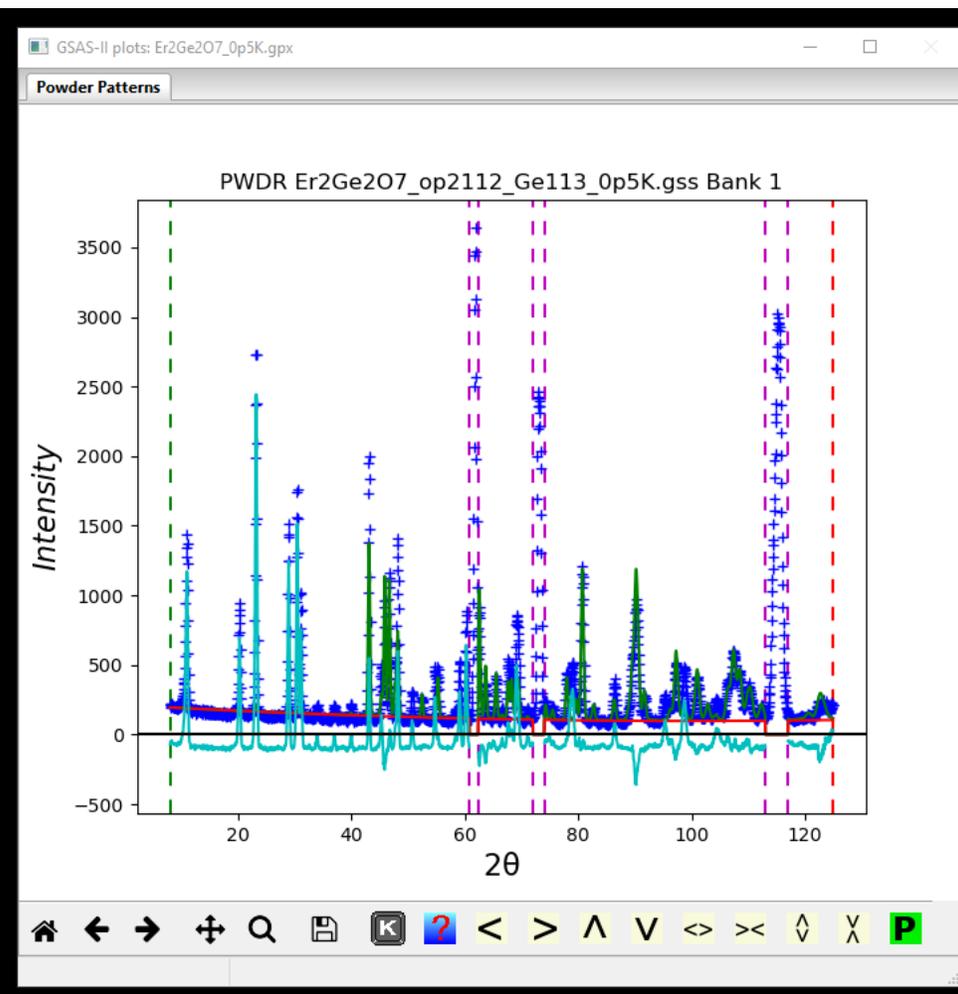
Test HKLs: 0 0 2 2 0 0 1 1 1 0 2 0 1 2 3

Monte Carlo/Simulated Annealing controls: Reflection set from: d-min: 2.8

MC/SA runs: 1 MC/SA Refine at 10.0 % of ranges.

MC/SA schedule: log slope: 0.9

Annealing schedule: Start temp: 0.7 Final temp: 0.1 No. trials: 250



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

Solving the structure

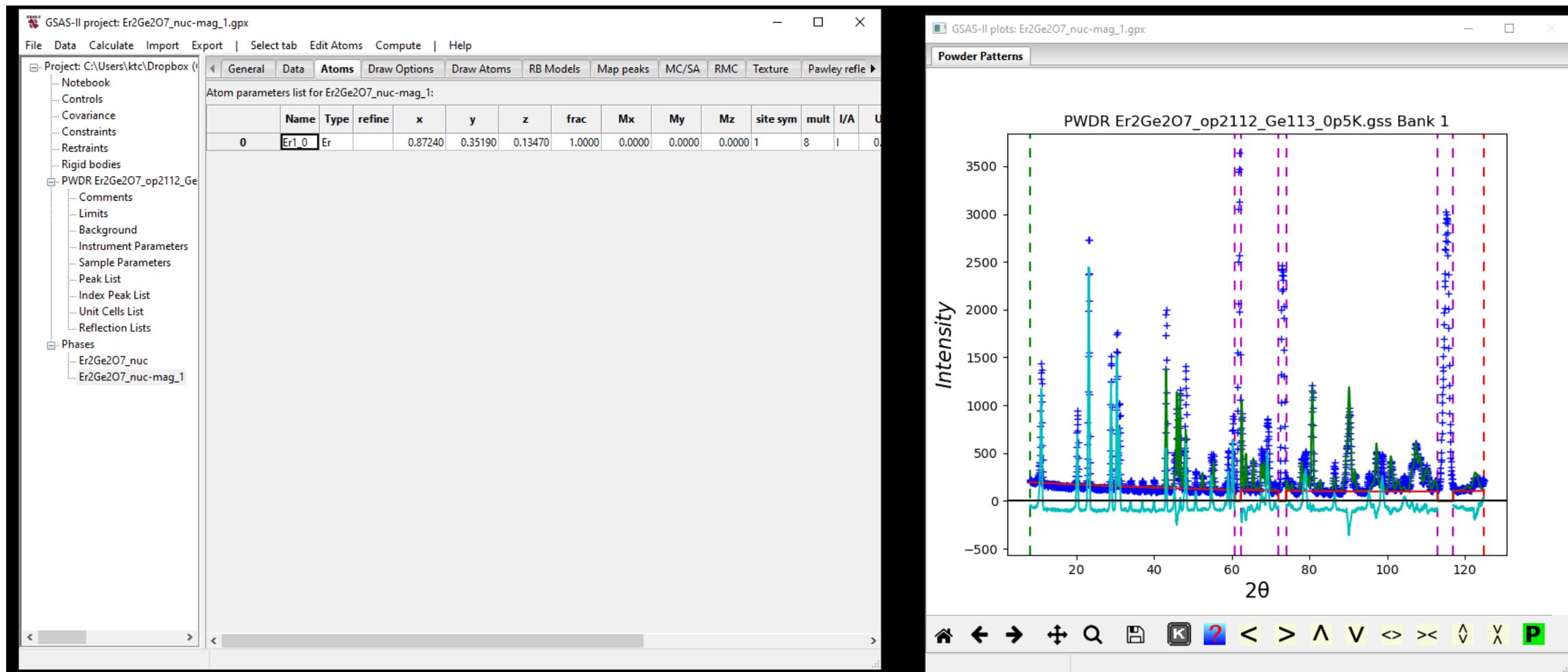
The image displays two windows from the GSAS-II software. The left window is a file explorer titled "Choose GSAS-II project file name" showing a list of project files in the "Er2Ge2O7" directory. The file "Er2Ge2O7_nuc-mag_1" is selected. The right window is a plot titled "Powder Patterns" showing a "PWDR Er2Ge2O7_op2112_Ge113_op5K.gss Bank 1". The plot shows Intensity versus 2θ with several peaks and vertical dashed lines indicating peak positions.

Name	Date	Type	Size
Er2Ge2O7_op5K.bak0	10/3/2022 10:12 AM	GSAS-II project	151 KB
Er2Ge2O7_op5K.bak1	10/3/2022 11:19 PM	GSAS-II project	150 KB
Er2Ge2O7_op5K.bak2	10/3/2022 11:19 PM	GSAS-II project	205 KB
Er2Ge2O7_op5K	10/3/2022 10:12 AM	GSAS-II project	254 KB
Er2Ge2O7_op5K_ma...	10/3/2022 10:31 AM	GSAS-II project	216 KB
Er2Ge2O7_op5K_ma...	10/3/2022 10:31 AM	GSAS-II project	237 KB
Er2Ge2O7_op5K_ma...	10/3/2022 10:32 AM	GSAS-II project	238 KB
Er2Ge2O7_op5K_ma...	10/3/2022 10:32 AM	GSAS-II project	239 KB
Er2Ge2O7_op5K_ma...	10/3/2022 10:32 AM	GSAS-II project	239 KB
Er2Ge2O7_op5K_ma...	10/3/2022 10:33 AM	GSAS-II project	239 KB

Map grid step: 0.25 | k-Factor (0.1-1.2): 0.1 | k-Max (>=10.0): 20.0
Test HKLs: 0 0 2 | 2 0 0 | 1 1 1 | 0 2 0 | 1 2 3
Monte Carlo/Simulated Annealing controls: Reflection set from: [] d-min: 2.8
MC/SA runs: 1 | MC/SA Refine at: 10.0 % of ranges.
MC/SA schedule: log | slope: 0.9
Annealing schedule: Start temp: 0.7 | Final temp: 0.1 | No. trials: 250

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

The image shows two windows from the GSAS-II software. The left window displays the 'Atom parameters list for Er2Ge2O7_nuc-mag_1:' with a table of parameters. The 'refine' column is set to 'M' for the Er1_0 atom, and the 'Mx', 'My', and 'Mz' columns are set to 3.0000, 3.0000, and -5.0000 respectively. The right window shows a 3D unit cell model with a green sphere representing the Er atom and a red cross representing the origin.

	Name	Type	refine	x	y	z	frac	Mx	My	Mz	site sym	mult	I/A	U
0	Er1_0	Er	M	0.87240	0.35190	0.13470	1.0000	3.0000	3.0000	-5.0000	1	8	I	0

Let's flag these for refinement (set the refinement column to M), give them nominal starting values (I'll do $M_x, M_y, M_z = 3, 3, -5$) 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

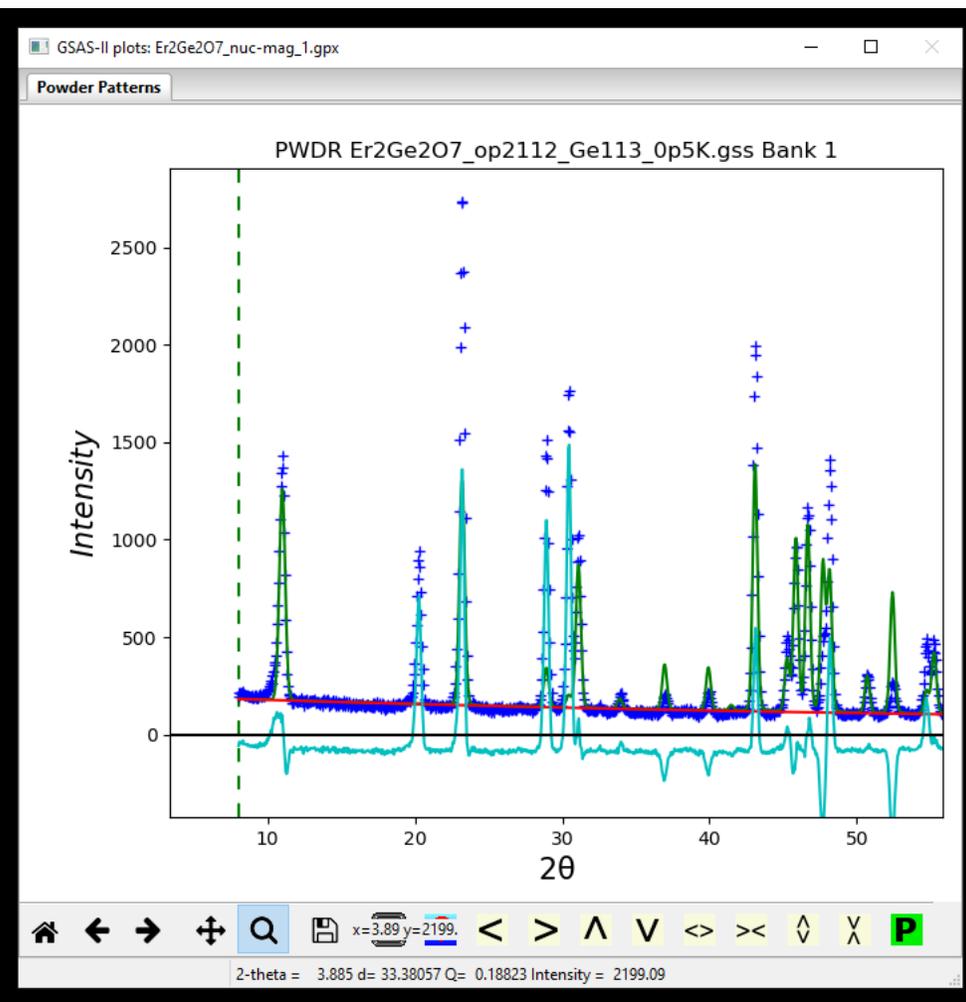
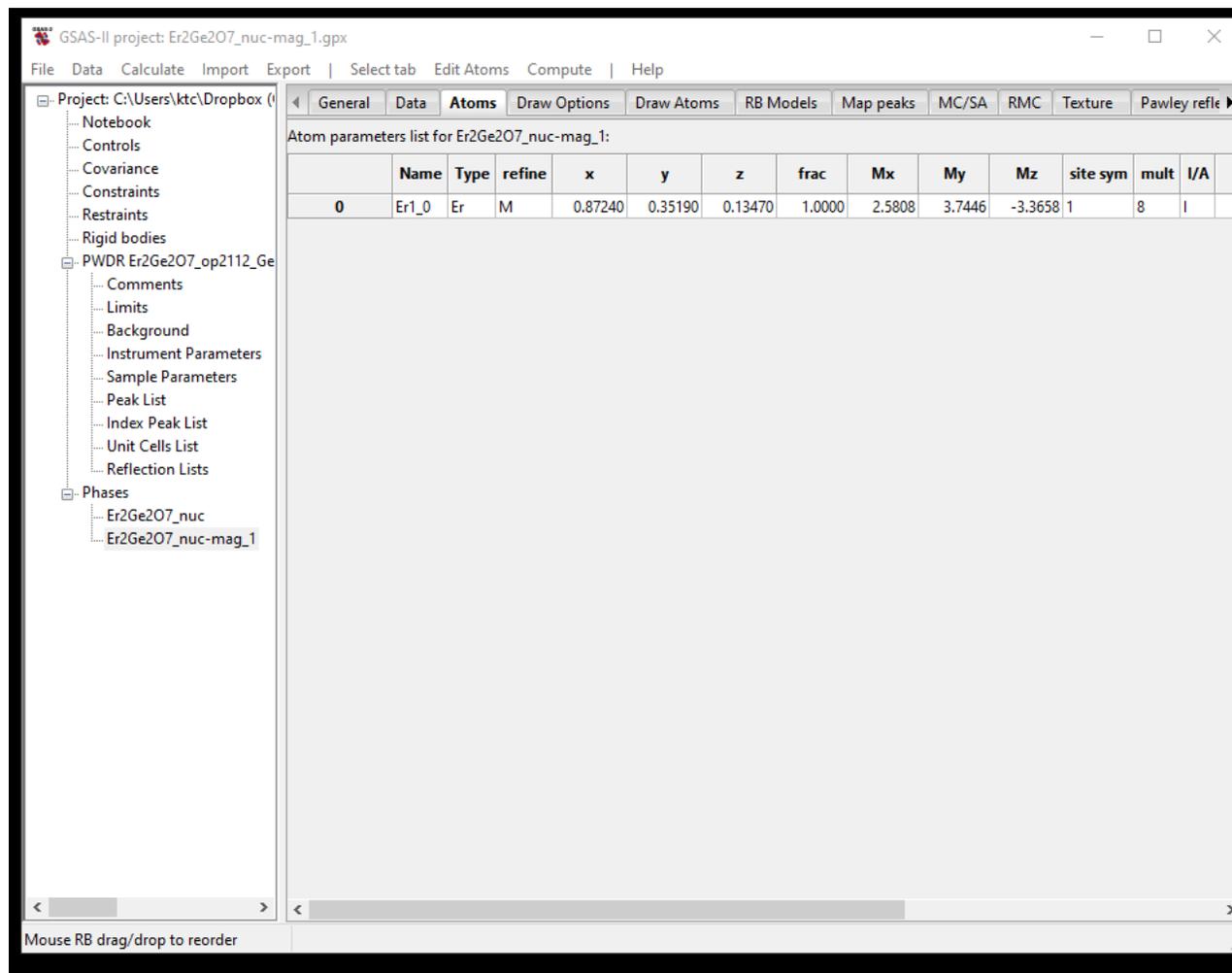
The image shows two windows from the GSAS-II software. The left window is the main project window titled 'GSAS-II project: Er2Ge207_nuc-mag_1.gpx'. The 'Calculate' menu is open, and the 'Refine' option (with keyboard shortcut Ctrl+R) is highlighted. Below the menu, a table displays the current atomic parameters for the Er atom:

Atom	Type	refine	x	y	z	frac	Mx	My	Mz	site sym	mult	I/A	U
Er	M		0.87240	0.35190	0.13470	1.0000	3.0000	3.0000	-5.0000	1	8	1	0

The right window is titled 'GSAS-II plots: Er2Ge207_nuc-mag_1.gpx' and shows a 3D visualization of the unit cell. The cell is a rectangular prism with axes colored red, green, and blue. A green sphere representing the Er atom is positioned inside the cell, with a grey rod extending from the center to its location. A red crosshair is visible at the center of the cell. The status bar at the bottom of the plot window shows 'View point: 0.5000, 0.5000, 0.5000; density: 0.0000'.

Let's try a refinement

Solving the structure: refining the moments



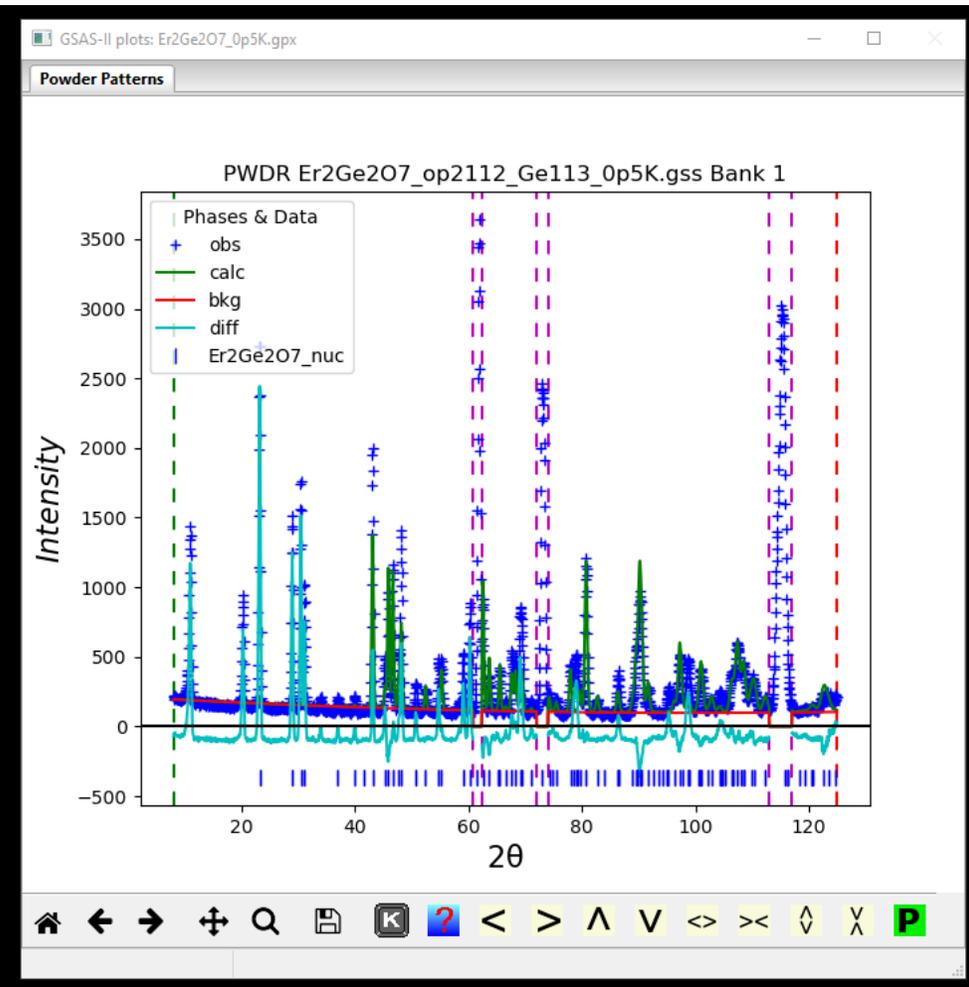
This doesn't look so great, intensity is off but we are also completely missing some peaks.
Let's try the second structure

Solving the structure: iterating through the structures

The screenshot shows the GSAS-II software interface for a project named 'Er2Ge207_0p5K.gpx'. The 'General' tab is active, displaying the following information:

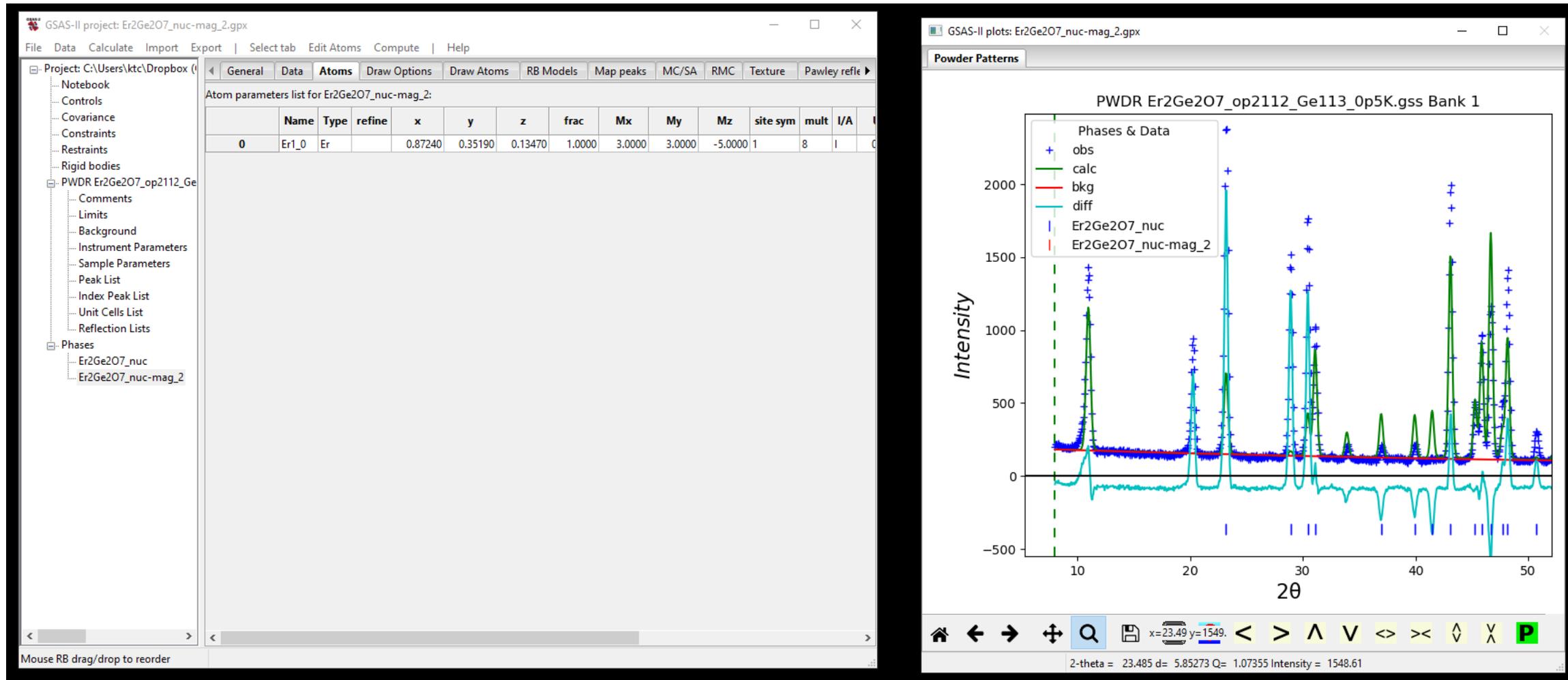
- Phase name: Er2Ge207_nuc
- Phase type: nuclear
- Space group: P 41 21 2
- Refine unit cell: a = 6.76494, c = 12.30005, Vol = 562.904
- Density: 6.982
- Elements: Er, Ge, O
- Isotope: Nat. Abund. (dropdowns)
- No. per cell: 8.0
- Atom weight: 167.2
- Bond radii: 1.96
- Angle radii: 1.76
- van der Waals radii: 1.70
- Default color: Green
- Pawley controls: Do Pawley (checkbox), Pawley neg. wt.: 0.0
- Fourier map controls: Map grid step: 0.25
- Charge flip controls: Refl. (checkbox), k-Factor (0.1-1.2): 0.1, k-Max (>=10.0): 20.0
- Map grid step: 0.25
- Test HKLs: 0 0 2, 2 0 0, 1 1 1, 0 2 0, 1 2 3
- Monte Carlo/Simulated Annealing controls: Reflection set from: (dropdown), d-min: 2.8
- MC/SA runs: 1, MC/SA Refine at: 10.0 % of ranges.
- MC/SA schedule: log, slope: 0.9
- Annealing schedule: Start temp: 0.7, Final temp: 0.1, No. trials: 250

A dialog box titled 'Make new magnetic phase' is open, showing a list of magnetic space groups. The second option, '(2) P4121'2; (a,b,c) + (0,0,0)', is selected.



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

Solving the structure: iterating through the structures



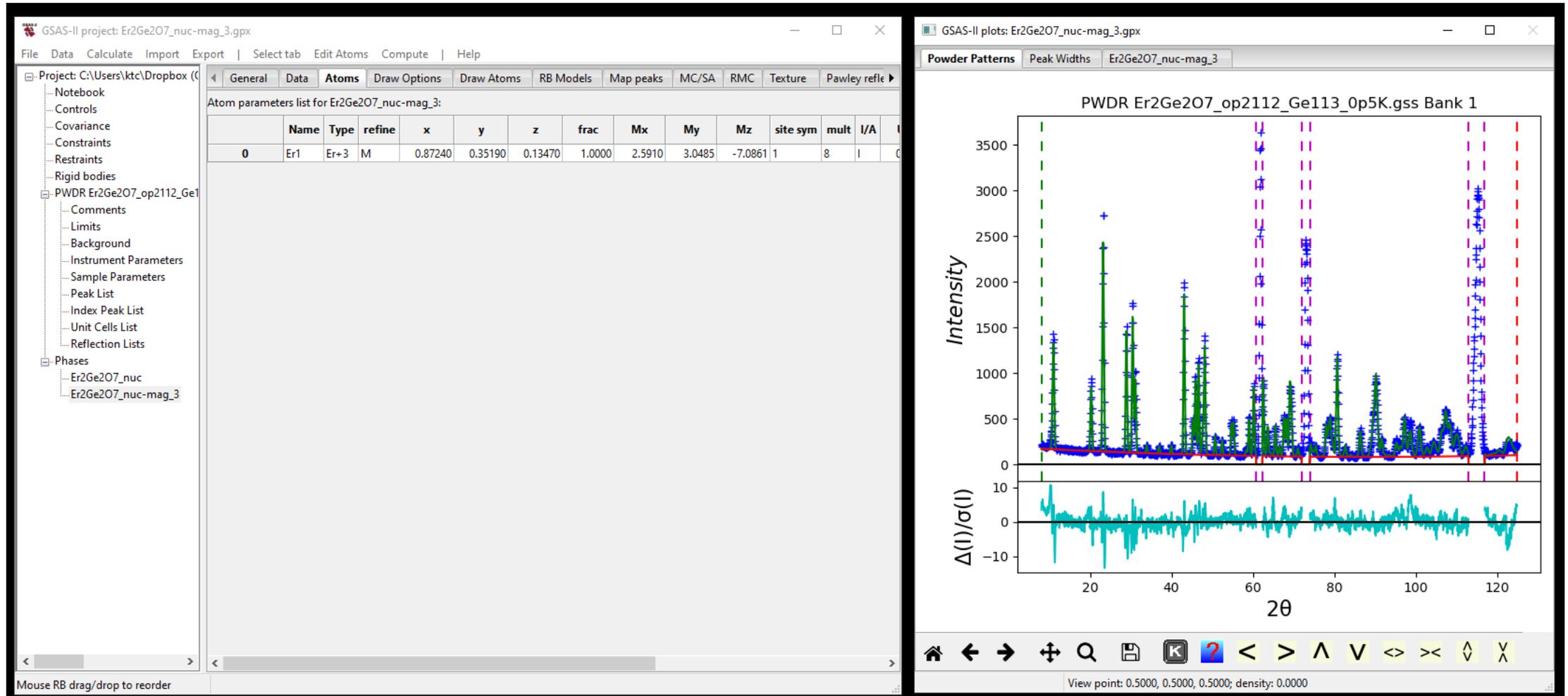
Also not even close, let's try the third.

Solving the structure

The image displays two windows from the GSAS-II software. The left window, titled 'GSAS-II project: Er2Ge2O7_op5K.gpx', shows the 'General' tab for the phase 'Er2Ge2O7_nuc'. The phase type is 'nuclear' and the space group is 'P 41 21 2'. The unit cell parameters are a = 6.76494, c = 12.30005, and Vol = 562.904. The density is 6.982. The elements are Er, Ge, and O. A dialog box titled 'Make new magnetic phase' is open, showing a list of ten magnetic space groups. The third option, 'P41'212'; (a,b,c) + (0,0,0)', is selected. The right window, titled 'GSAS-II plots: Er2Ge2O7_op5K.gpx', shows the 'Powder Patterns' for 'PWDR Er2Ge2O7_op2112_Ge113_op5K.gss Bank 1'. The plot shows Intensity vs. d, Å, with a fitted curve and a residual plot below it. The x-axis ranges from 2 to 16 Å, and the y-axis ranges from 0 to 3500. The plot shows several sharp peaks, with the most intense peak at approximately 12.3 Å. The residual plot shows the difference between the observed and fitted data, with a maximum deviation of approximately 50 units.

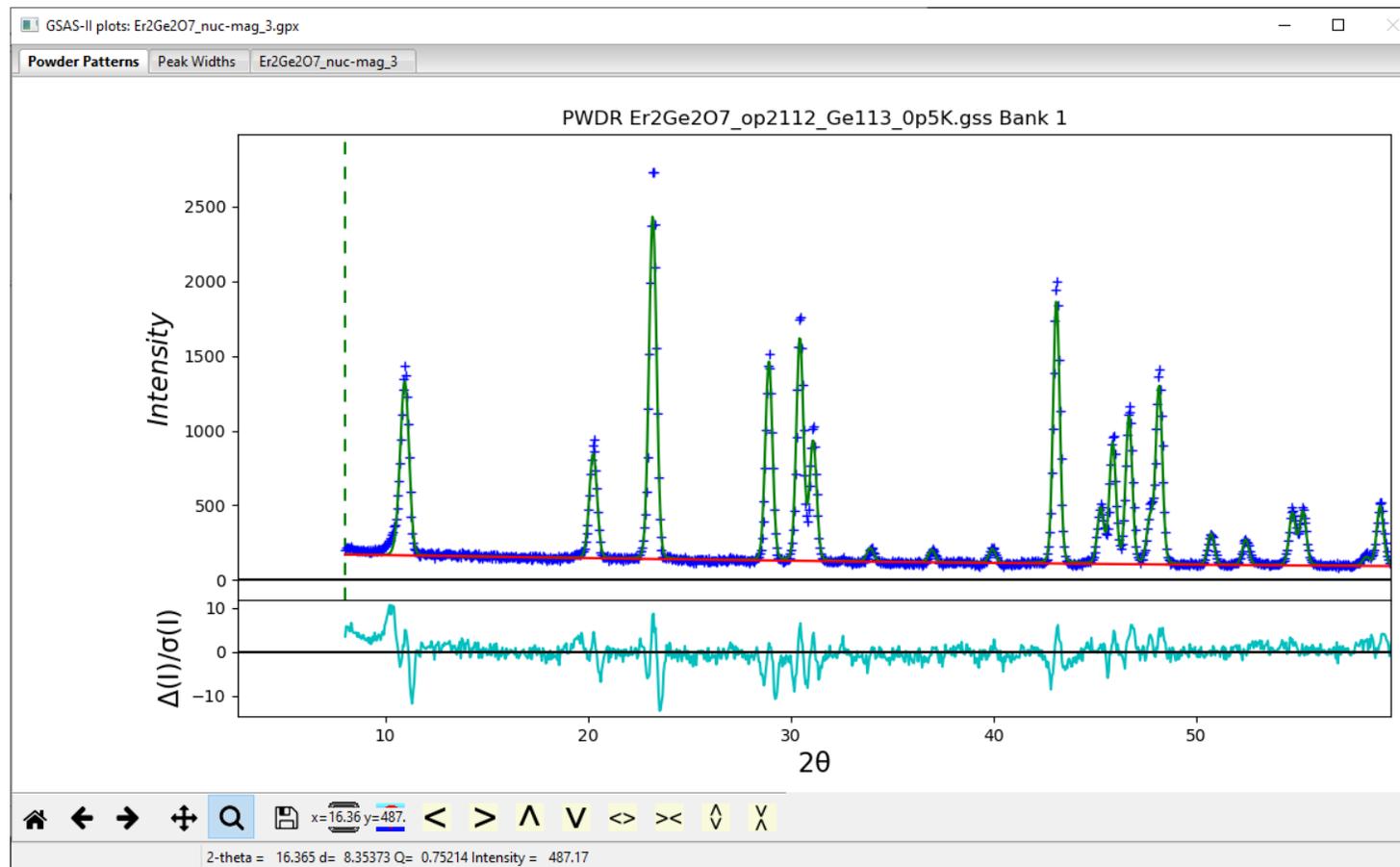
Go back to your 0.5 K save and try the third structure

Solving the structure: refining the moments



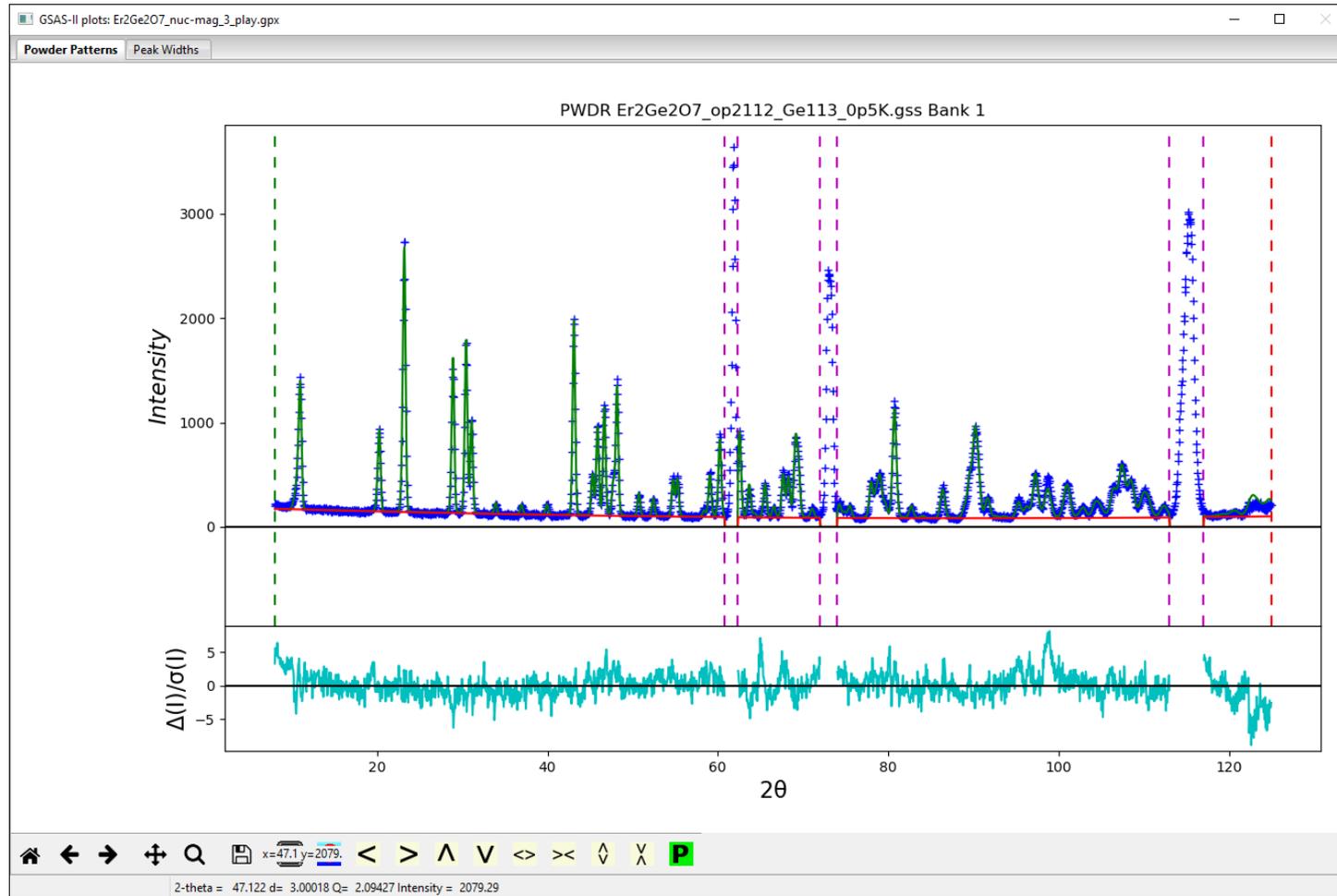
This one looks quite good

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!

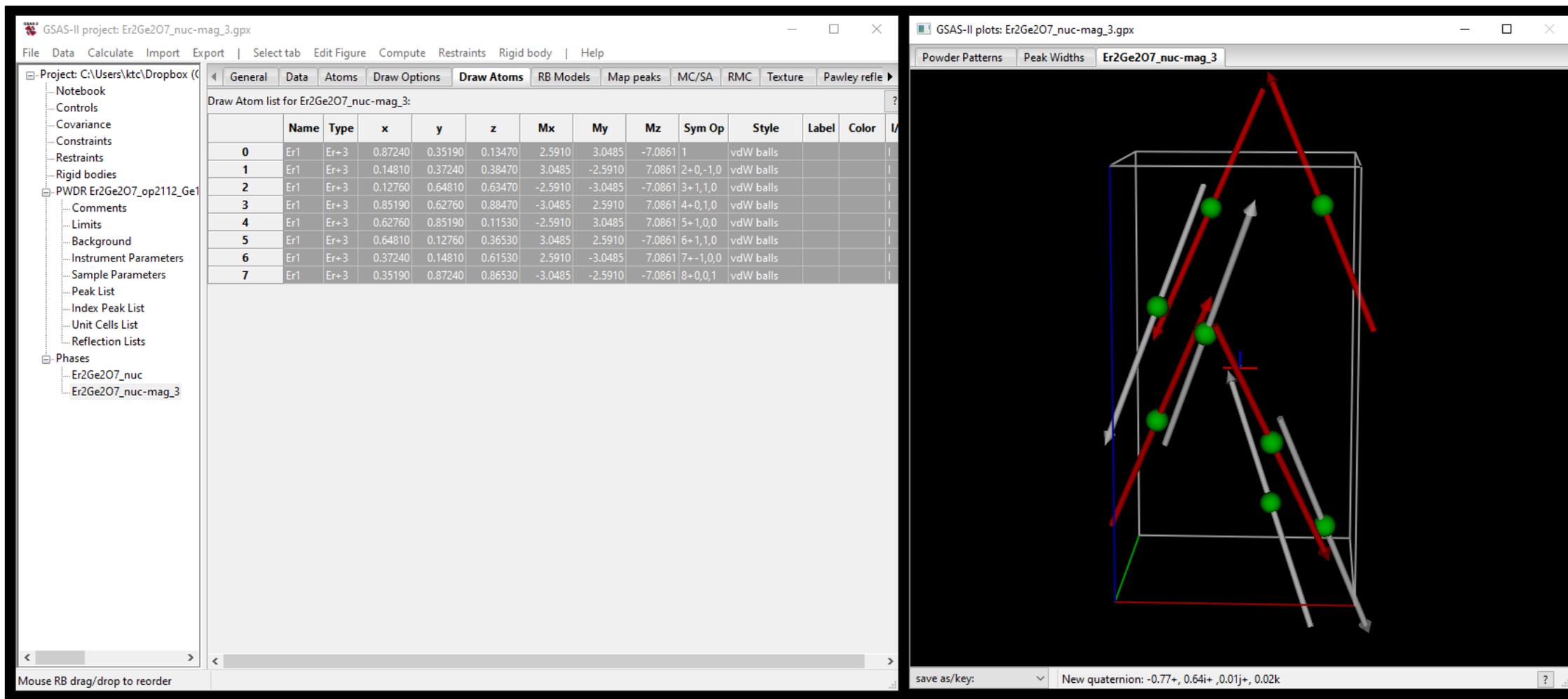
Visualizing the magnetic structure

The image shows two windows from the GSAS-II software. The left window, titled 'GSAS-II project: Er2Ge2O7_nuc-mag_3.gpx', displays the 'Draw Atoms' tab. A red circle highlights the 'Draw Atoms' button in the top menu. Below the menu, a table titled 'Draw Atom list for Er2Ge2O7_nuc-mag_3:' is visible. The table has columns for Name, Type, x, y, z, Mx, My, Mz, Sym Op, Style, Label, Color, and I/A. The first row shows an atom with Name '0', Type 'Er1', and coordinates (0.87240, 0.35190, 0.13470). The right window, titled 'GSAS-II plots: Er2Ge2O7_nuc-mag_3.gpx', shows a 3D visualization of the magnetic structure. It features a unit cell with axes colored red, green, and blue. A green cross is centered in the cell, and a green sphere is connected to the center by a grey arrow, representing the magnetic structure.

	Name	Type	x	y	z	Mx	My	Mz	Sym Op	Style	Label	Color	I/A
0	Er1	Er+3	0.87240	0.35190	0.13470	2.5910	3.0485	-7.0861	1	vdW balls			

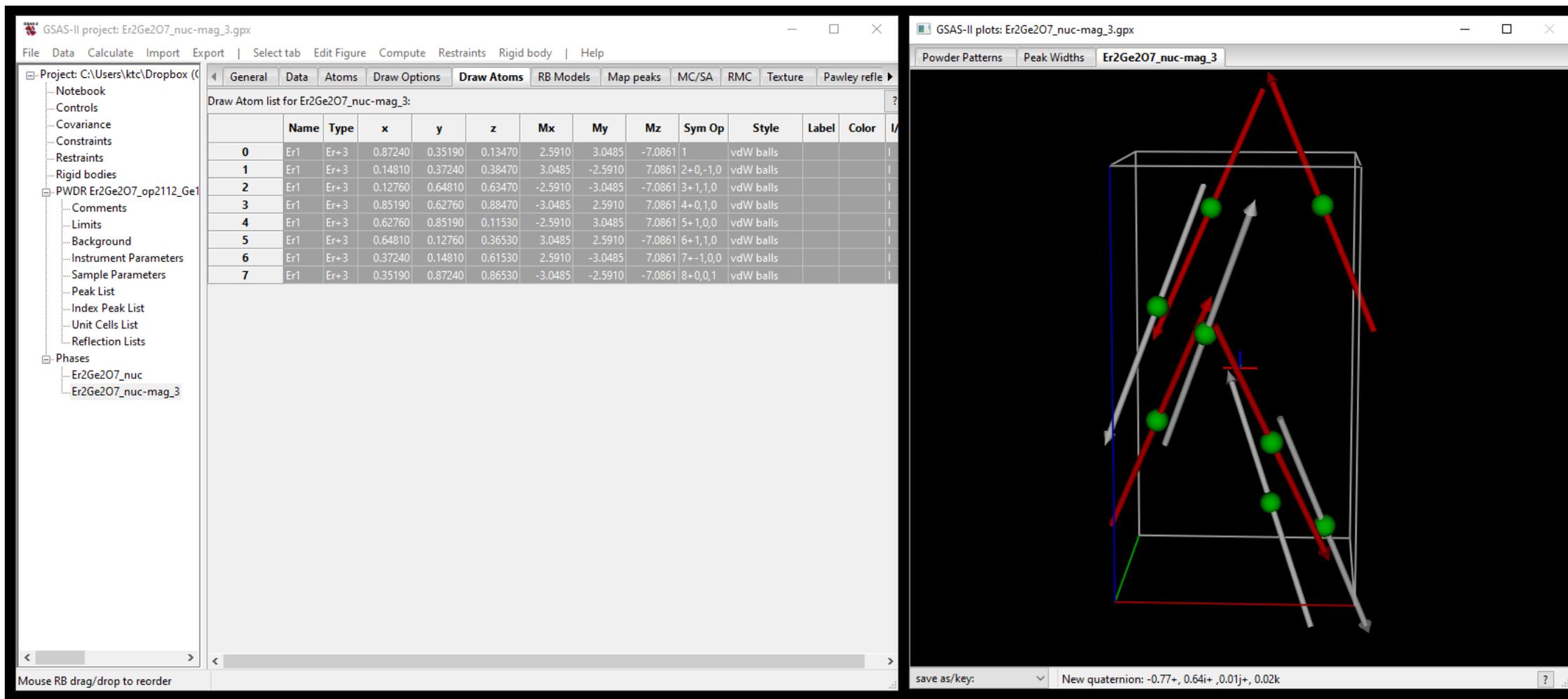
To see what this looks like click on the 'Draw Atoms' tab for the magnetic phase.
Then use the same trick as before to show all the atoms

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

