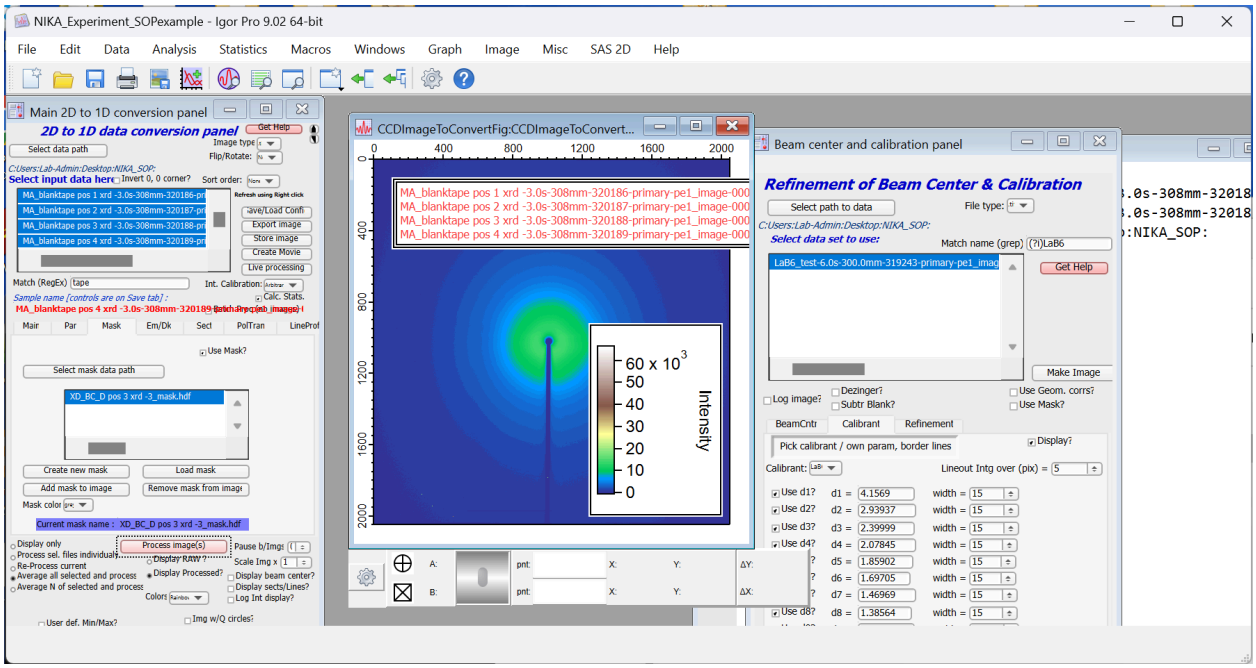
STEP 1: Convert 2-D to 1-D

Laue patterns are converted to 1-D diffractograms by integrating the intensity along each circumference for each ring - the lowest 2-theta is closest to the center.

Downloading Igor Pro and the Nika Package:

Once you have your .tiff files, install Igor Pro (version 9 or later). This program can be downloaded from wave metrics here https://www.wavemetrics.com/order/order_igordownloads.htm. Once you have Igor Pro pulled up on your computer, you'll need to download a package (referred to as a 'macro') called Nika, which enables 2D to 1D data reduction. There is a really helpful Youtube video that is old but still applicable (https://youtube.com/watch?v=ckggsCl0T9E), which shows you how to install Nika. Essentially, you go to the 'install packages' tab at the top of the screen and select 'Open Github GUI' which will open up a panel with instructions. I found it easiest to install directly from the Github repository, and that's what I would suggest for anyone doing this, and feel free to send us an email if any questions come up.



Although Igor/Nika will be the focus here, Dioptas is still helpful for looking at the CCD images and the diffractogram side by side and seeing which rings correspond to which peaks. It might be a good practice to look at each sample in both softwares. There is a very well-written Dioptas tutorial that's publicly available online. https://dioptas.readthedocs.io/en/stable/index.html

If you're using Dioptas for 2D to 1D conversion, make sure to save the diffractogram file as .chi.

Working in Nika:

Igor and Nika are both complex programs with many capabilities and functions, and this tutorial is meant to use Nika for the 2D to 1D conversion step. To streamline this process, the use of a preexisting Nika file (referred to as an 'experiment') is highly recommended, and you can find one associated with this document. Within the experiment, there are several different windows. Among them, the 'Beam center and calibration panel' and 'Main 2D to 1D conversion panel' are most important.

Refinement of Beam Center and Calibration:

First, take a look at the beam center and calibration panel. Nearest the top, there is a button that says 'Select path to data.' This is where you select your local folder containing your 2D data (the same goes for the Main 2D to 1D conversion panel). To the right of that button, you can select your file type (.tif for our purposes), and just below that you should see your .tif files populate. If you don't see them at first, make sure that the right path is selected and that your files are in the right format. About halfway down the panel, you'll see three different tabs: Beam Center, Calibrant, and Refinement. For the beam center, these values are typically obtained every day for a beamline using the calibrant (LaB6), which yields an image with rings that may not be perfectly circular, and the system corrects itself according to the dimensions of the calibrant rings.

Open the laB6 file in the refinement of beam center and calibration window and then use the poni file to find the values to enter into the refinement of beam center and calibration window - You can click on laB6 in the refinement of beam center and calibration window to open up the image

- Sa-det distance (mm) is the 'Distance' value (FYI our February 2024 beam was 500 microns in diameter)
- Refine it again to make sure everything is nice and centered
- If you let Nika do the calibration, values will auto populate in the 2D to 1D conversion panel
- If you lose any of the panels, just go into SAS 2D and reopen it, you can get a better view

Still in main panel:

Sect tab:

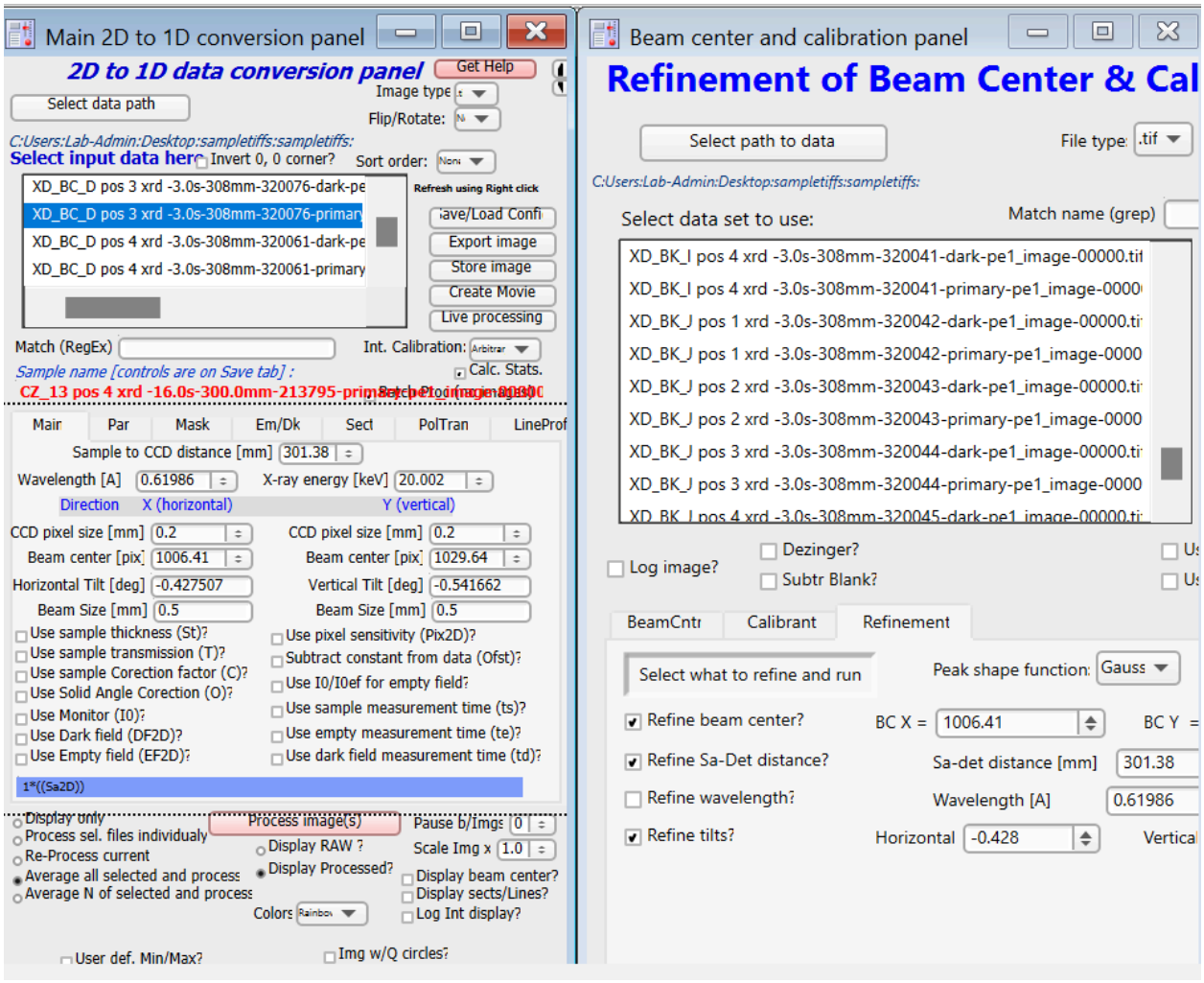
Number of points: set this based on pixel size (200 microns) and the size of the detector, this determines the number of points on your x-y plot

1250 is a good place to start
PolTran – you could perform the analysis with just a pie slice of the image

- This function normally isn't worth it because you might as well use the entire image to get a representative diffractogram; however, this may be helpful if there is excessive noise or distortions associated with a certain region of the tif file

Save/Exp – this is where you select where you want things to save
Here you can choose to take part of the annoying long name off
Look at the 1D output: if there is funky noise in the low thetas (right now we're getting a point for every pixel), we can lessen the number of points in the Sect tab to reduce this noise.

This calibration process provides the beam center (X and Y), which changes regularly and needs to be updated. Please contact Professor Rob Root (rroot@arizona.edu) for information regarding the beam center for your specific analyses. Again, make sure that under the calibrant tab, that LaB6 is selected as the calibrant, and the d spacings should be automatically populated with NIST-certified values. When performing this refinement, refine the beam center and the Sa-Det distance separately using estimates. (you can't do them at the same time).



Up until this point, we have focused exclusively on the laB6 file for calibrations and preparing your system for the 2D to 1D conversion, and now we're ready to look at the .tif files of your samples.

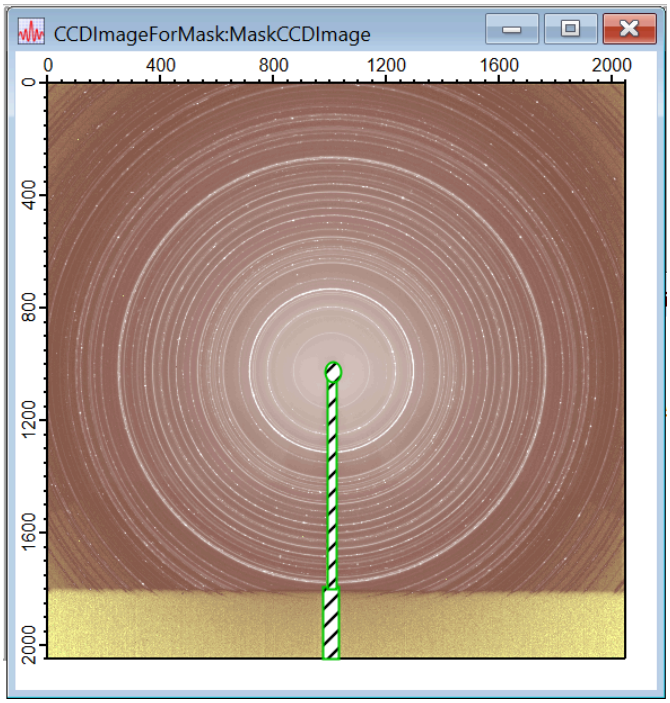
Processing the .tif files of your samples

Once you've selected your data path and see your sample/image names populate in the window, you can select one (note: make sure to select and analyze only the primary images, the images with 'dark' in the name should not be analyzed in this way. Under the main panel, the wavelength in angstroms (which can be calculated based on the energy of the beam in keV), CCD pixel size, beam center, tilt and beam size should be entered manually (check with Rob to make sure you have the right values for the specific day your samples were analyzed). Down by the bottom of the panel, select the 'Average all selected and process' so that you can select all 4 of your beam points per sample (the corners of our coordinate system) and also select 'Display Processed?' so that you can also see the averaged image. Before hitting the red 'Process image(s)

button, toggle over to the ‘Mask’ tab so that we can hide the beam stop from our processing techniques.

Creating a Mask:

Once your images are showing up under ‘Select data set to use:’ highlight one of your images (it doesn’t matter which one) and click ‘Make Image,’ then click ‘Start MASK Draw,’ which will allow you to draw a mask around the beam stop. We want to trace around the vertical rod from the base of the image all the way up to the center of the image; this involves drawing boxes that together serve to cover up the beam stop. I normally draw a thicker rectangle at the very bottom with a thinner one on top of that and finally a small circle on top of that (see example below).



Once you’re happy with your mask (trying not to cover up any more of the image besides the beam stop), click ‘Finish MASK’ and then type in a name for your mask and click ‘Save MASK.’ Now, for each of your .tif images you can simply load this mask prior to image processing. Next, since Nika is sensitive to the amount of characters in any given file name, we’ll have to go and cut down our file names to only show what we need to properly identify them. For example, the file names from BNL read something like ‘XD_BC_D pos 3 xrd -3.0s-308mm-320061-primary-pe1_image-00000.tif,’ and Nika does not like this. In the folder you specified as your path, edit the file names to just include what you need. Once you do this and you’ve highlighted the 4 (or however many) in the ‘Main 2D to 1D data conversion panel,’ you’re almost ready to process your image, but first there are a few more steps: 1) select all of the replicate .tiff files you

want to average and (on the Main 2D to 1D conversion panel, at the bottom of the panel) check 'Display only' and 'Display RAW.' This will effectively load the raw, averaged images into Nika and it will pop up with the names of the replicates at the top of the image (this is how you can confirm that the image is an average of your replicates and not just an individual replicate. Once you see the image pop up (still in the Main 2D to 1D conversion panel under the 'Mask' tab, NOT the 'Create MASK panel,' click 'Add mask to image,' and select the correct path to the mask file you created. You should see the mask pop up in the averaged image you generated. It is very important that you pull up the images first before converting them, and once you have the averaged CCD image with as many replicates as you collected for that sample, you can check 'Average all selected and process' and 'display processed,' which should provide a 'LineoutDisplayPlot' with your two theta vs. intensity 2D plot.

For clays: Sect – q, d, or 2 theta, select d for d space, this is helpful for looking at clays

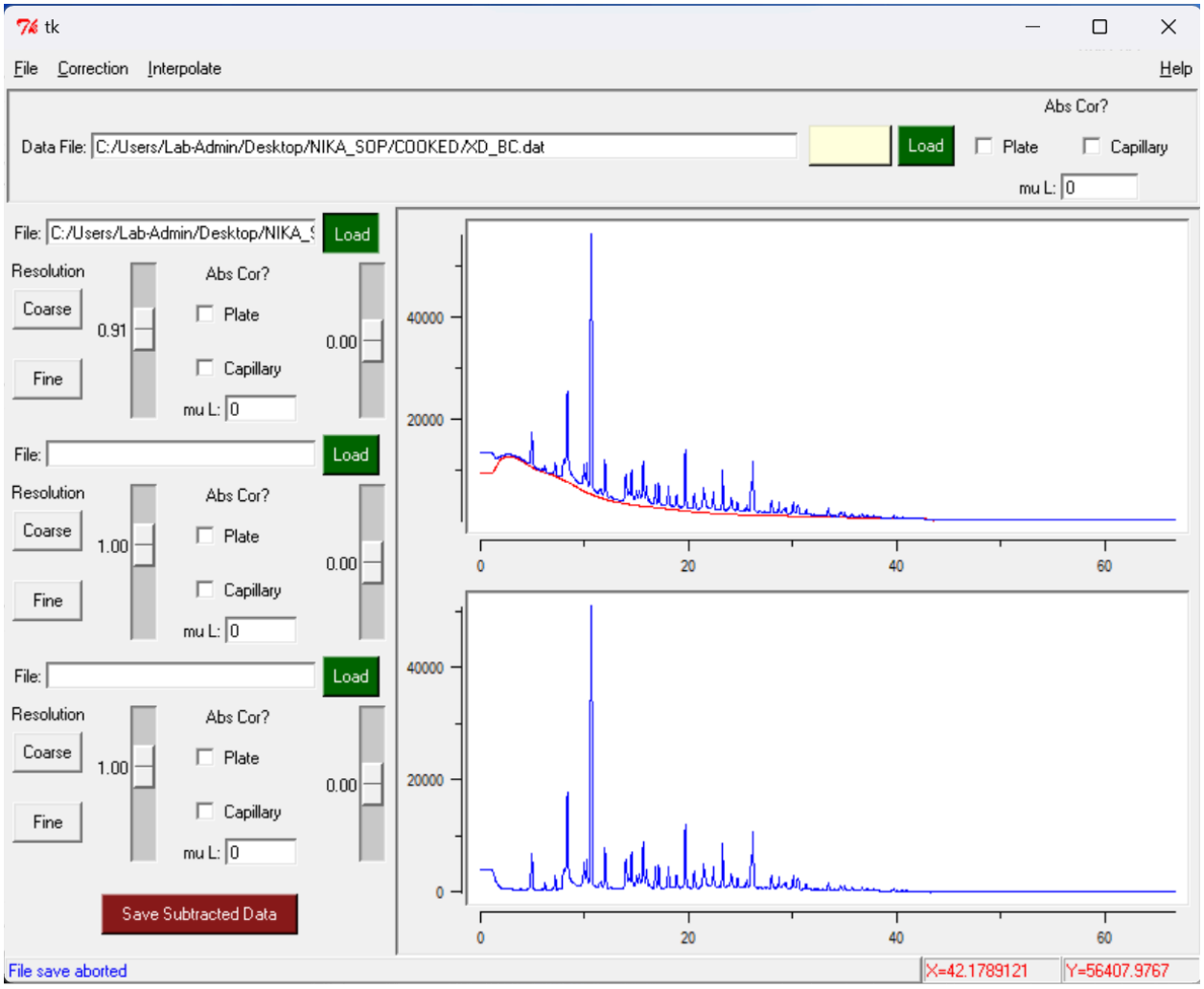
Moving into XRD BS:

- A few additional notes: when using the laB6 to calibrate, make sure that you create an average image from the 4 points, rather than just picking one of the 4 sampling points arbitrarily
- Before you proceed to the next step, you also need to average the 4 sampling points of the blank/tape and convert that into 1D so that we have something to subtract in the BS program (making sure to apply mask to the image beforehand)
- Now that you have a 1D xy file for the blank tape, we can subtract this from all our averaged xy files using the BS software

STEP 2: Remove background

The sloping background is from the tape support, Compton scattering, air scattering, and sample absorption. We collected an empty tape that can be used to subtract the background to create a "flat image". To subtract the tape and misc. scattering we use ***XRD_bs,*** free software from me. The contribution of the tape background can be scaled with a slider to fit your data (below blue is data, red is background). XRD_bs **only** reads ascii (2 column) data files with the extension .dat or .chi. You need to strip the header and save as chi or dat (not txt) using ***Notepad*** or similar.

The background subtracted (bs) file is then converted from synchrotron radiation to Cu ka. Since lambda=2d sin theta, the diffractograms converted from Laue patterns need to be converted from 20.0 keV (0.62 angstrom wavelength) to Cu ka radiation (1.5406 ang) to allow comparisons to conventional reference databases. If you prefer to work in d-space or reciprocal q-space (advanced user) this conversion is not necessary.



BS software download (.exe)

Once you have the BS (background subtract) software downloaded on your computer, you need to make some modifications to the .dat files you created in Igor before you can load them in. In your Nika-generated xy .dat files, there is quite a bit of overhead text that needs to be removed, otherwise the software will get confused. So, pull up the 1D (x-y) .dat files (texteditor will suffice for this) and delete all the comments at the top of the files. After this step, you should see four columns of numbers and that's it. Now you're ready to load them into the software.

Begin at the top of the BS program window and click on the blank white button to the left of the green load button – this will allow you to find the path to your mineral 1D xy file. Once you've selected it and the path shows up in the white bar to the right of 'Data file,' you can click the green load button to the right of that and your 1D XRD mineral data will show up in the upper portion of the program window. Now, on the left side of the program window you'll find similar load buttons. Since we are only making one subtraction per sample we only need one of these, and you can use the top one. Load your tape 1D .dat file here and you will see it appear as a red line below the mineral 1D data line. On the lower half of the screen you will find the subtracted version of the 1D data – this is the raw XRD mineral data minus the tape XRD data.

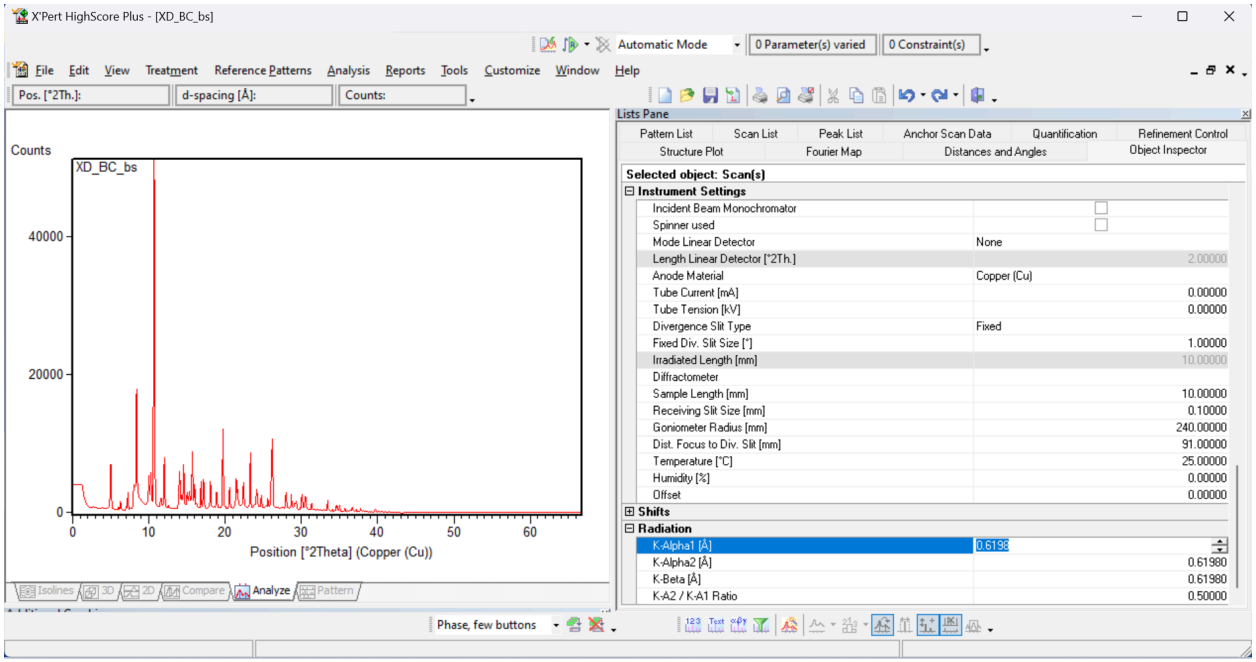
There are a few adjustments that need to be made to ensure that we are fully subtracting the tape signal from the raw data without over-subtracting and compromising the data. Below the path of your 1D tape . dat file there are two sliders that you can adjust to optimize the tape subtraction – for our purposes we will only use the one on the left. First, play around with this slider to see what it does – you'll find that as the slider goes up, so does the red tape line. We want this red line to be as close to our blue mineral 1D data as possible without passing it. Start by looking at the top part of the screen with the two lines and bring the red line up to the bottom of the blue line but not high enough so they're touching. Then, look at the bottom part of the screen where the subtracted pattern is. If you slide the slider too far up, you'll see that the resulting line will no longer be flat, the right side will slide up, and the left side will slide down, creating a distortion. As the red line approaches the blue line from below, you'll see the left side of the bottom blue line will get lower and lower until finally it crosses the zero line, at which point the right side of the blue line will swing up. This is the point where the red line exceeds the blue line in the upper plot. So, we can use both of these plots to make sure that we are fully subtracting the tape signal without subtracting any of our mineral signal. Once you've moved the slider to a good spot, click the red 'Save Subtracted Data' as an **.asc file**. This is the file that we will use in the next program, X'Pert HighScore Plus. It may be helpful to put these BS-processed files in a unique post-BS folder, especially if you have a lot of data.

Weird note: when the BS program is pulled up, it also pulls up this blank black window that almost looks like a chrome browser window – don't close this.

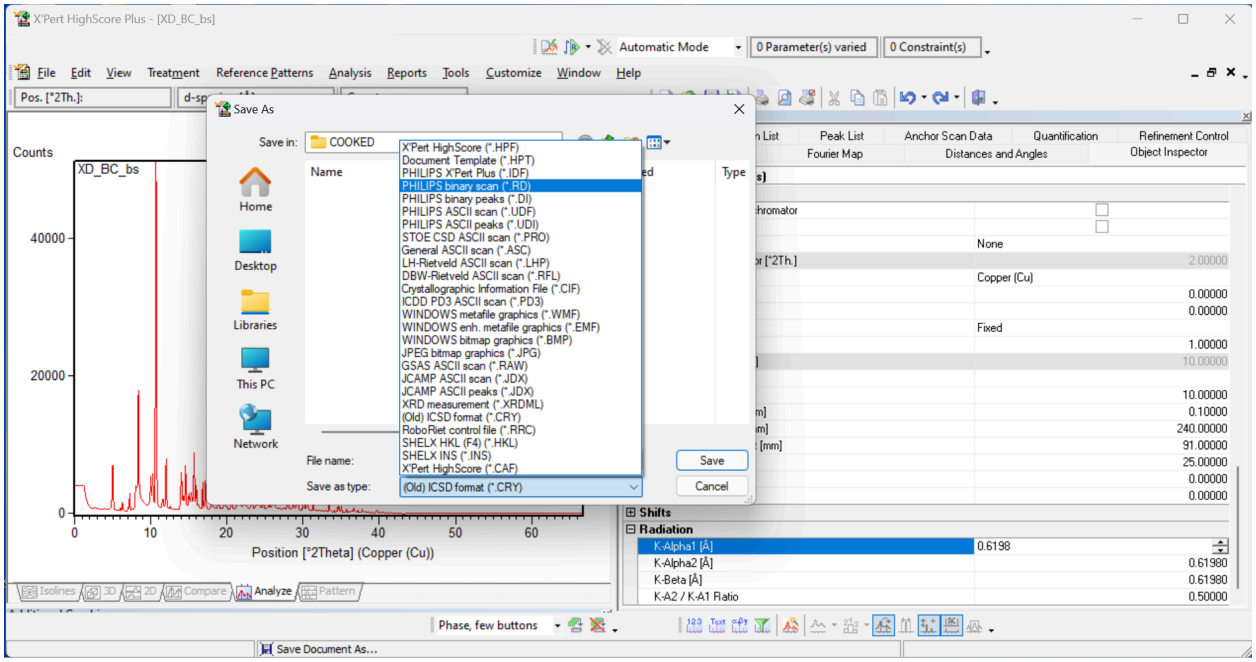
Working through X'Pert HighScore Plus

First, go to file open to open the **.asc file** you just created, then click on the 'Scan List' tab to the right of the screen. For K-alpha1, K-alpha2, and K-beta, type in our energy

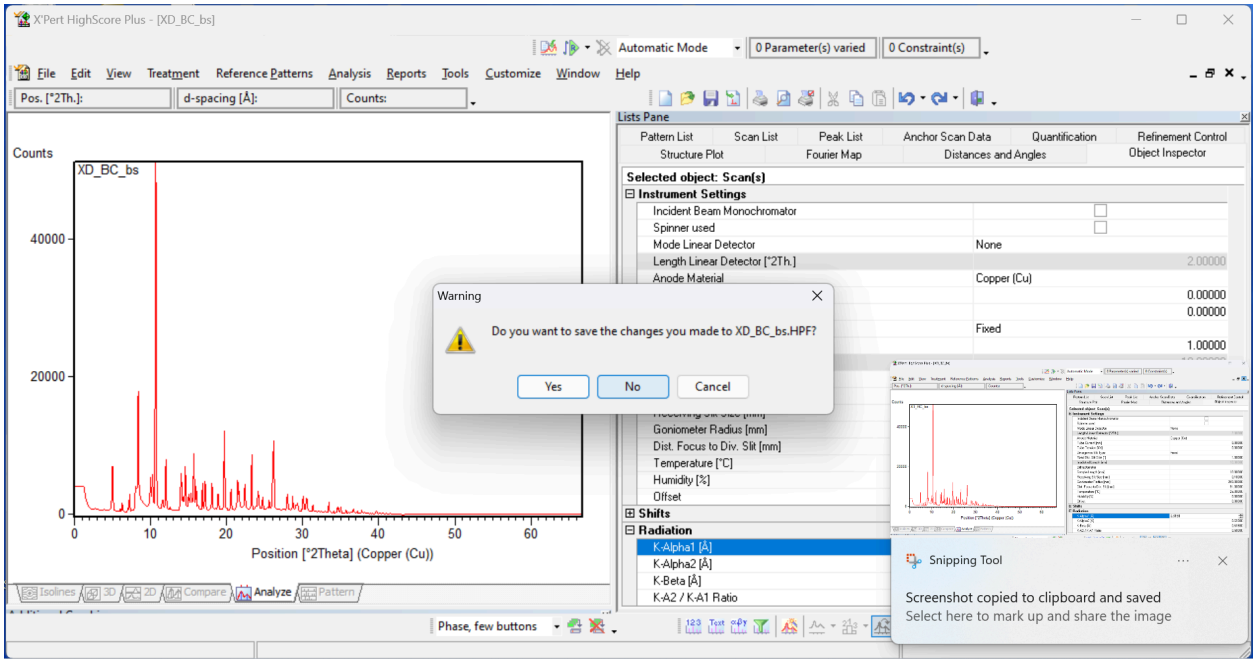
(for example, February 1st was 0.6198). This is the wavelength. Energy and wavelength are interchangeable with Plank's constant and speed of light



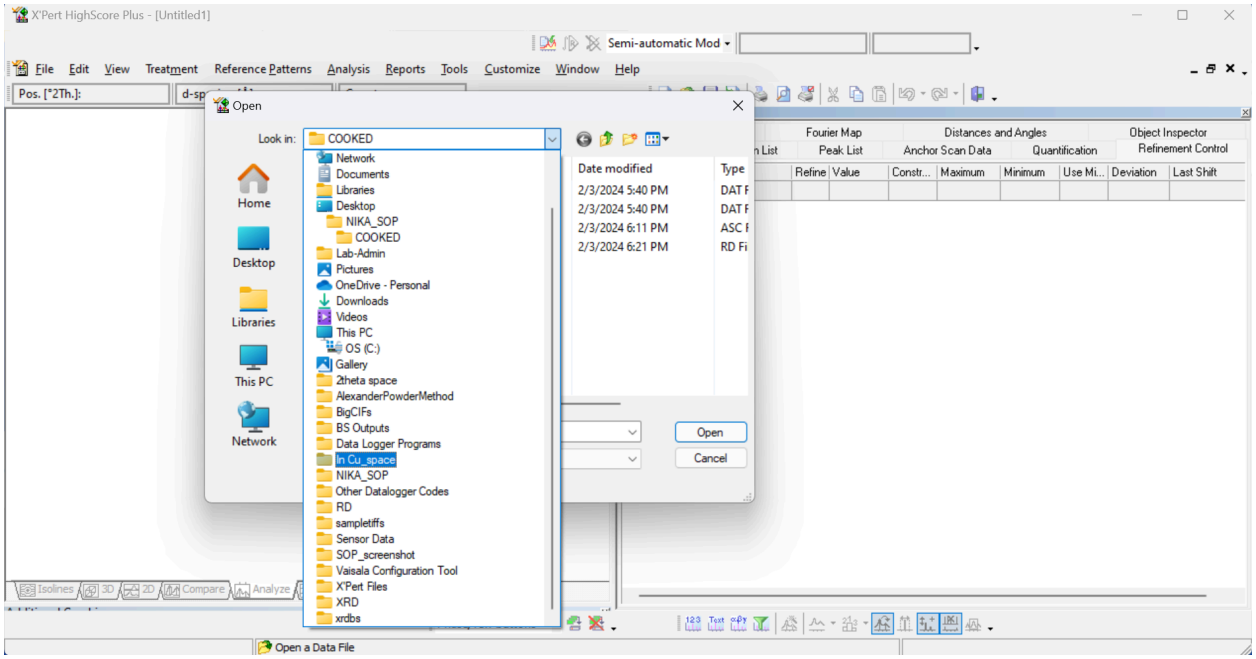
Once you've entered in the correct values, click file and 'save as' and save this file as a .RD file.



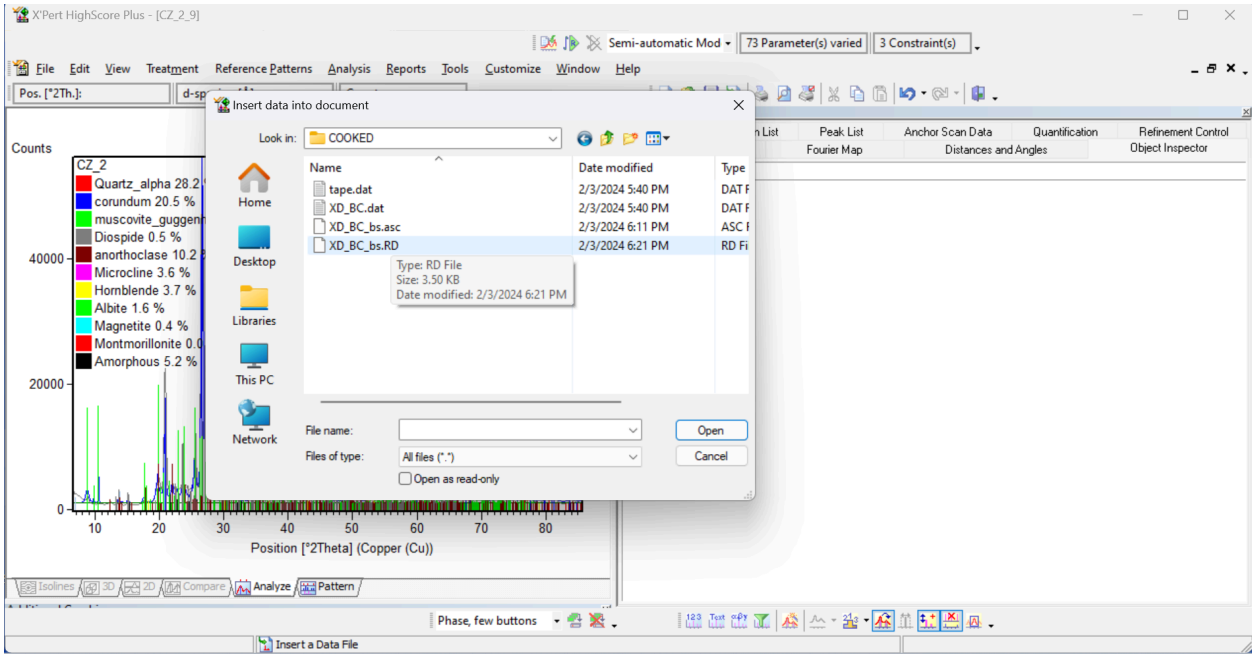
With all these different file types, I recommend creating a separate folder titled ‘RD’ on your computer so that everything stays organized. Once you’ve saved it as a .RD file, you can close out of this X’Pert HighScore Plus file (.HPF). When the program asks if you want to save changes to the .HPF file, click ‘no.’



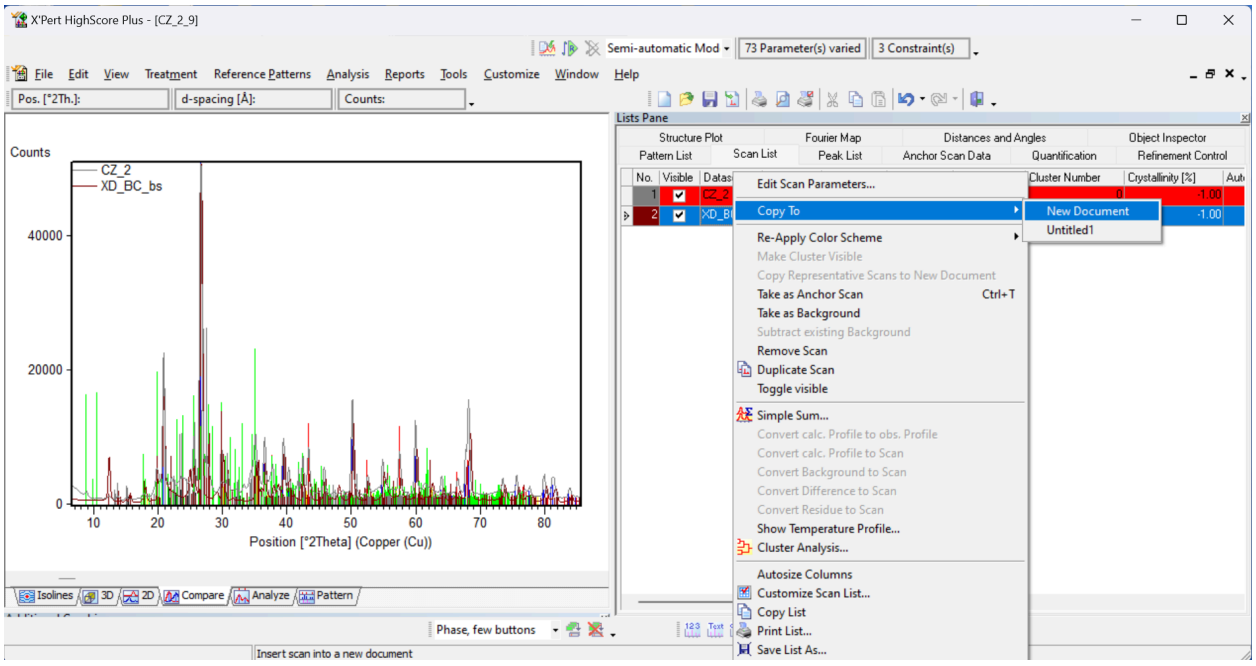
Next, we need to open a .HPF file with data that is already in Cu space. To do this, you'll want to use a file from Rob (or Alexander) that has already been converted. Open this file in X'Pert HighScore Plus.



Once the file that's in Cu space is open, go to file **insert** and insert the .RD file you just created.

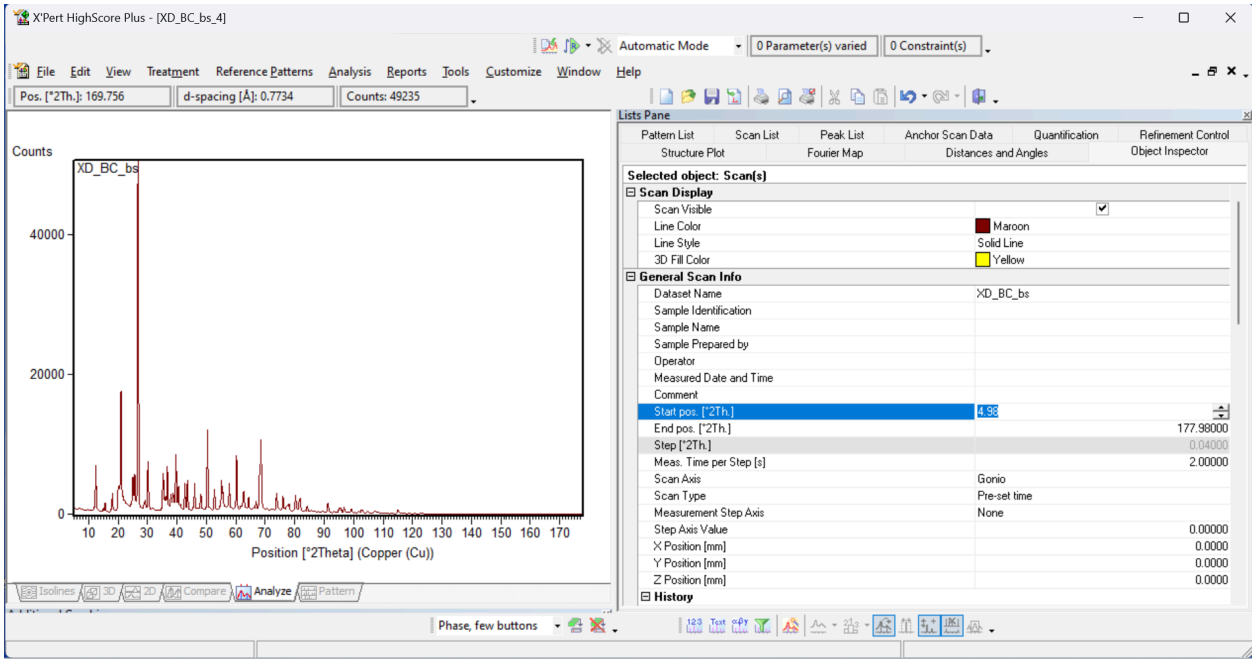


When you do this, the data from your .RD file will appear on top of the other data. Go to scan list – you will see that there are two different files; right click on the new one you just inserted and Copy To New Document.



This new .HPF file is your data in Cu space. This is the diffractogram you will use to search and match all your peaks.

Before we search and match peaks, lets trim the ends of your diffractogram. Go to the ‘Object Inspector’ tab and, under ‘General Scan Info’ look at the Start pos. and End pos. – adjust these so that the beginning and end of the diffractogram are free of any unnecessary noise.



It’s a good idea to do this here so that you can pick a starting and ending position and stick to it. From 5 to 90, for example. These values will depend on your specific material and dataset.

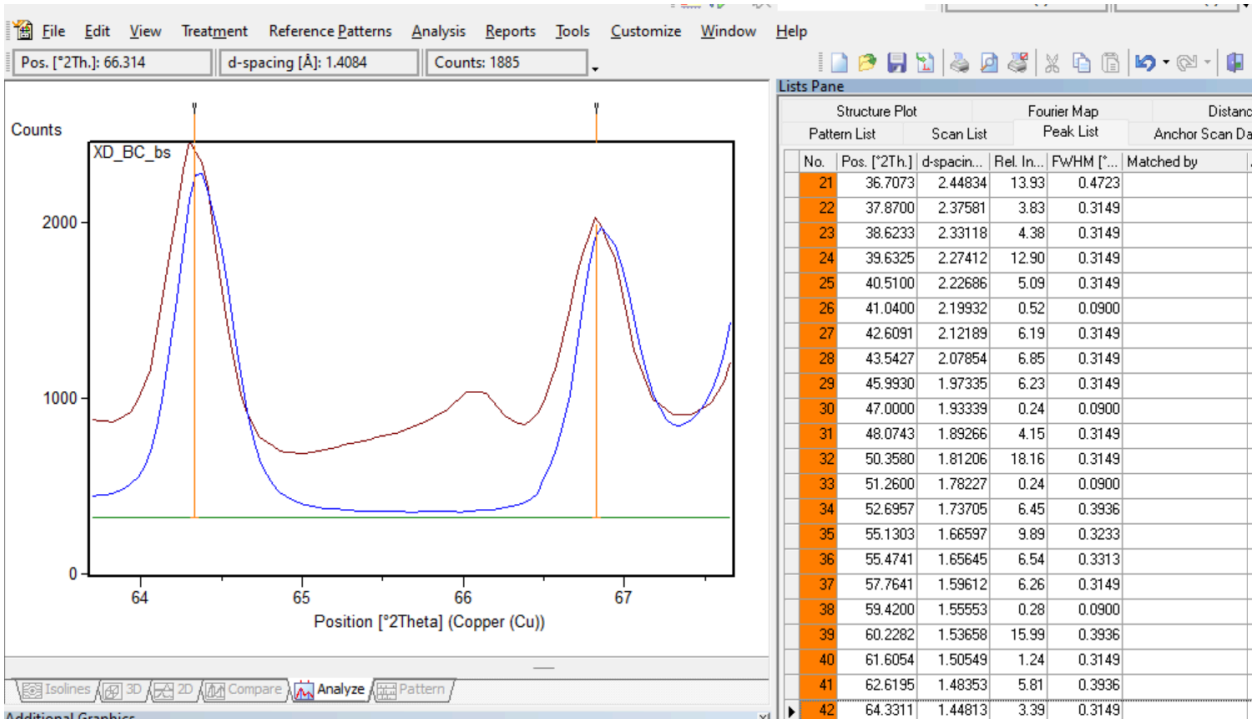
Performing Peak Searches

Once you have your properly formatted diffractogram, it’s time to search it for peaks. Go to the treatment tab → search peaks. Then, you’ll have several different ways to constrain your search: minimum significance, minimum tip width, maximum tip width, and peak base width (don’t change the method). Generally speaking, the defaults are a good place to start (0.1, 0.3, 1, and 2, respectively), and you can iterate an individual diffractogram until the program finds all of your peaks while fitting them correctly - click ‘Accept’.

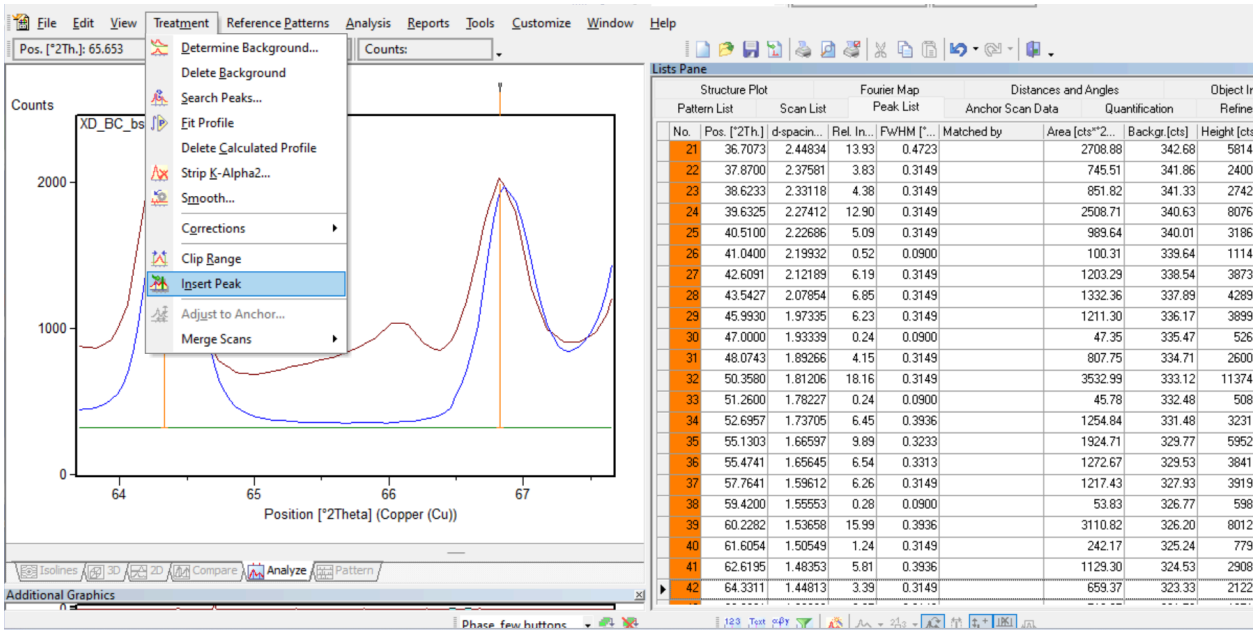
With the default parameters, there will inevitably be some peaks that the software misses or miss-identifies. For this reason, it is important to zoom in closely to your diffractogram and move from left and right to see how the software identifies the peaks. Each peak will have a small orange line with (and a black hat, if it hasn’t yet been accounted for by a candidate) at the top of the diffractogram - we need to see if any peaks are poorly fitted or not fitted at all.

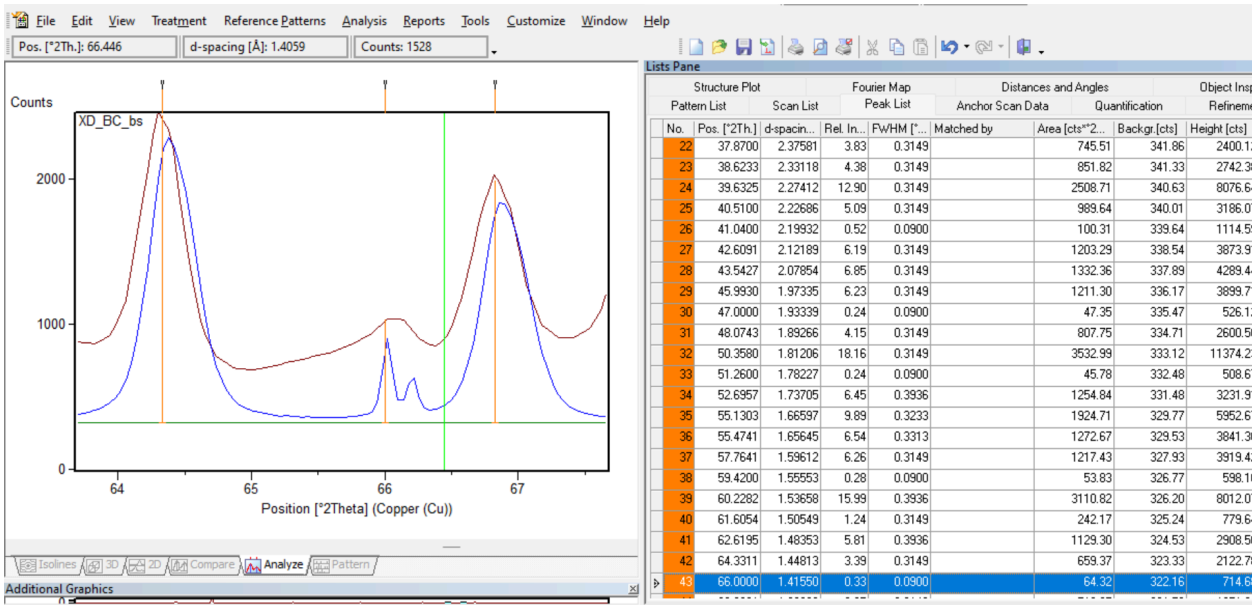
Evaluating peaks

As you move from right to left and look at all the peaks, you will find some small ones (that don't meet the minimum 'significance' indicated in the search parameters) and thus don't have an orange line indicating their identification by the software. In these cases, you'll want to add peaks manually – you can do this by going to the treatment tab insert peak and then dragging your cursor over the unidentified peak. Make sure to un-select the insert peak feature, otherwise it will continue to add peaks wherever you click. Here's an example of a peak that wasn't picked up by the software.



So, we'll need to add it manually.





Once the peak is added you'll see that when you hover your cursor over the peak, the same peak will be highlighted under the 'Peak List' tab - this is helpful for looking at FWHMs. This is also helpful if you need to delete a peak - you might notice some peaks whose orange line is off-center. In this case, by clicking on the same 'Peak List' tab on the right side of the screen and hovering over the peak on the diffractogram, you'll see that the peak you hover over is highlighted as a row off to the right – you can then hit the 'delete' button on your keyboard to remove it, and then you can manually add a better one as specified earlier. Once you've gone across your diffractogram a couple

times with this workflow and feel good about your peak identification, it's time to perform a search and match. This step is not for peak searching (we've already discussed that). This step is searching for candidates that can match the peak patterns in our diffractogram.

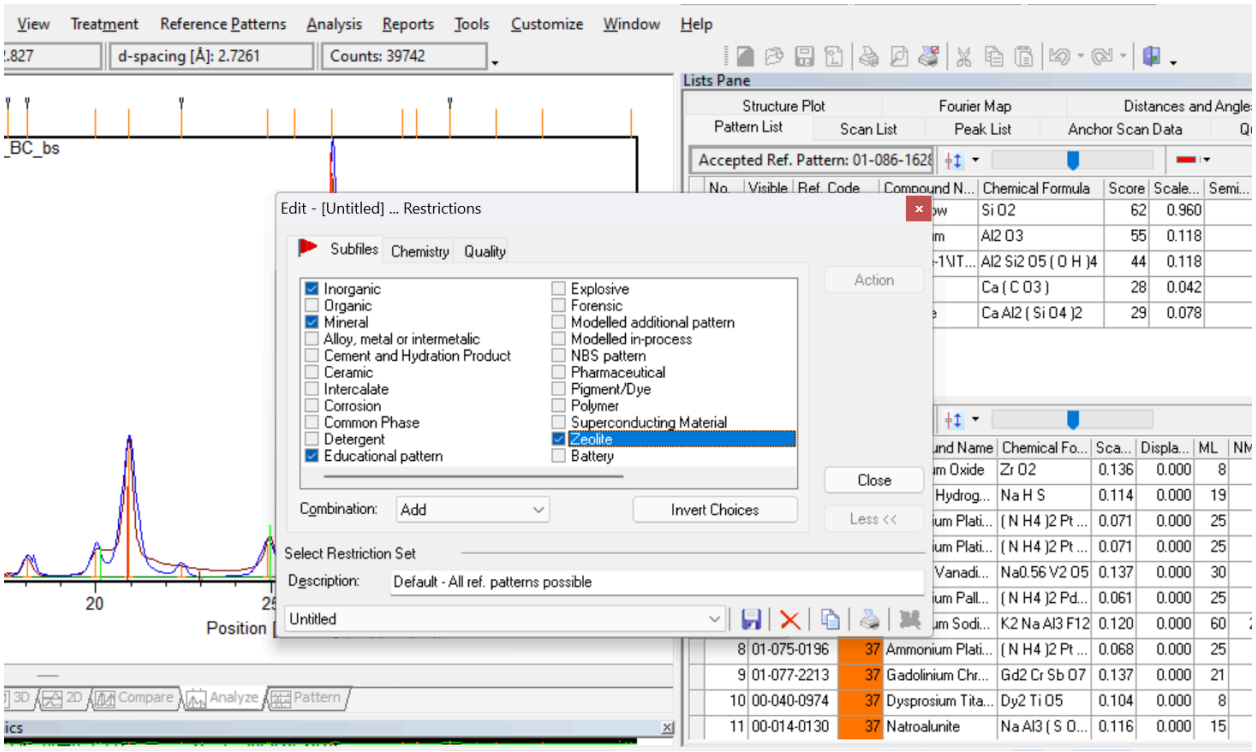
Performing search and match involves two main steps: 1) constraining your analysis and 2) evaluating candidates, both of which are best done after a rudimentary understanding of your sample is obtained. In other words, what kind of stoichiometry do you expect? What is the lithology of your sample, and what kinds of primary and secondary minerals might you expect from the parent material and weathering environment? Of course, this analysis is meant to answer these questions, but a refined hypothesis will aid in the proper selection of your mineral constituents.

A note on restrictions: for the first search and match of a sample, it's a good idea to run the search and match without any restrictions to see all of the different candidates recommended by the software. Most likely, the majority of candidates won't be realistic (you'll know this if you have a basic chemical understanding of your soil, for example, do you expect high levels of vanadium or cobalt? If not, then minerals with these elements are poor candidates); this preliminary step ensures that you're not discounting any important candidates by the restrictions you impose on the search in the following step

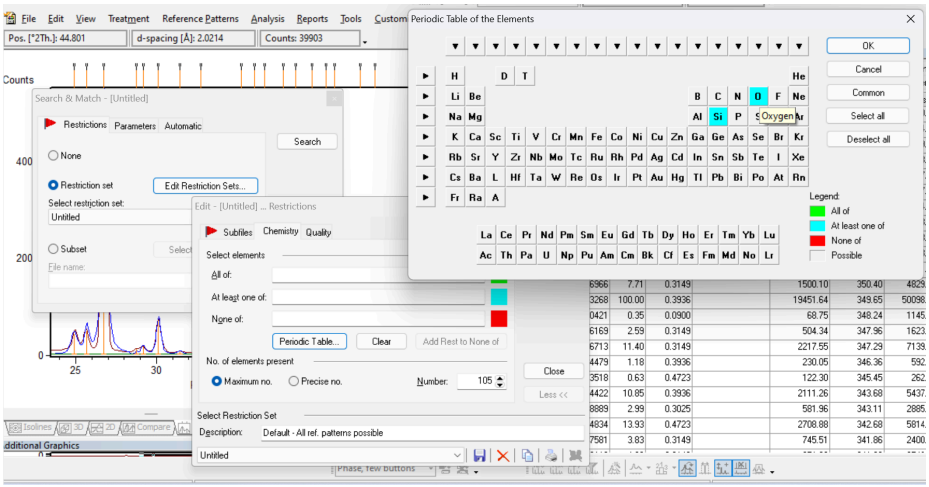
1) Constraining your analysis through restrictions

- Begin by going to the Analysis tab → Search and Match → Execute Search and Match, and this will bring up a pop-up window with a restrictions tab. Instead of checking 'none' check 'Restriction set,' and to the left of that filled circle there is a button that says 'Edit Restriction Sets…' click this - it presents another window with three additional tabs: restrictions, parameters, and automatic.

Subfiles: here you can check the categories of minerals that make the most sense for your samples, see example below



This way, the search and match function will only suggest candidates who fit within these categories. To further refine the candidate search, we can go to the ‘Chemistry’ tab. Here, you can use the ‘Periodic Table…’ button to select elements - the first click is green (all of), the second is blue (at least one of), and the third click is red (none of). This way you can customize your restrictions based on your chemical understanding of your study site. Here’s an example using these windows:

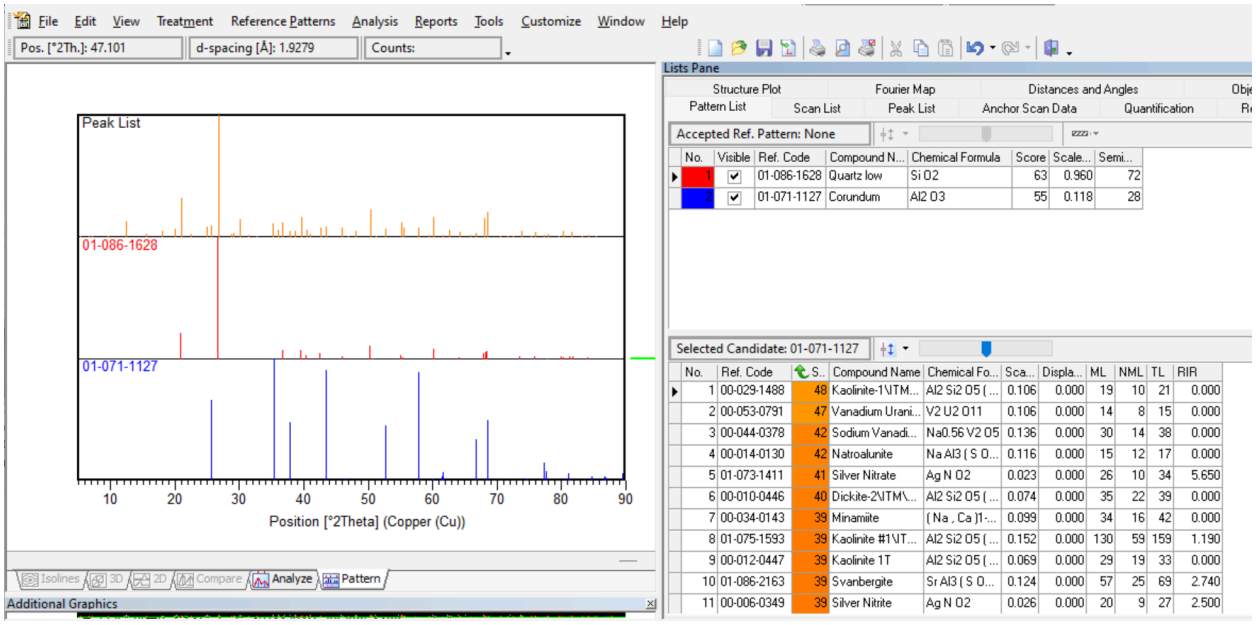


As you'll see with these candidate suggestions, there are several options for the same mineral, they just have different reference codes. After you execute the search and match, look under the 'Pattern List' tabs to see all the candidates. You can simply drag them from the lower half (candidates) to the upper half (accepted reference patterns), and you'll see the black hats disappear for the peaks which were satisfied by that candidate.

What I found helpful was to get to this point of data analysis for every sample in a profile (or site, depending on your sampling scheme) and figure out which candidates had the highest score across the entire profile (or site). For example, I took note (in an Excel spreadsheet) of the top 5 corundum candidates across all my samples, then did the same for quartz (assuming quartz is present), then I selected the reference code with the highest overall score. Next, I accepted these reference patterns to see which peaks they satisfied. This process takes some time but will ensure that you have consistency across your sample set.

You will likely find that for a given profile (or site), there are many of the same minerals, and its helpful if, once you find the minerals that are common across your profile or site that you save them as a group so that you can apply them (with exact reference codes) to a new sample without having to hunt around for them. You can do this by going File → Save As and then saving the list as a .CRY file. Then you can open this file for other samples and it makes things easier. Alternatively, you can search for a specific mineral by its reference code by going to the 'Reference Patterns' tab → Reference Code… and type in the reference code associated with the particular mineral you're looking for.

Note on choosing the right mineral: although the score (value from 0-100 that also has a color with it) is a helpful means of identifying suitable minerals, it is also important to see just how each candidate's peaks intersect with yours. The software is great at doing this, and the score reliably represents the extent of similarity between the peaks in your sample and those of the candidate, but it's helpful to see this visually and assess if there are any specific peaks that don't look right or if there's an especially high intensity peak in your sample that doesn't seem to align with the candidate, this may be reason to check out other reference codes, which may differ slightly but noticeably from each other. I found the pattern tab (beneath the diffractogram) to be helpful for this. Here's an example that has one of Xenia's diffractograms with a Quartz and Corundum candidate accepted.



Here, the top pattern shows all the peaks identified and their intensities, and beneath, there are two patterns: red and blue, which correspond to the accepted Quartz and corundum on the right, respectively. With this pattern viewing option, we can clearly see that the quartz is a great match for the corundum, although it has a decent score, does not share the most prominent peaks in the sample and has many high intensity peaks that we don't see in our diffractogram. In this case, we need to check and see if the other corundum options look any better.

This guess-and-check workflow can take a while to get used to, but once you have things streamlined you'll get closer to having that .CRY file I mentioned before, which contains the pattern list that you're confident in. Only then are you ready to begin the quantification through Reitveld refinement.

Quantification through Reitveld Refinement: I haven't written this section yet,

https://rruff.geo.arizona.edu/AMS/result.php is where you find CIF files - you need one CIF file for every accepted candidate - this will be uploaded into the Refinement Control Section and saved as a list