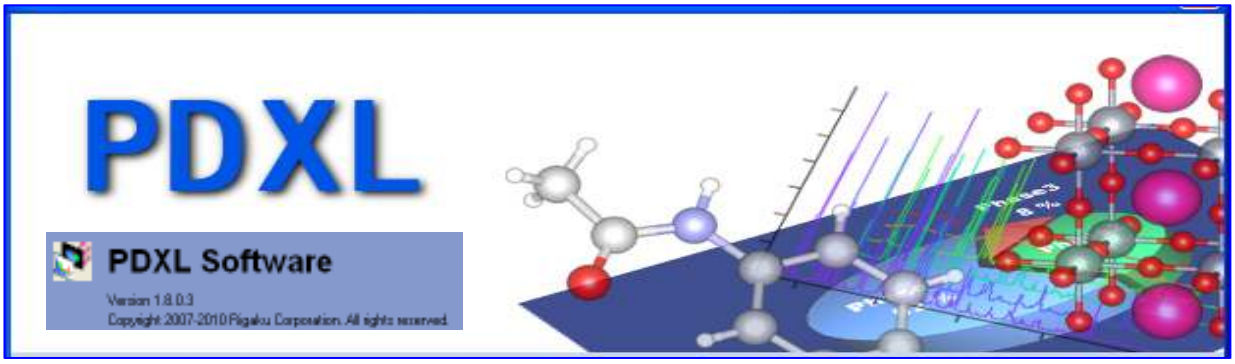
Rigaku

PDXL 1.8.0.3



Rigaku PDXL Software

Version 1.8.0.3

Rigaku 9009 New Trails Drive The Woodlands, TX 77381 281-362-230 modified 06/14/2012

PDXL 1.8.0.3

Table of Contents:

SPECIAL ANALYSIS MODULES:

PDXL 1.8.0.3

FREE PDXL UPGRADES ARE AVAILABLE ON-LINE FOR THOSE WITH RECENT VERSIONS. KEEP YOUR LICENSED COPIES UP TO DATE …

1. To download the newest version, go to your PDXL help menu, click on " Update PDXL". 2. If your computer is hooked to the internet, the following window will appear or type Rigaku PDXL download in any search engine such as Google.

Website: http://www.rigaku.com/downloads/software/pdxl.html

Input your name, email address, and PDXL catalog number and click "Next". The catalog number is the four numbers, one letter, three number code written after "Cat.

No." on the top page of the PDXL User's Manual. Also, a copy of the PDXL should be located on your computer desktop and a third source of the catalogue number can be found on your PDXL CD Label.

I. Opening the Program

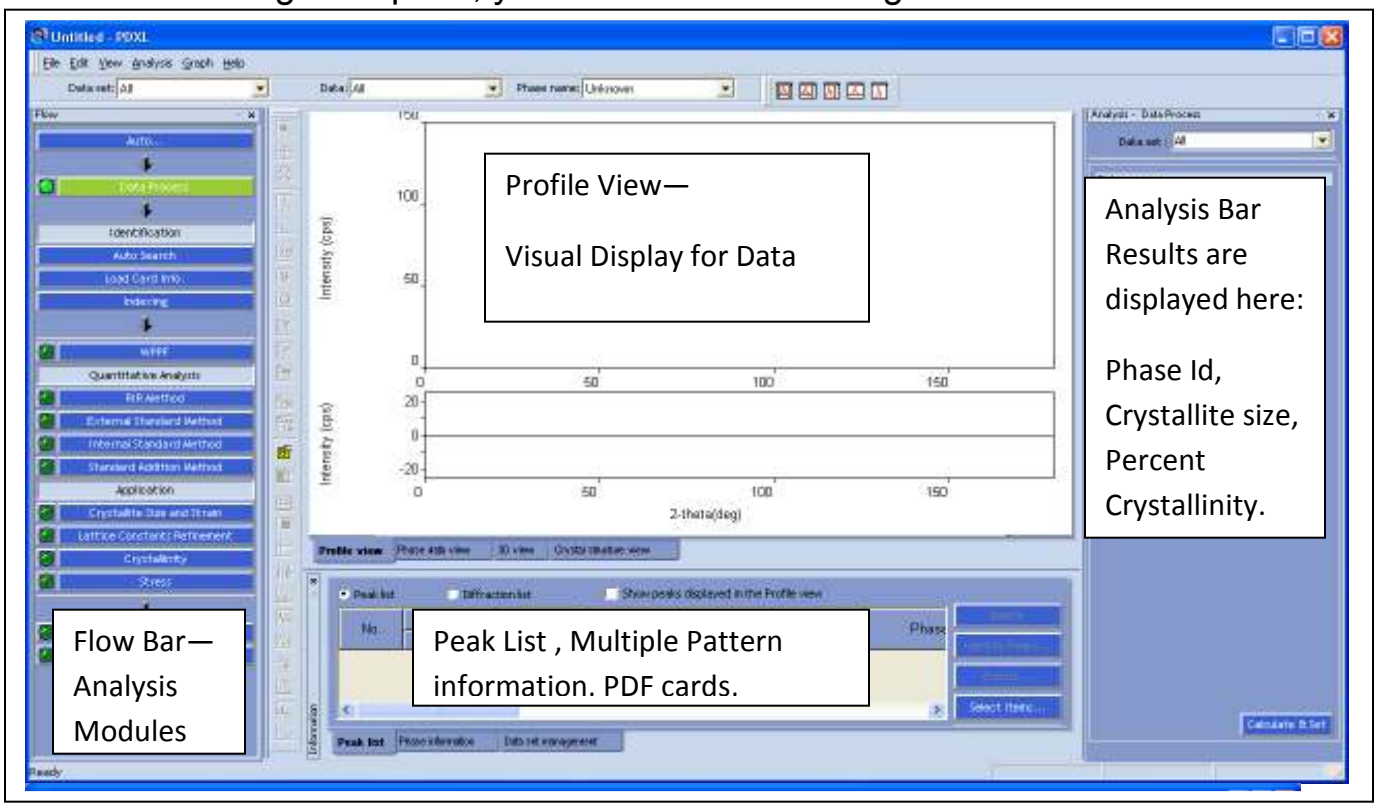
To open PDXL:

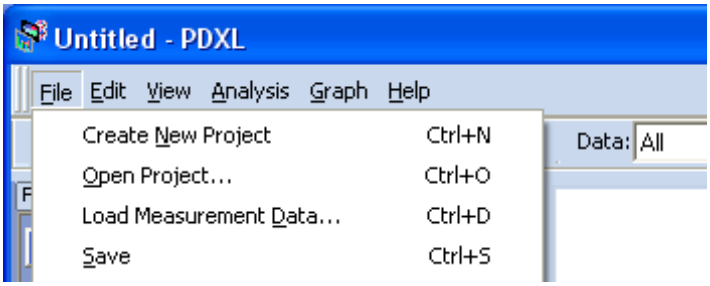
Use "Administrator as the User login:

No Password is needed; just press the "OK" button:

II. Window Layout:

1. When the Program opens, you will see the following series of windows:





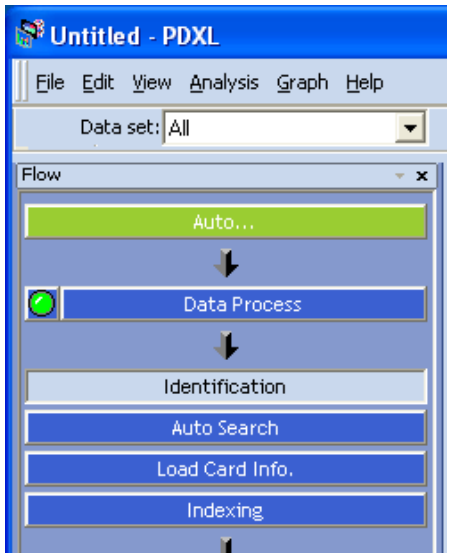
III Creating a New Project/Opening Data:

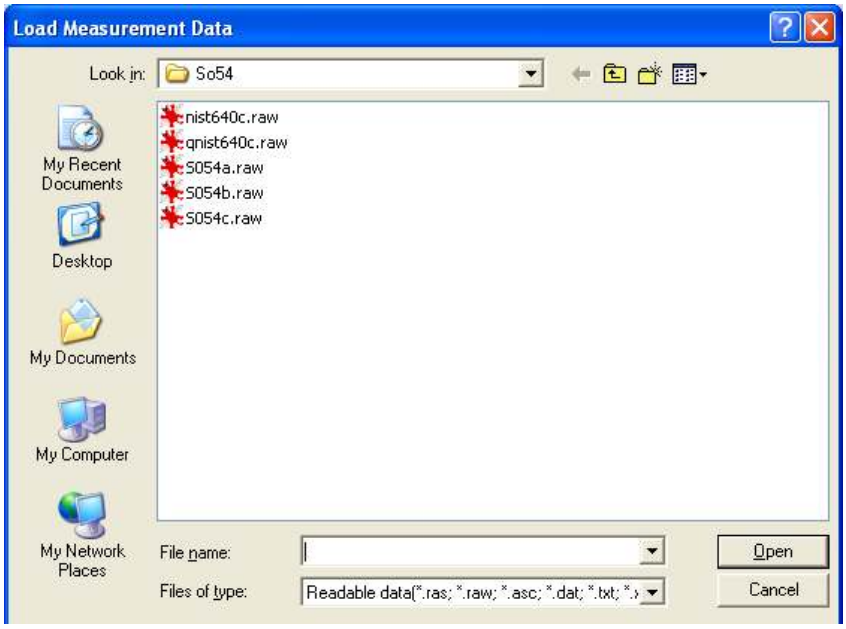
1. First Time Analysis, Click on “File”, then “Create New Project”.

   This is will create a new set of data folders to store all your data and results in one place. After reopening a Project, all associated files and results will be included in the project. Individual data plots can be selected and viewed or a comparison view is also possible.

2. If individual patterns are desired (only single data files-no grouping), proceed to next step.

3. Click on the “Auto” button on the Flow Bar and find the data of interest.



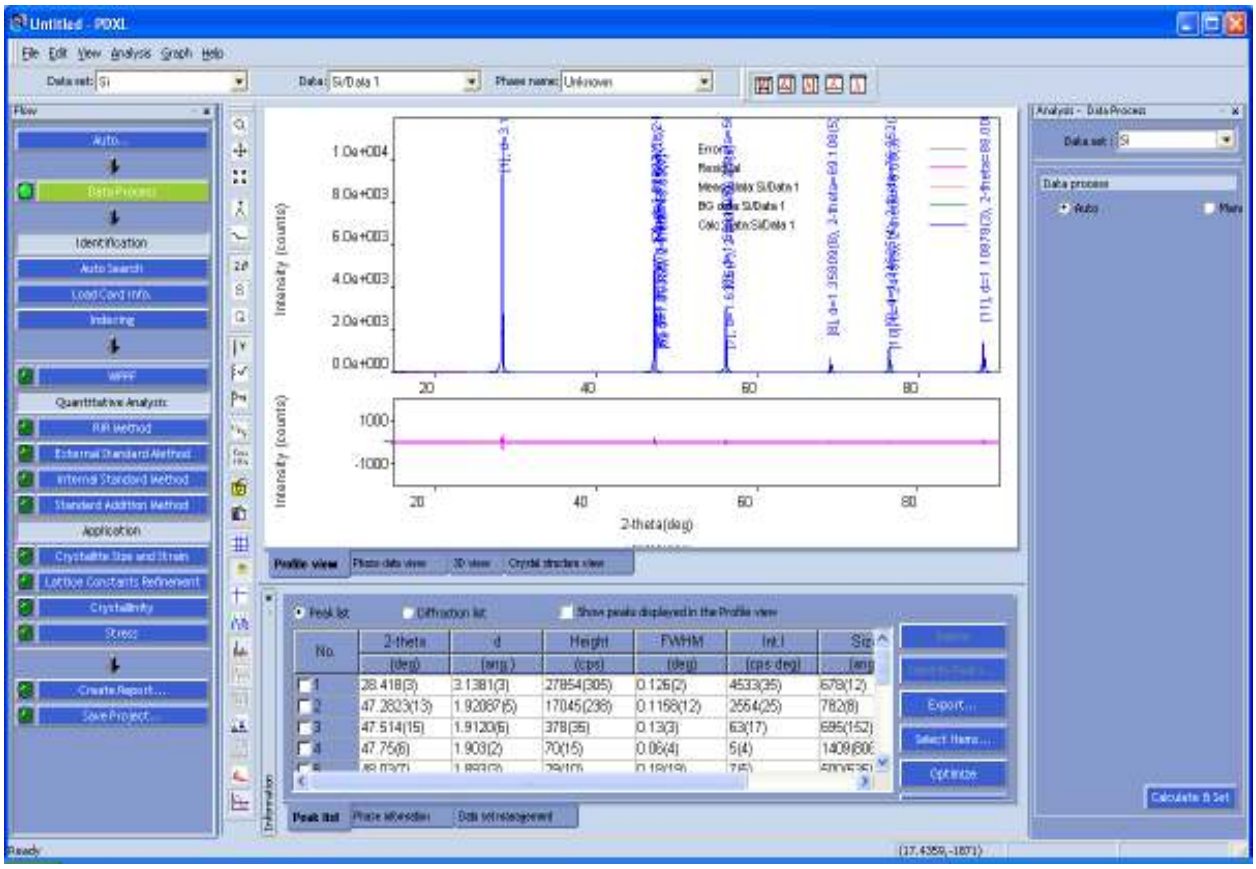


4 .Highlight the desired file, then press the “Open” button.

The raw data will appear in the Profile Area.

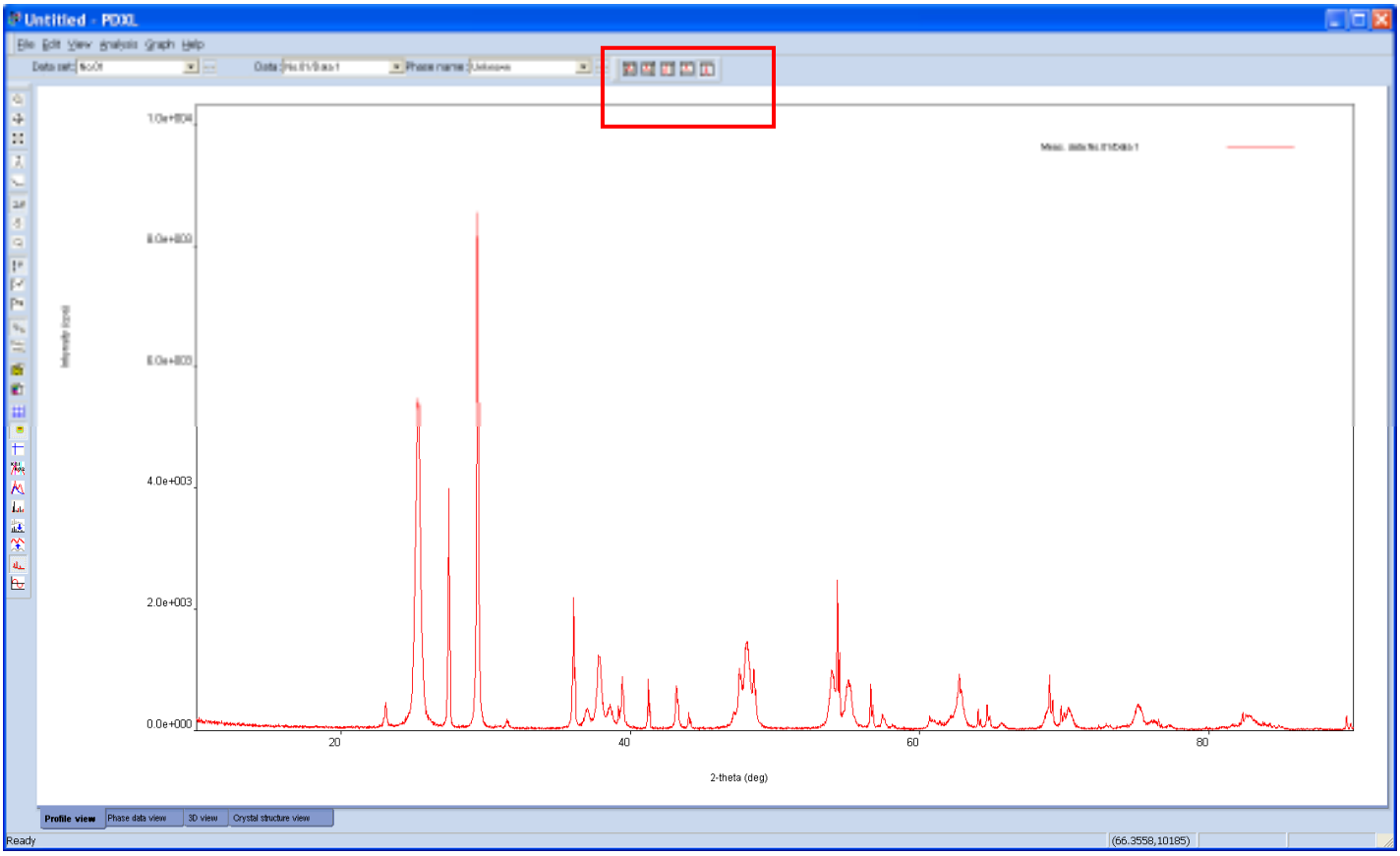
5. Several key calculations have already taken place. To view the automatic calculations, make sure you are on the peak list tab (located at the bottom of the Profile View area).

--Peak positions, intensity and FWHM are already determined from automatic integration procedures.

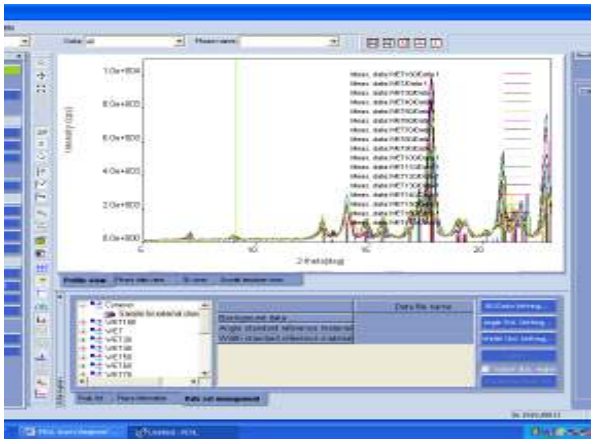


This information is displayed in the center low region on the Peak List tab. Visual confirmation can be seen in the Profile View region on the Profile View tab. All information and pattern processing can be edited at this time: Backgrounds can be made visible for editing (adjusting), additional peaks may be added, existing peaks may be modified or deleted, and intensities may also be adjusted (see page 10 for editing instructions).

6. Using the five tabs on top of the diffraction pattern (see red tab below) the view can be changed at any point to get better visual of the pattern/analysis bar/flow bar/peak list etc (see below).

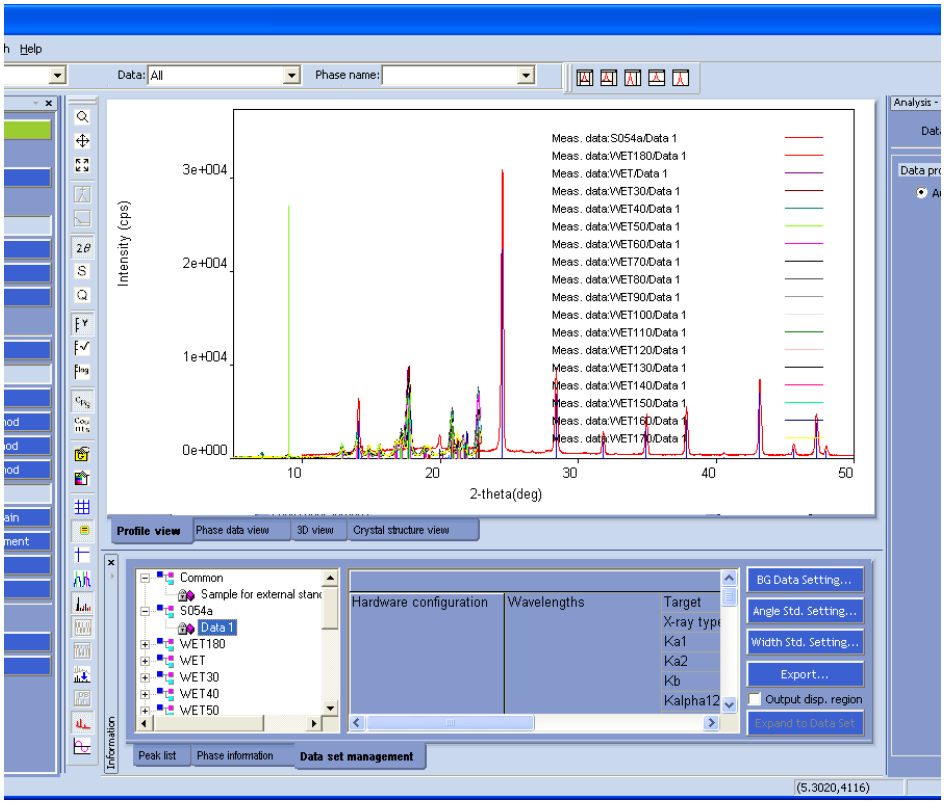


7(a). To open multiple files, click on the Auto button on the Flow Tool bar and highlight several files at the same time. Press the Open button.

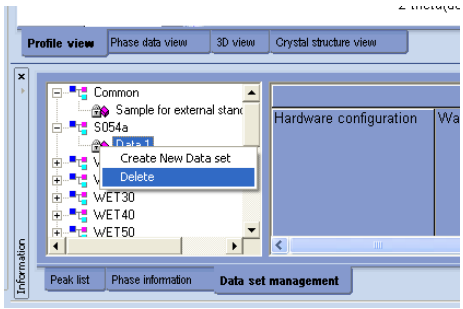


7(b). Also, consider displaying the data in 3D for better comparisons of more than 2 patterns. Go to the 3D view tab in the middle section. Rotate patterns to display the data in the best format.

8. To delete an overlaid raw data pattern, go to the “Information area” and click on the “data set management tab”:



8. Expand the file to see the data file that needs to be deleted. Right mouse click on the data and click on delete. Press the Yes button when asked to delete.



9. Right mouse click on the data and click delete.

10. Click on the “yes” option when asked to confirm.

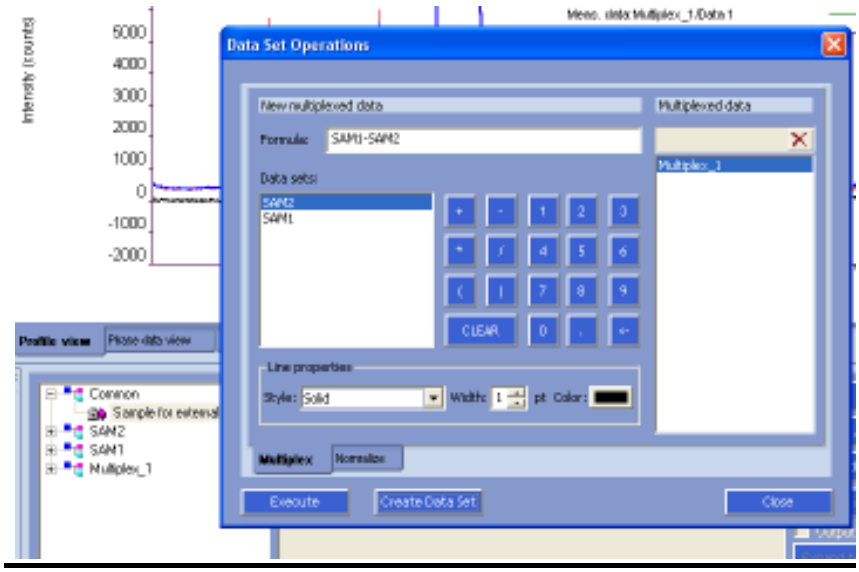
IV Add and Subtract Overlays (or any other mathematical function, i.e. averaging):

Open multiple files as illustrated above.

Go to the Data Set Management tab. Click on the plus sign to expand the information until the Data 1 file is observed. Click once to highlight one of the raw data files.

On the right side a button, a new button will appear, “Data set operations”.

Click on Data set operations”. Double click on each raw data file to be calculated, add a mathematical function between the file names. For subtraction see example:

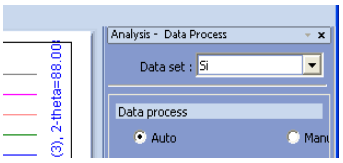


Press the execute button when formula is complete. To save the newly created file, click on the file name in the multiplexed data column, and then click on the Create Data Set button on lower left.

Notice the new file that will appear under the Data Set Management tab.

Please also be aware of the “Normalize” tab located behind the “Multiplex” tab. Data files can be normalized if desired.

V . Automatic Pattern Processing:



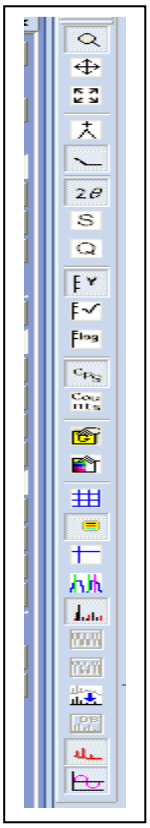
1. When a raw data pattern is opened in PDXL, it is automatically processed (background added, peak search and profile integration are automatically performed. This is a VERY convenient function and performs majority of all functions needed for further analyses.

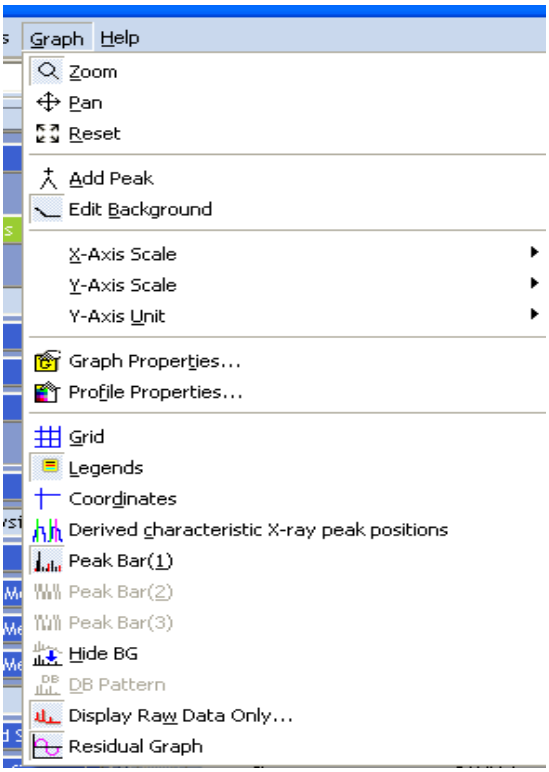
2. To change this setting and perform all the functions manually, click on the manual button on the right side of the analysis window as shown below.

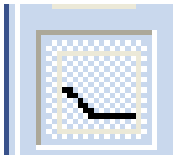
V. Editing the Profile information:

1. Find the PDXL tool bar (located vertically between the Flow Bar and the Profile View.

PDXL Tool bar-graph buttons:



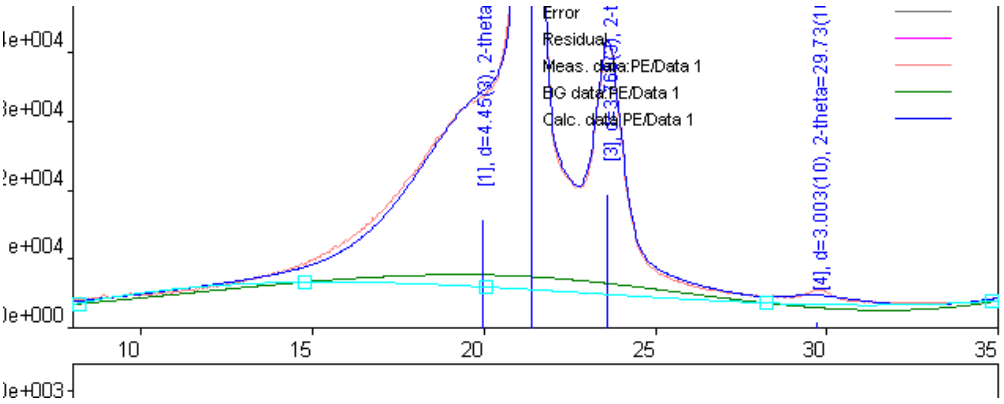


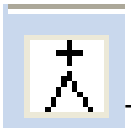
2.  To view and modify the background,click on the Edit Background Button

The background will appear as a light blue line with squares to designate the exact points of the background. To the edit the background either click and drag the squares to a new location.

To add a new data point, move the mouse cursor to the desired location and right mouse click. This will add a new point and modify the fit of the current background.

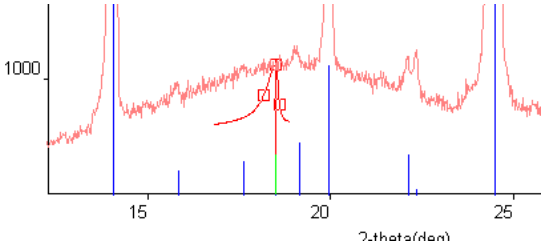
To remove a data point, right mouse click on a blue square and the point will be deleted..

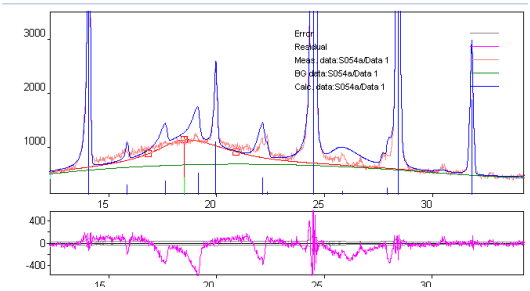




3. To add peaks click on the Add Peak Button, move the cursor to the desired location and right mouse click to add peaks (makesure your Y scale is in cps mode).

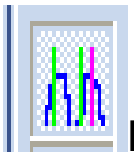
To edit peak position, shape and intensities (if needed), UNCHECK the Add Peak button, and click on the peak position in peak list. Then, 3 red squares will appear on the peak (in Profile view). These squares will edit the shape and intensity of the peak. To edit the position, click and hold the peak bar and drag to new location.



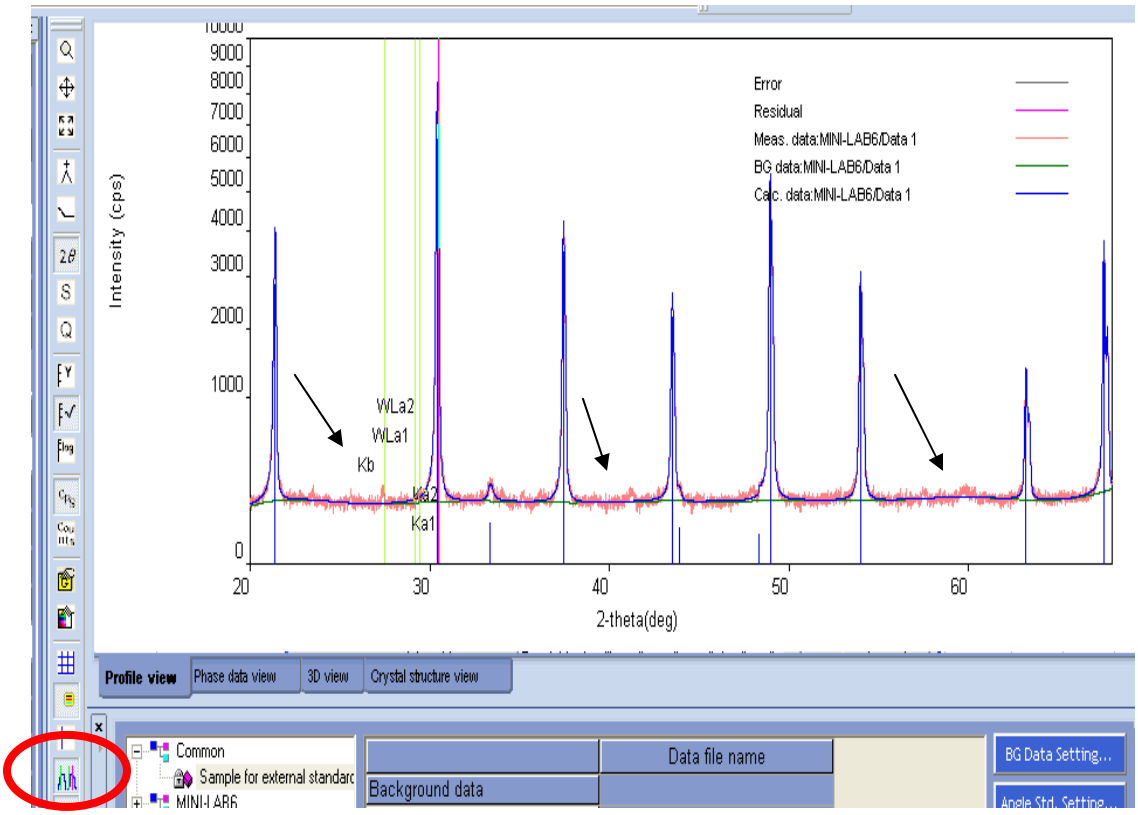


Edit the peaks until the blue difference line matches the red raw data pattern.

VI. Editing or Identifying Kbeta peaks:



PDXL Cu Kbeta Peak Template cursor



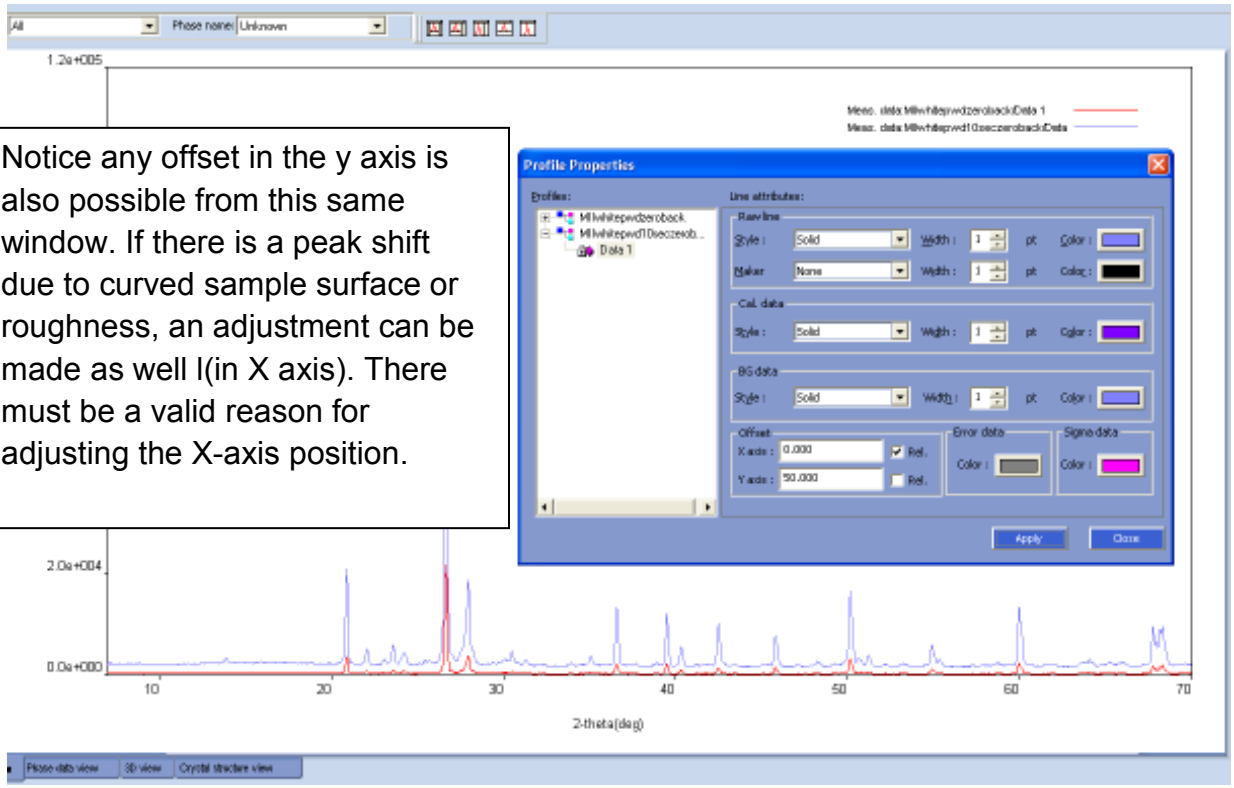
The small peaks immediately preceding each major LaB_6 peak are Cu Kβ peaks. Remember your Kβ filter only removes 99% of all the Kβ radiation wavelengths. In most samples with average counts Kβ peaks are not usually visible above the background counts. However, with a highly crystalline material, such as LaB_6, the counts will be much greater and the Kβ peaks will be observed above the normal background fluctations. Note the Kβ cursor position left (lime green marker).

If your system has a monochromator, no k beta peaks will be observed

VII. Changing Graph Properties:

1. To change colors of overlays or to offset patterns for better visibility, click on the “Profile Properties” button on the vertical tool bar.

Select the pattern that you would like to change in the profile window. Select the line color, then press “apply” button.



Notice any offset in the y axis is also possible from this same window. If there is a peak shift due to curved sample surface or roughness, an adjustment can be made as well l(in X axis). There must be a valid reason for adjusting the X-axis position.

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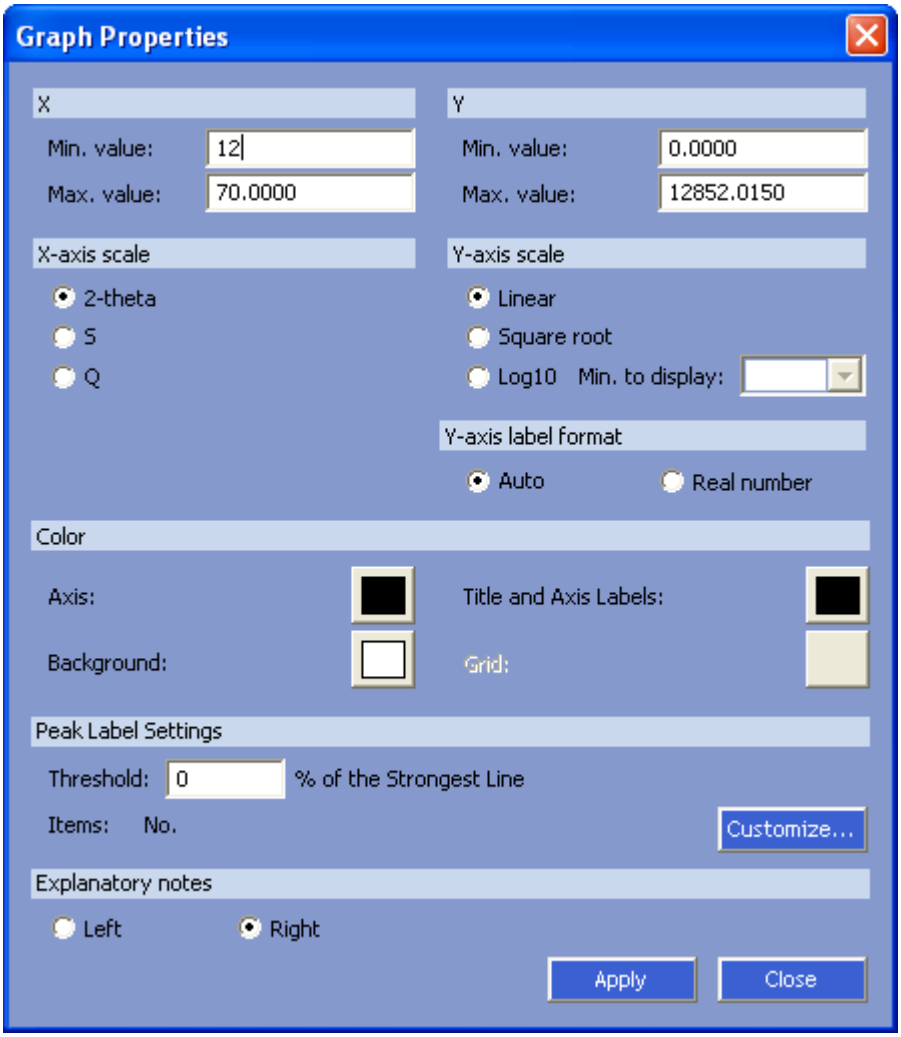
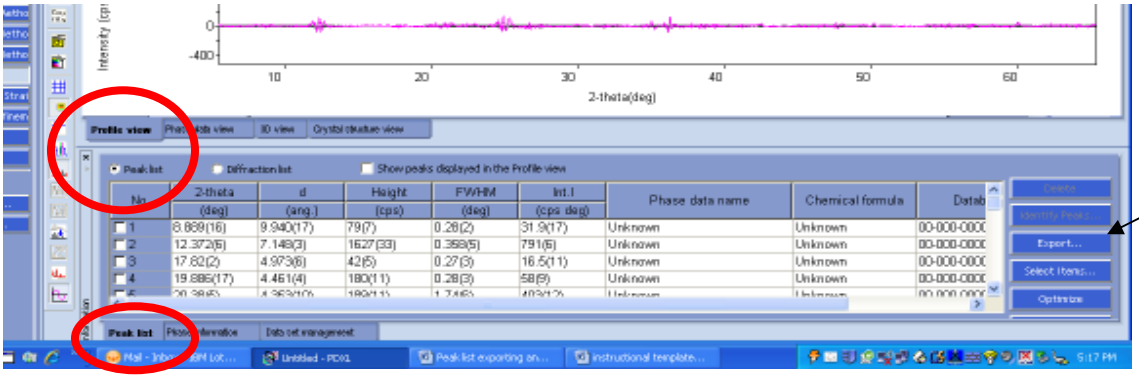
2. Changing the graph properties for printing or visual presentations



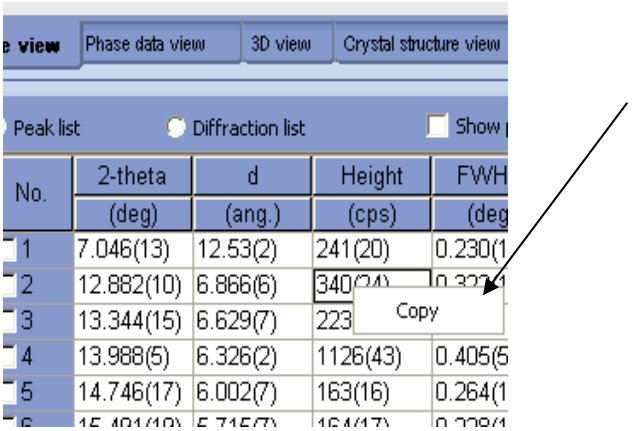
VIII. Peak List exporting and Raw Data exporting:



1. PDXL will automatically perform the area integration or profile fitting as soon as the raw data is open. The results of the profile fitting will appear in a list at the bottom center window on a tab labeled “Peak list”.

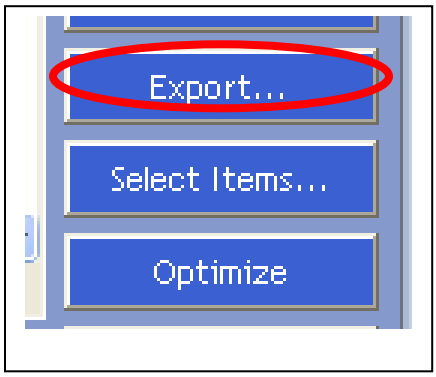
To export the peak list, 2 methods are available. To use either method, please make sure that you can are on the “**Profile view** “ tab at the top center window and also on the **“Peak list” tab** at the bottom center window, and that the **circle in front of “Peak List” is** also selected.

2. The first method is the fastest and easiest method. However, it will export the data in the exact same format as it is visually displayed. To use this method, click anywhere within the peak list on any cell, then click the right mouse button and click on the **copy** option in the menu. Open the program of choice and paste into that program. Excel is the recommended program.

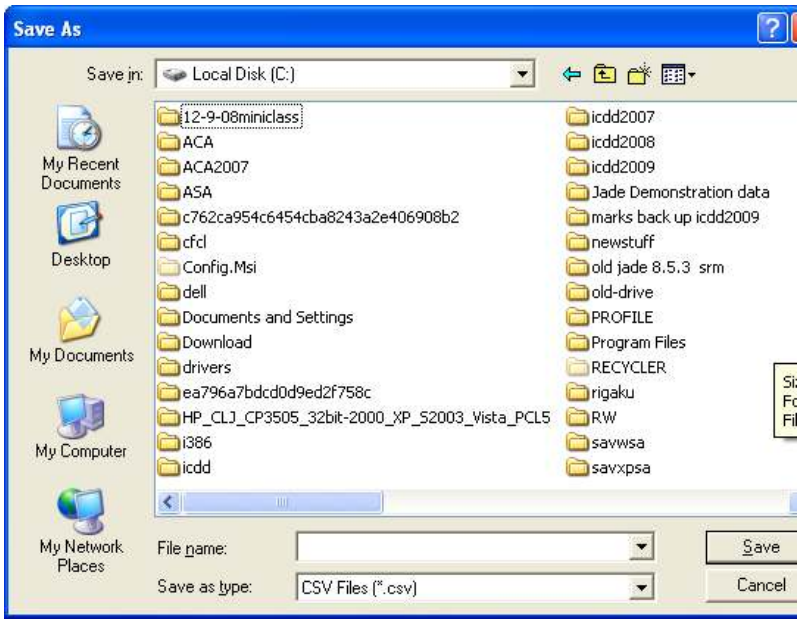


2theta	d-spacing A	height (CPS)	FWHM (deg)
7.046(13)	12.53(2)	counts	0.230(18)
12.882(10)	6.866(6)	340(24)	0.322(11)
13.344(15)	6.629(7)	223(19)	0.269(17)
13.988(5)	6.326(2)	1126(43)	0.405(5)
14.746(17)	6.002(7)	163(16)	0.264(16)
15.491(19)	5.715(7)	164(17)	0.228(18)
15.97(3)	5.546(9)	99(13)	0.19(3)
17.162(12)	5.162(4)	1381(48)	0.406(17)
17.727(4)	4.9991(12)	6326(103)	0.316(6)
19.174(9)	4.625(2)	508(29)	0.296(14)
20.869(4)	4.2529(8)	3375(75)	0.278(3)
21.700(5)	4.0918(10)	1190(45)	0.307(6)
22.763(5)	3.9032(8)	4140(83)	0.334(7)

3. The second method to export peaks will export all of the peak information but with everything separated into a list format (profile fitting or area integration information from PDXL). To export this data, click on the “Export” button to the right of the peak list…

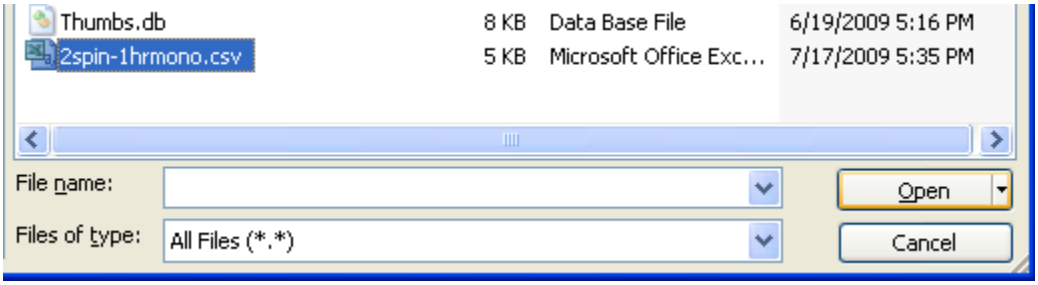


3b. A window will appear asking for a file name to export and save the data as a *.csv file (column separated view)



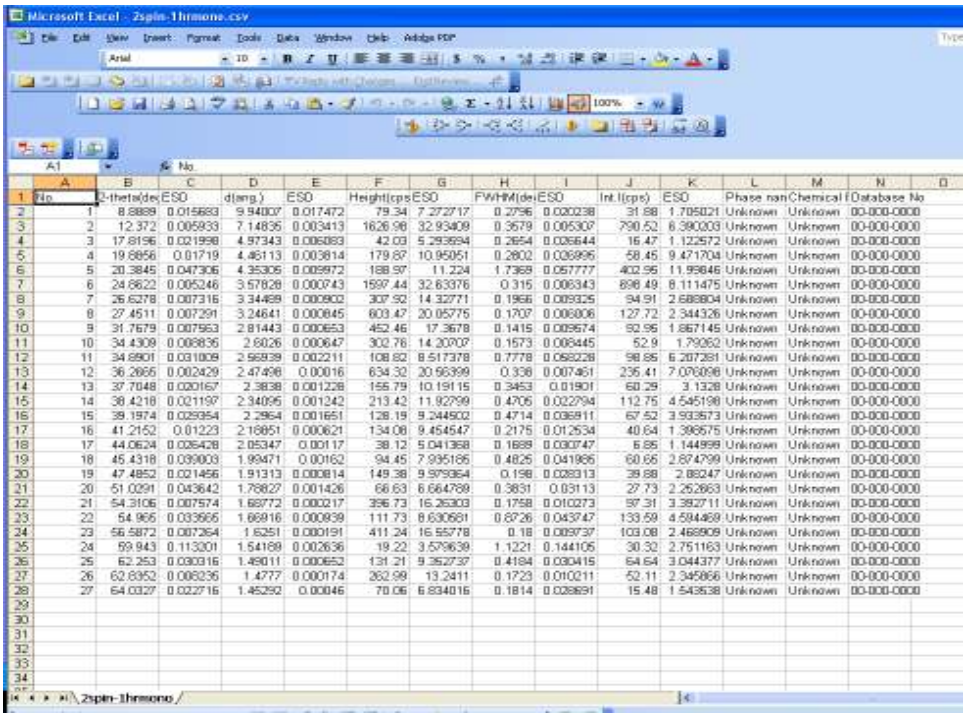
4. When saving the peak list, type in the name of the file. This is the name of the file to be retrieved in excel. It is a good idea to use the same raw data name but just change the file extension (i.e. 2spin-1hrmono. Raw should be saved as 2spin-1hrmono. Csv).

5. Open MS Excel, use the “all files” view and look for the csv file you just created:



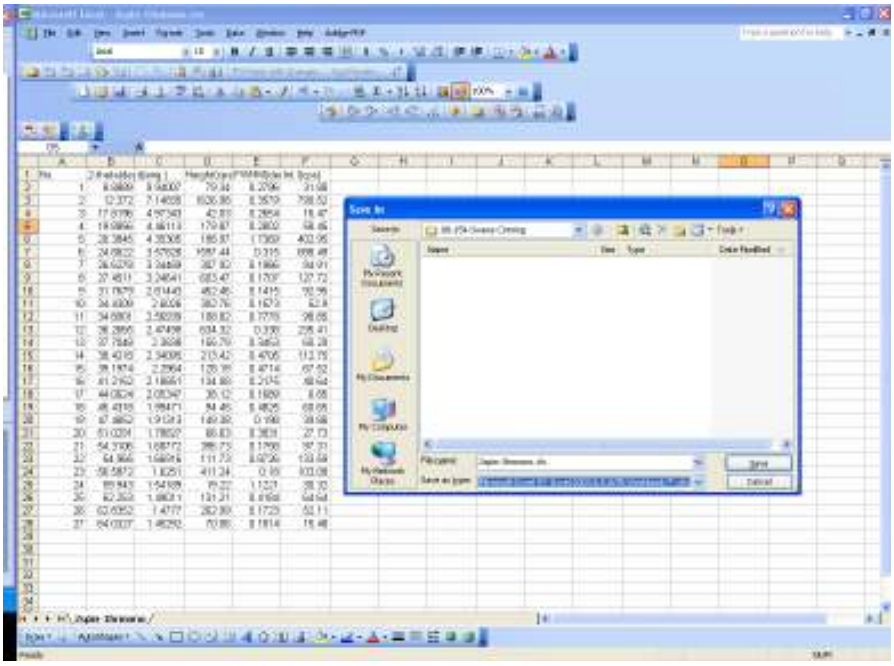
6. Click or highlight the file labeled *CSV and press the open button.

7. The data will appear in excel on a very limited format (csv not a true excel file).

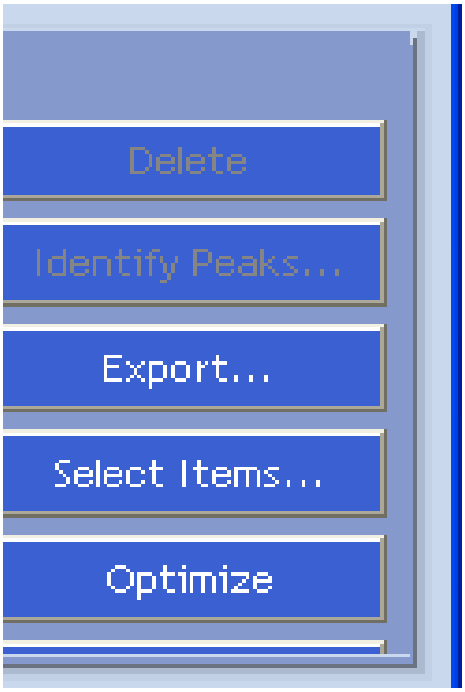


Please note that each characteristic of the peak list is separated, including the estimated standard deviations (ESD).

8. Edit the data to keep what you would like to save (hint, most people remove the esd columns) and then save the data as an excel file.

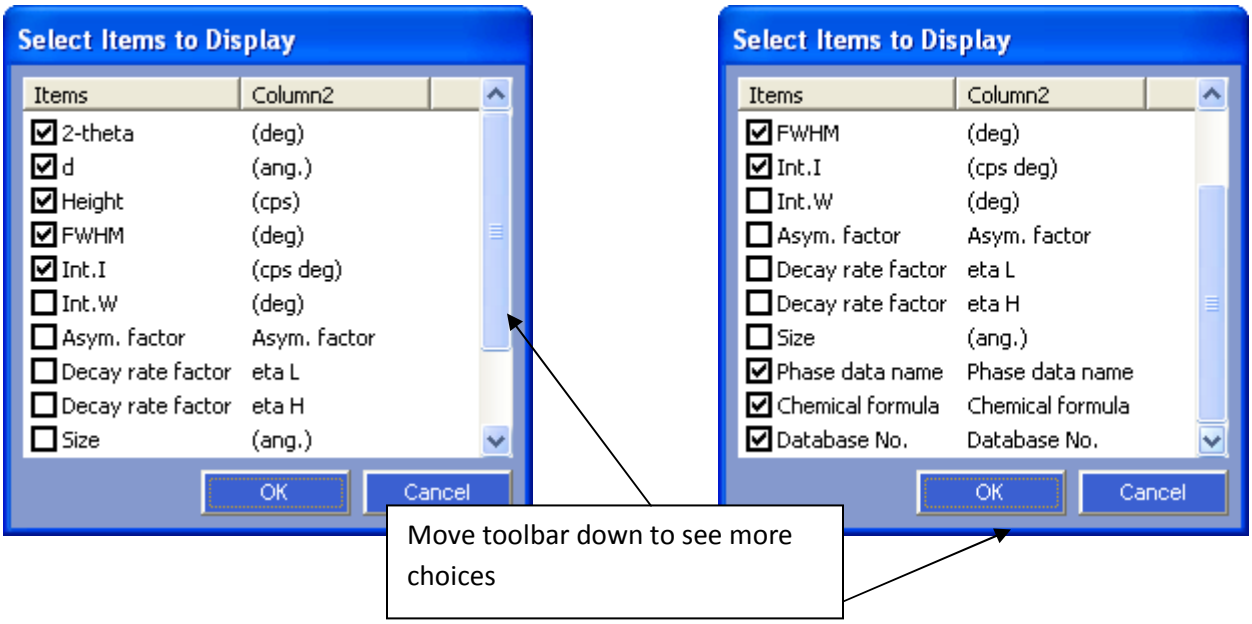


9. The information exported from PDXL into other programs can be customized .by clicking on the “Select Items…” button Inside PDXL just below the “Export “button.



9b.

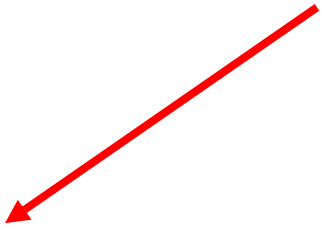
The following window will reveal all the choices of exported information:

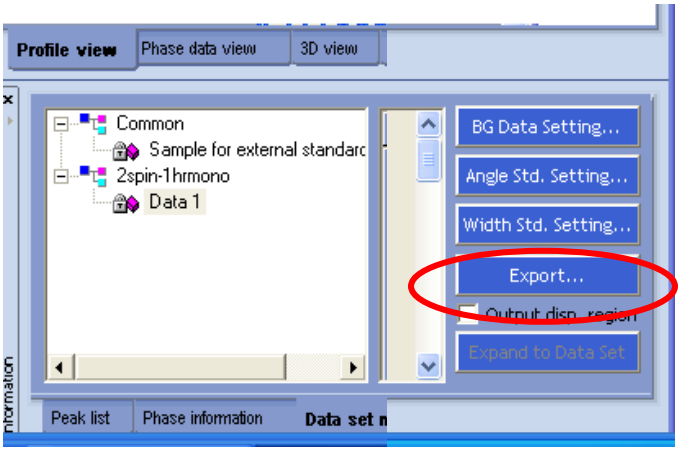


10 To export the **entire raw data** to plot or view in another program as either a csv or an ascii file:

11. Click on the “Data Set Management” tab on the bottom center window.

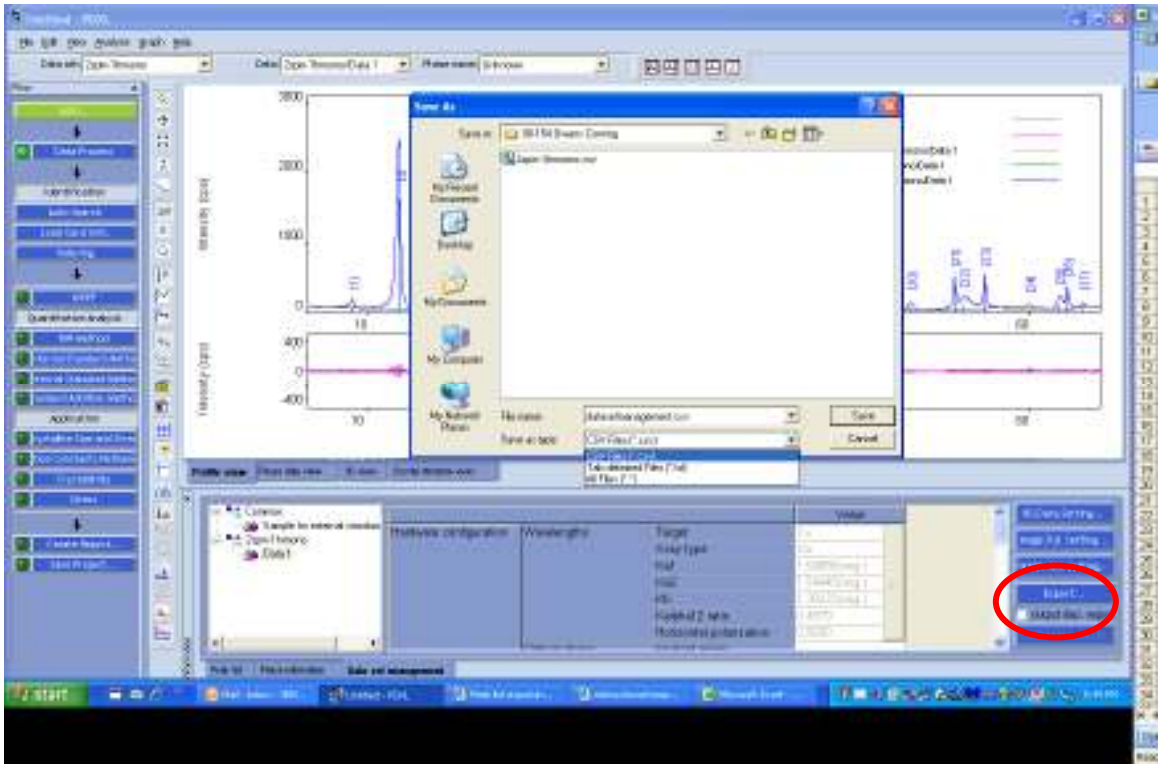
12. Find the data file that you would like to export and expand the file path by clicking on the plus sign (will turn to a negative sign) until you see the data with a purple square beside the name:





13. Click on the file name Data 1

14. To the bottom center right of the Data 1 area, find the button that is labeled “Export” and click.

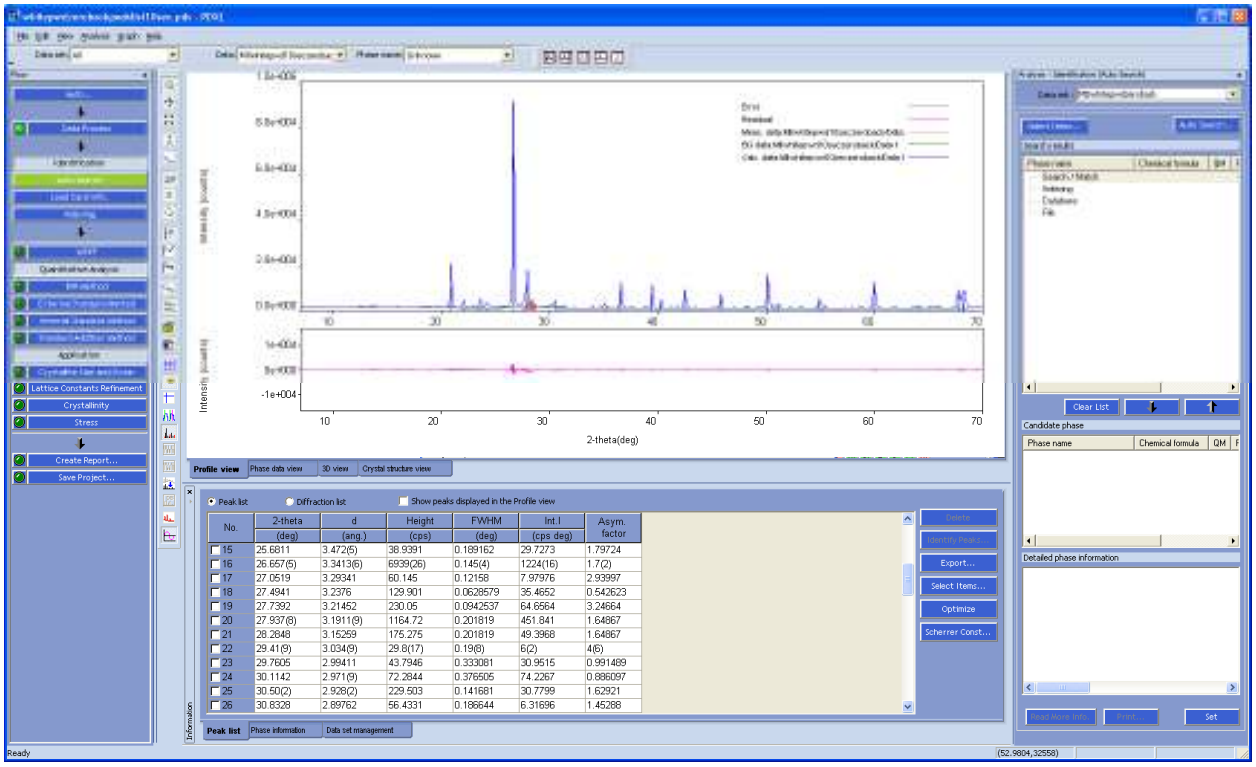


15. A window will appear asking for a file name to save the data. The standard default is csv, but the ascii or text format can also be selected. Save the data.

16. Open it in excel using the format above..

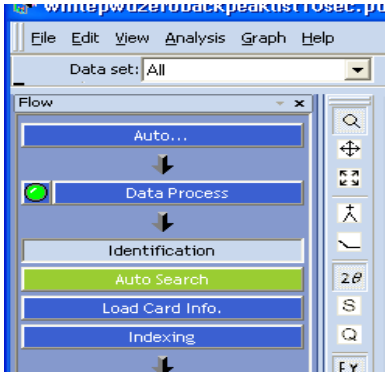
The entire raw data will be available in MS Excel as either a csv or ascii format:

IX. Phase identification:

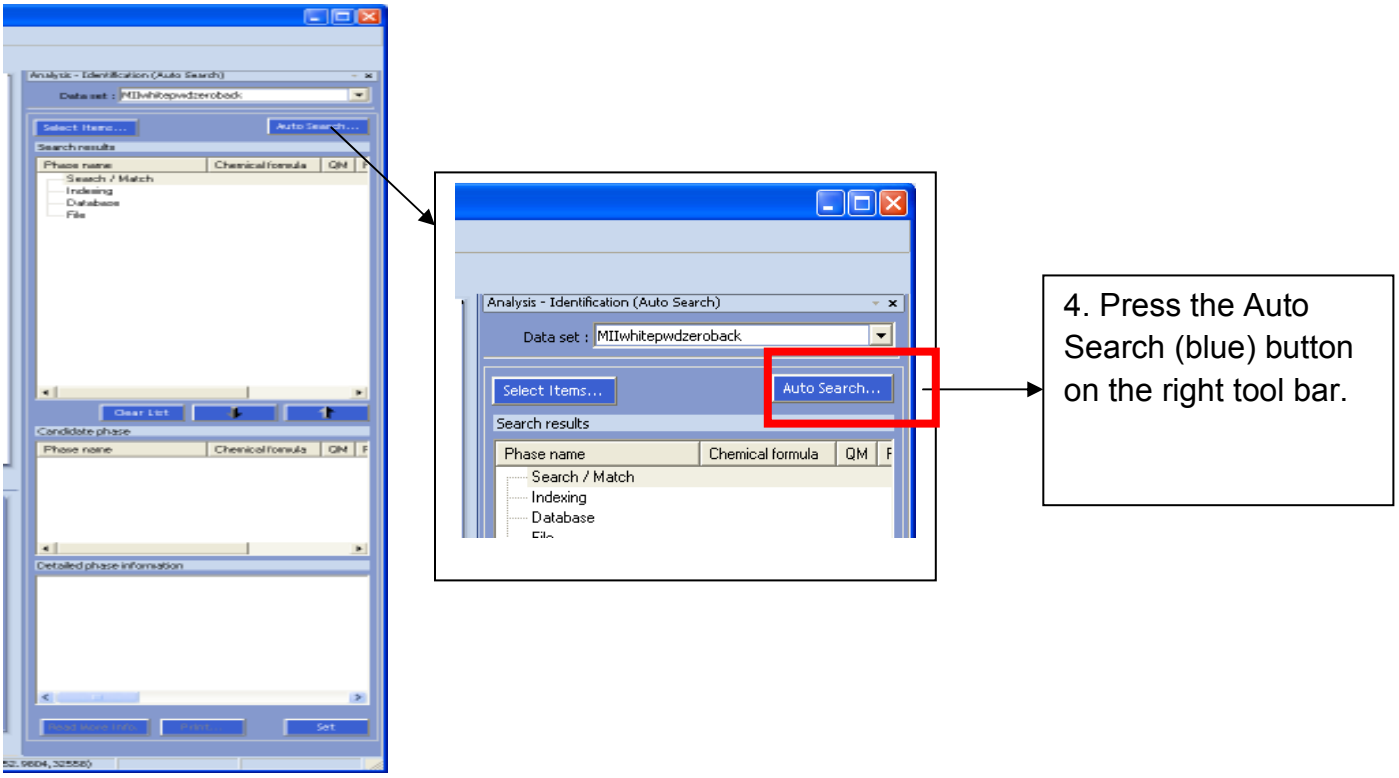


1. Check automatic peak list for thoroughness or completeness. If a peak of interest has not been automatically picked in the deafly mode use add button to add that peak in your list. Hit optimize button (in the middle section, bottom) and hit execute to optimize the peak list. When peak list is complete go to step 2.

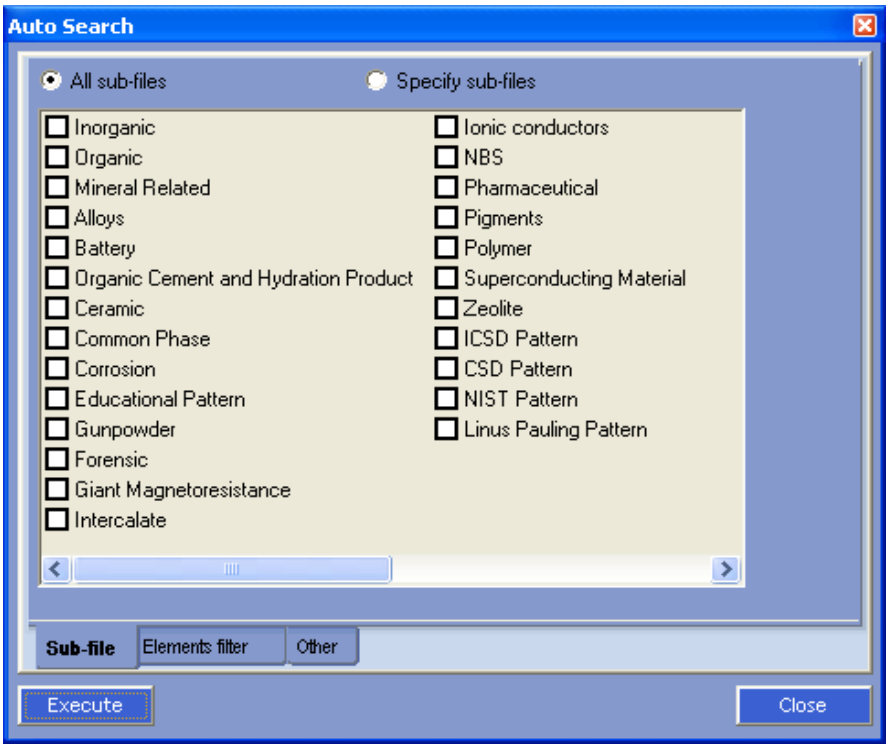
2. Press the next button on the flow chart (on left toolbar) called Auto Search.



3. Window on the far right changes to the search identification mode (Analysis- Identification, Auto Search).



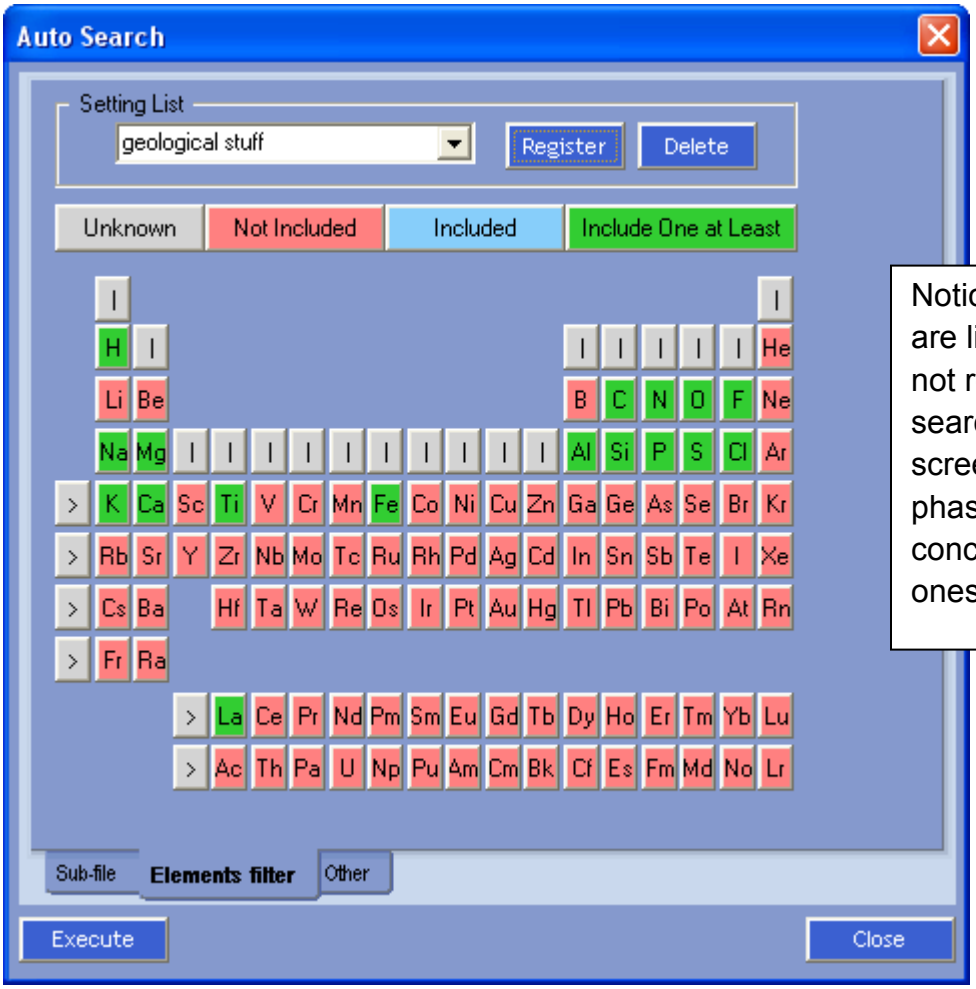
5. Select a subfile or series of subfiles to search. If you do not know which files to search, start with a wide approach and check the “All sub-files” option.



6. Press the “Elements filter “tab at the bottom of this menu. Set your chemistry filter to narrow down the choices.

7. Grey is no filter, red is excluded or NOT in Boolean logic, light blue is REQUIRED or must be present) AND in boolean logic, green is possible (OR in Boolean logic)

8. Chemistry filters for “common” geological material. Set the filter for a fairly wide search…

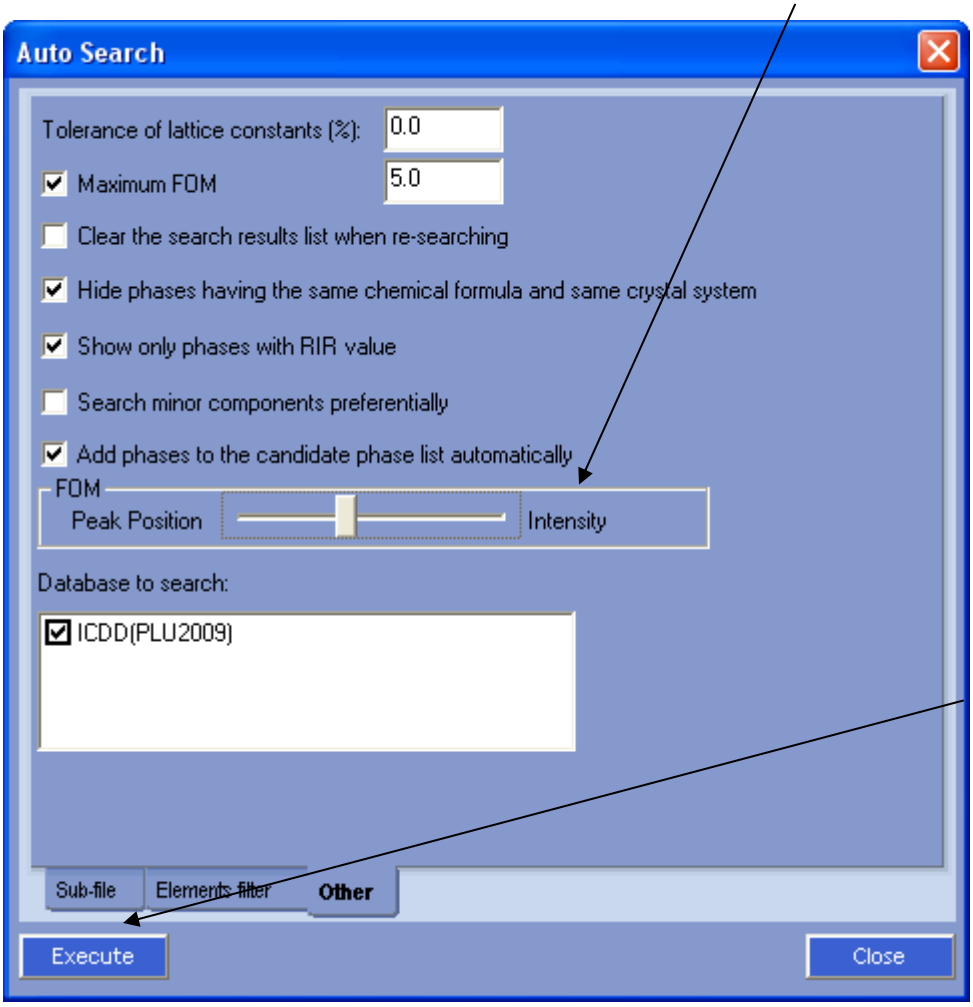


Notice that most of the elements are listed as possible or green, not required. This is a wide search but not wide open. It will screen out some unnecessary phases and allow you to concentrate on just the best ones

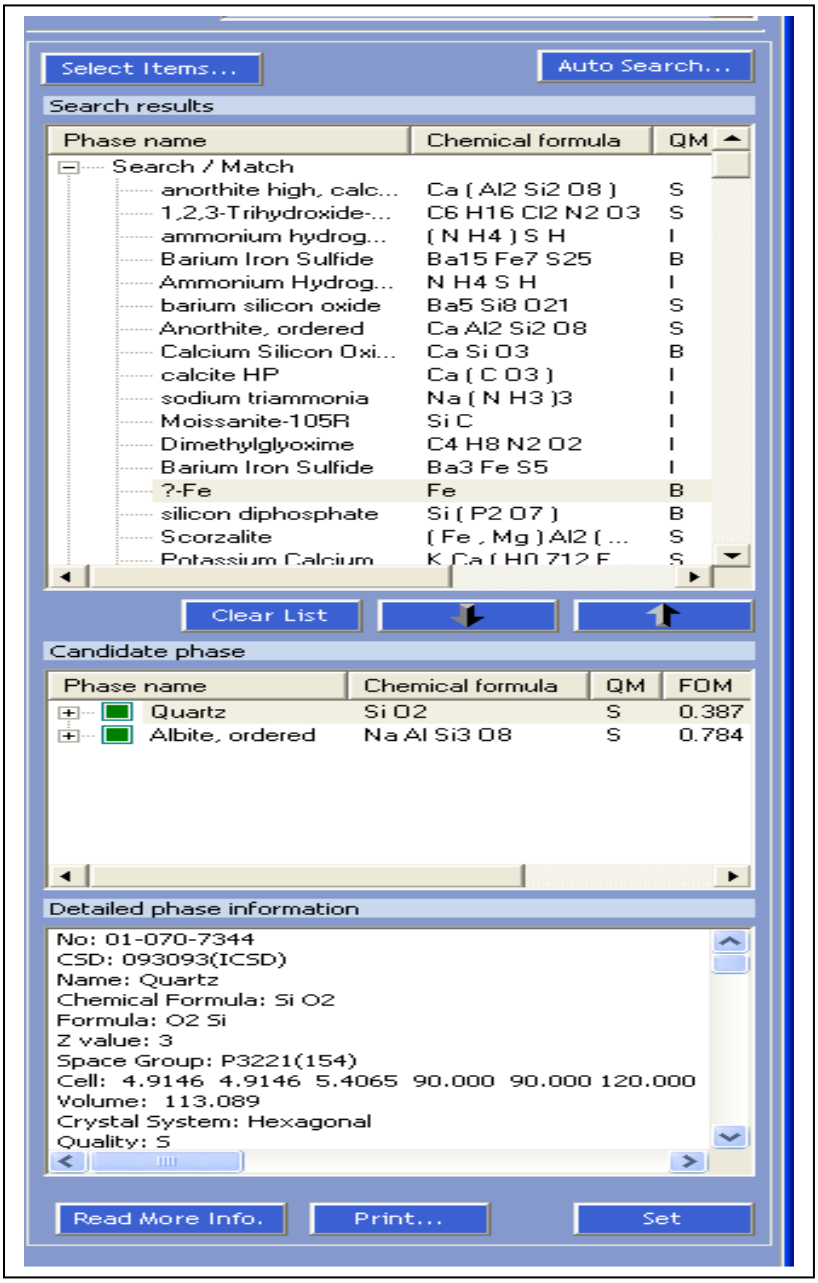
9. Next tab at the bottom “Other” is advanced or special options.

10. Type in “0” for the tolerance of lattice constants (5) shift option. Check the Maximum FOM box and set the value equal to 5.0 or smaller. A setting of 3.0 is very picky and can sometimes discriminate against phases in a poor data set or with lots of atomic substitutions or hydrates. If you set the range too small, you may not find the material you seek.

11. There is a way to weight the data on either better peak positions or on better intensity matching. This is good to change if you have preferred orientation in a sample. Move the slide closer to peak position for preferred orientation as the intensities will not match as well.



Press the execute button to start the search.



12. A list of possible options based on your search criteria appear in the right side “Analysis-Identification (Auto Search) menu.

Information is listed in three windows; top window is possible matches, middle window is Good Candidate phases that have a high statistical match potential, and lower window is a detail information view of the selected or highlighted phase in the candidate window.

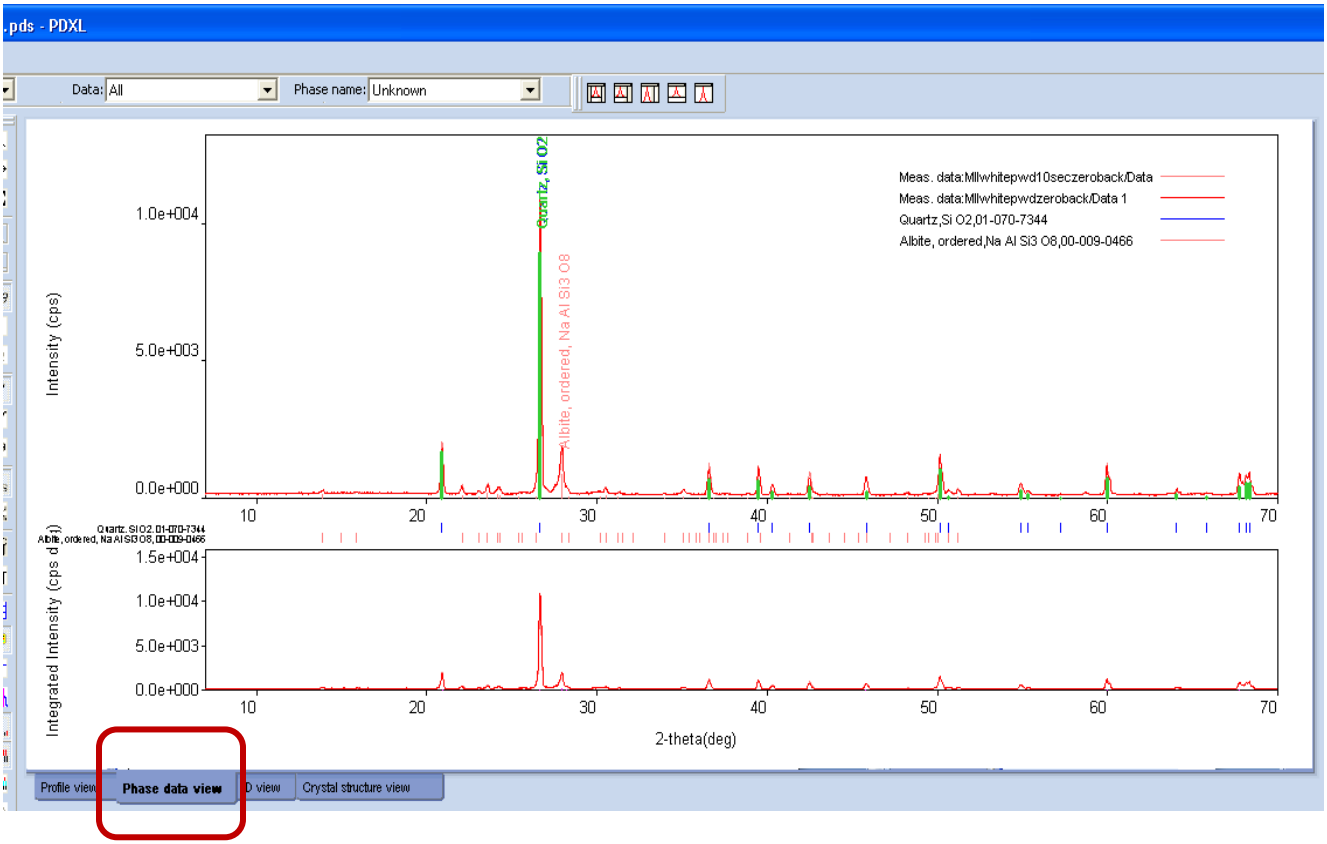
A search on this XRD pattern, revealed two good potential candidates (boxes are green in front of both phases):

Quartz and Albite are the two best possible Primary phases found (FROM OUR SEARCH CRITERIA).

They are both Star quality phases (QM column) and the FOM or figure of merit number for both of these phases is very low (low FOM are desired). The ROM for Quartz is 0.387 and for albite the FOM is 0.784.

Confirmation of Phases:

13. To visually check the fit of the two potential phases, click on the “Phase data view “tab at the bottom of the XRD pattern plot:

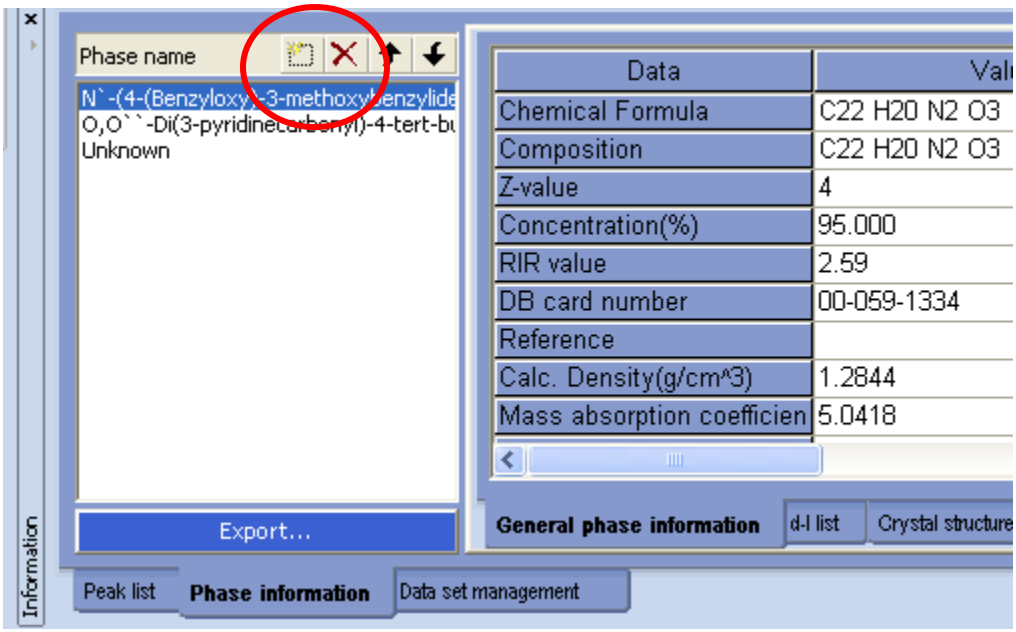


14. Peak Heights of Phase Id markers can be increased or decreased to check visual confirmation. Click once to highlight the phase in the candidate list to the right and then click on the graph (in the “Phase Data view” tab. Roll the mouse wheel up or down or move the mouse up and down while holding the left mouse button, to increase the Phase id bars on top of the graph.

15. Other choices from the upper level window can also be evaluated by clicking on the phase name. If a phase in the upper level window is considered to be a good fit, click on the phase name the click on the downward arrow located between the two windows. To remove a phase from the candidate list, click on the upward arrow located between the two windows. When the phases are

considered to be accurate, press the “Set” button on the lower right side window to lock in the results of the phase identification.

If phases need to be removed after the set button has been pressed, click on the “phase information” tab located on the lower center window, highlight the incorrect phase, and click on the large black ”x” for removal.

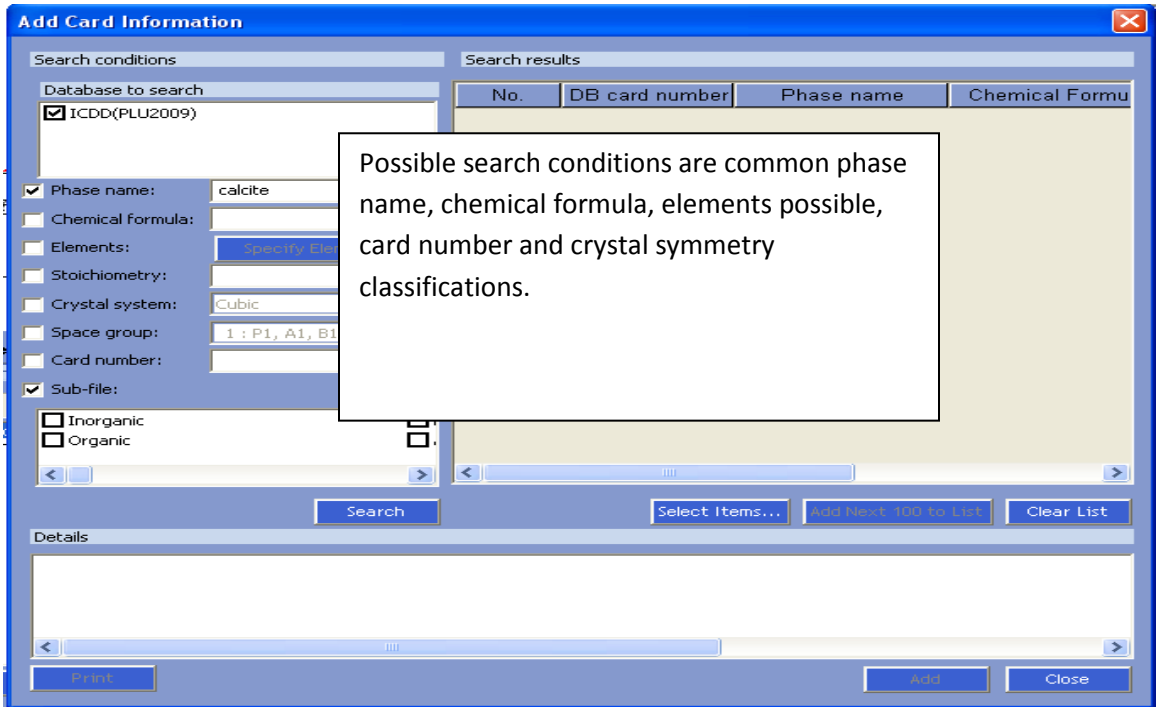


Direct Database Searching (without matching pattern or DATABASE MINING).

1. To find a pattern manually, click on the next button down in the flow bar labeled --“Load card info”.

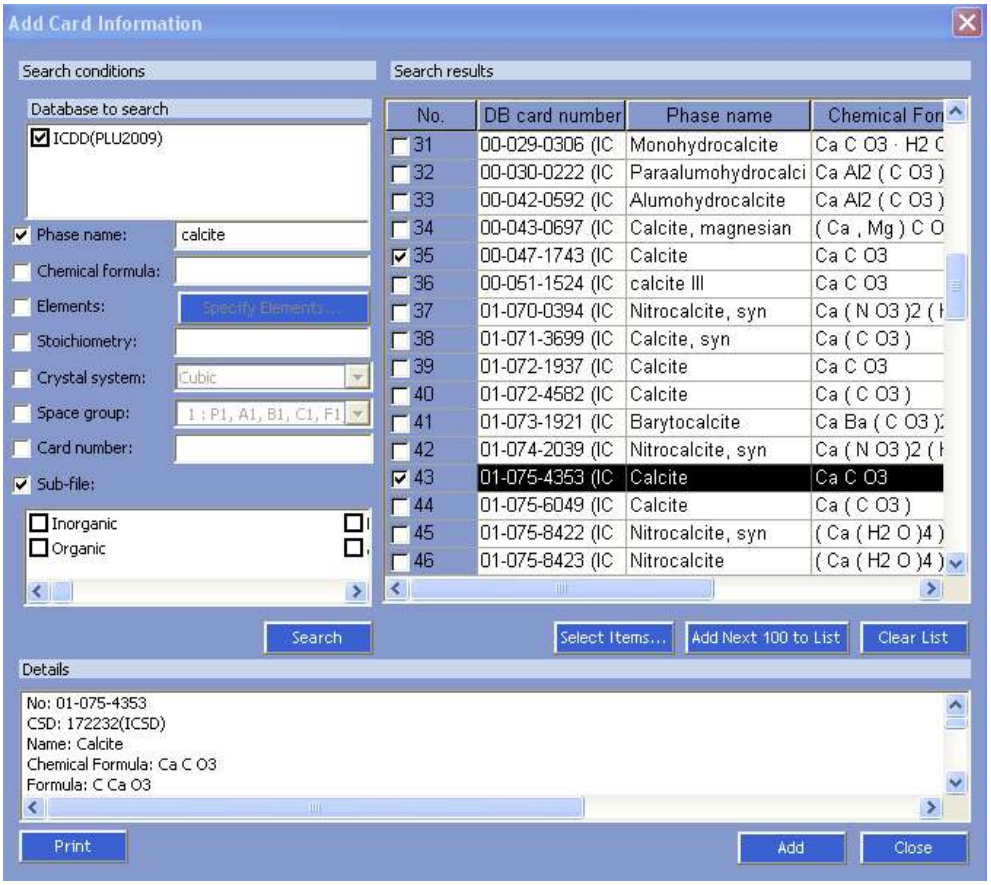
2. Two additional buttons will appear on the far right side window labeled “Card Info” and “Import”.

3. Click on Card info and enter information about desired phase…



4. When the search conditions are entered, click on the search button to observe all phases that meet listed requirements.

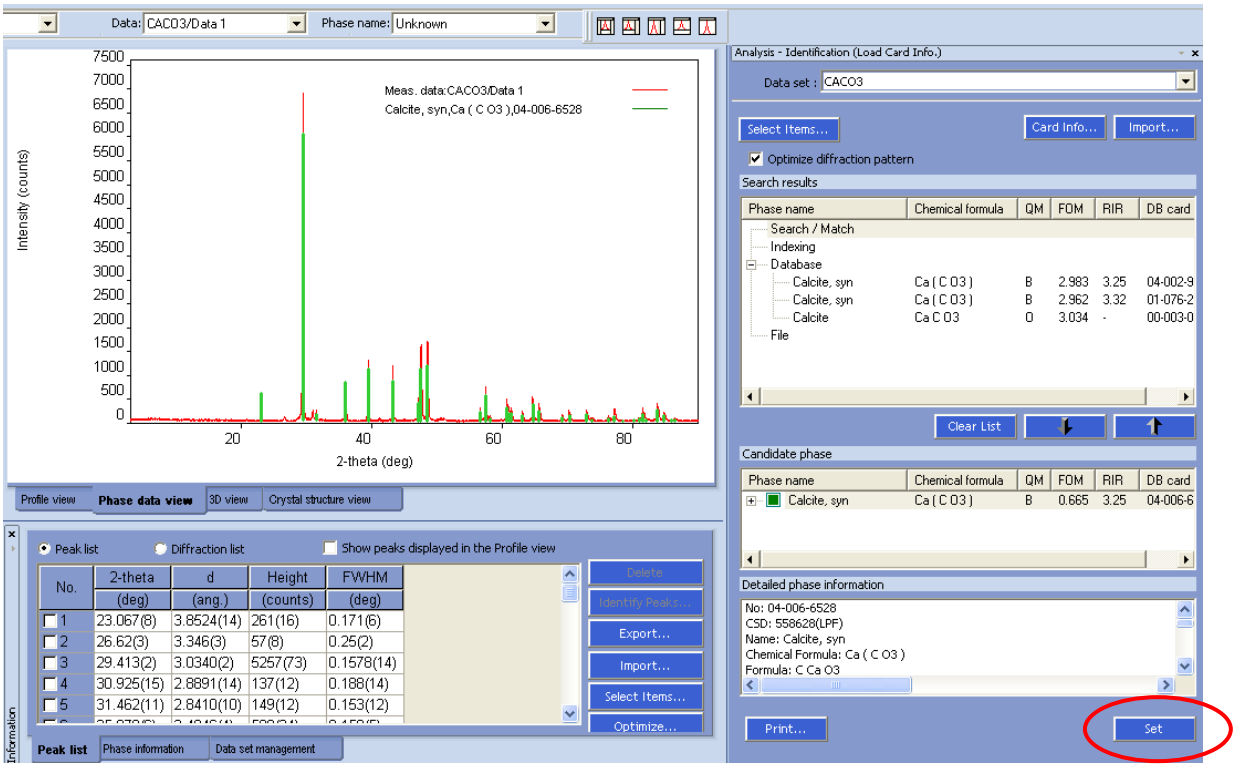
5. Results will appear in the same window to the right of the search parameters.



6. Possible phases can be visually evaluated by clicking on the boxes in front of each phase.

7. To add all selected phases(check boxes in front of each line) to the Search Match window , click on the “Add “ button in the “Add Card Info” window. Close this window. Elevate the FOM and visual goodness of fit for newly added phases. If chosen, click on the phase in the upper window and then move into the Candidate window by pressing the downward arrow.

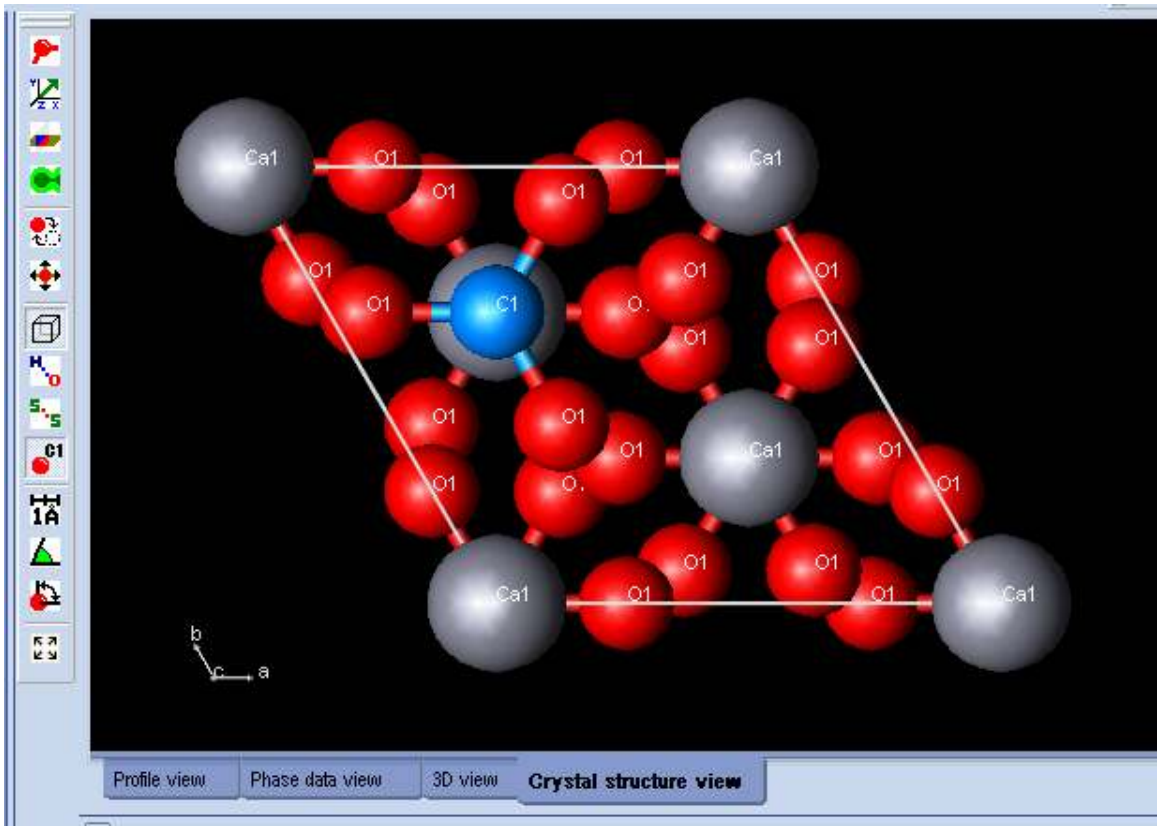
9. Don’t forget to press “Set” that registers the results when finished with the Search Match.



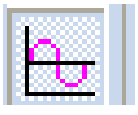
To add CIF directly, Click on the LOAD CARD INFO button on flow diagram (on left hand side).

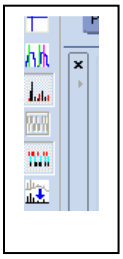
1. Click on the “import” button on the far right hand side and locate cif file. Click to open.
2. 

3. Once phase is loaded, click on the downward arrow to move it into the Candidate Window. Press set button on lower right hand side then click on the Crystal View tab to see 3D view.

4. 

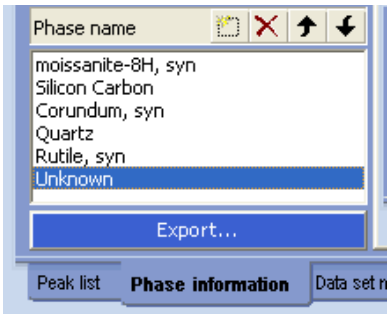
PRINTING PHASE IDENTIFICATION REPORTS

1. When phase identification is complete, this information can be put into a report format. Make sure that the difference window appears under the plot. If it does not click on the last button in the PDXL tool bar (vertical one)—button is called “Residual Graph” 

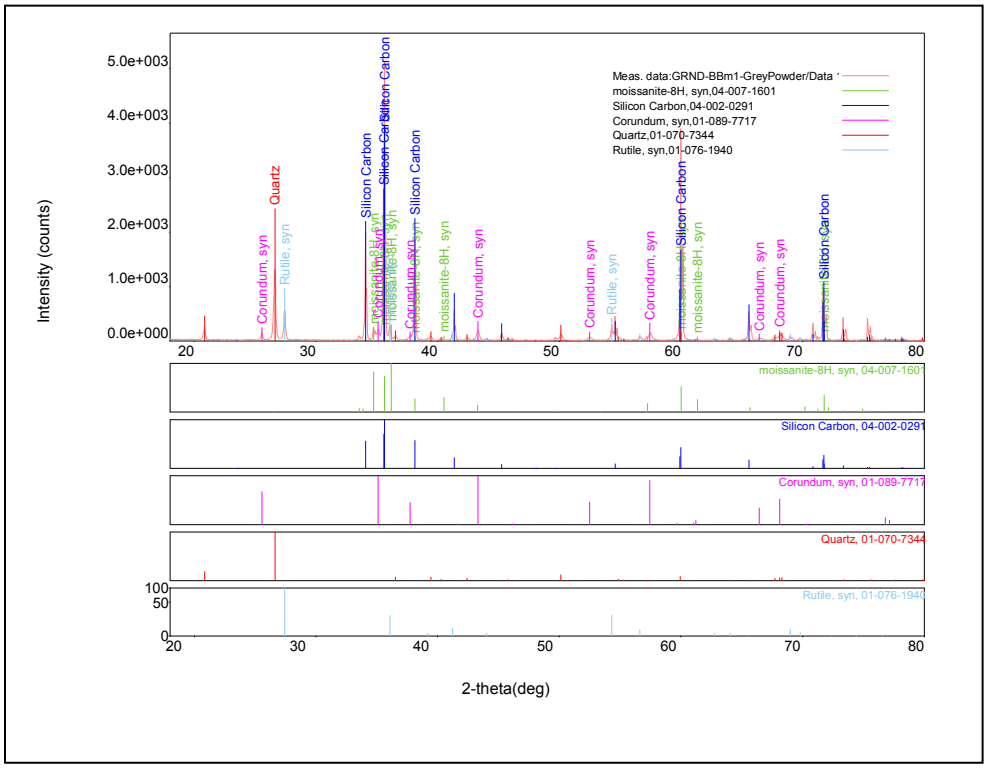


2. Then Click on the third peak bar button on the same vertical tool bar. Button is labeled “Peak Bar (3)”

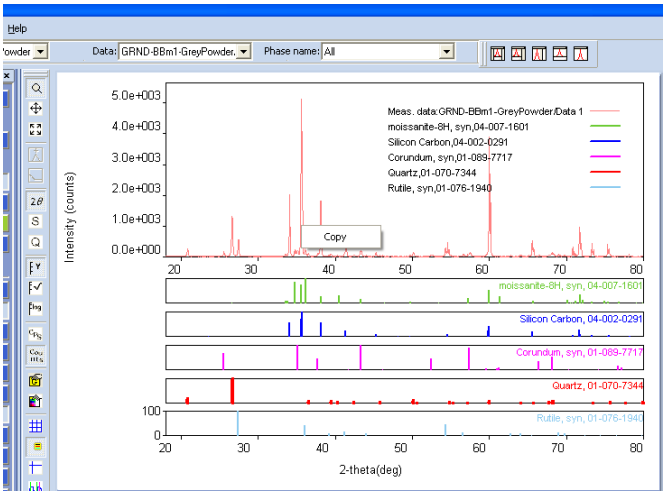
3. Then delete the “unknown” phase from the “Phase List” tab located in the lower center window. To delete, highlight the “unknown” phase with a single click and then click on the X or delete button at the top of the same window…



Graph should look like this…

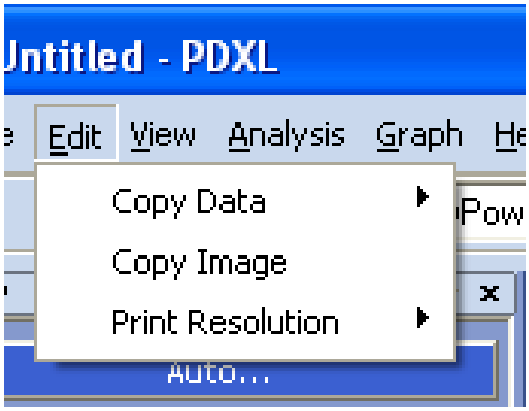


4. To remove labels on top of peaks, click on the first “Peak Bar (1) button that looks like this…



5. Plot should look like this…Right mouse click on the Red raw data pattern, till you see the word “Copy” appear. Click on the word “Copy”. Open a document or excel spreadsheet or power point and press “Paste” in the document to paste image of plot.

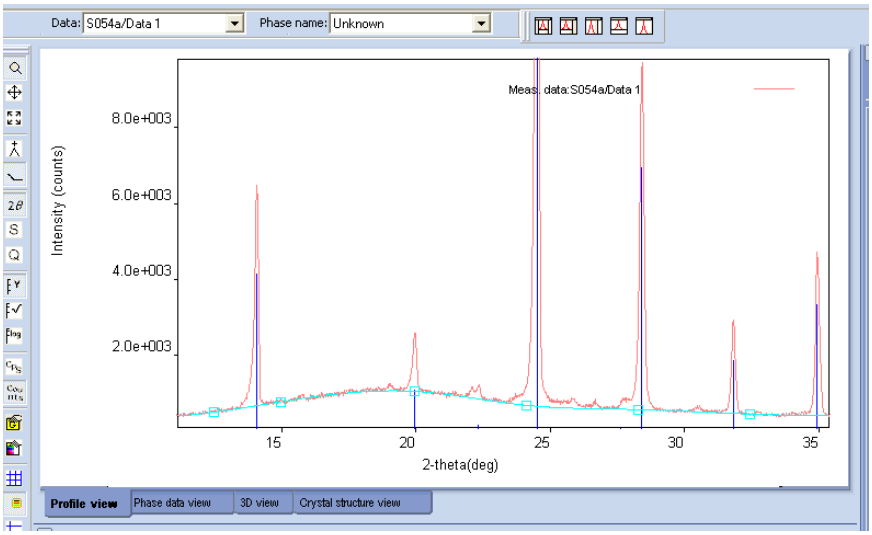
6. An alternative method to copying the plot is to go to the menus at the top of PDXL…”Edit” then “Copy Image”…then open the document and paste into your normal report format.



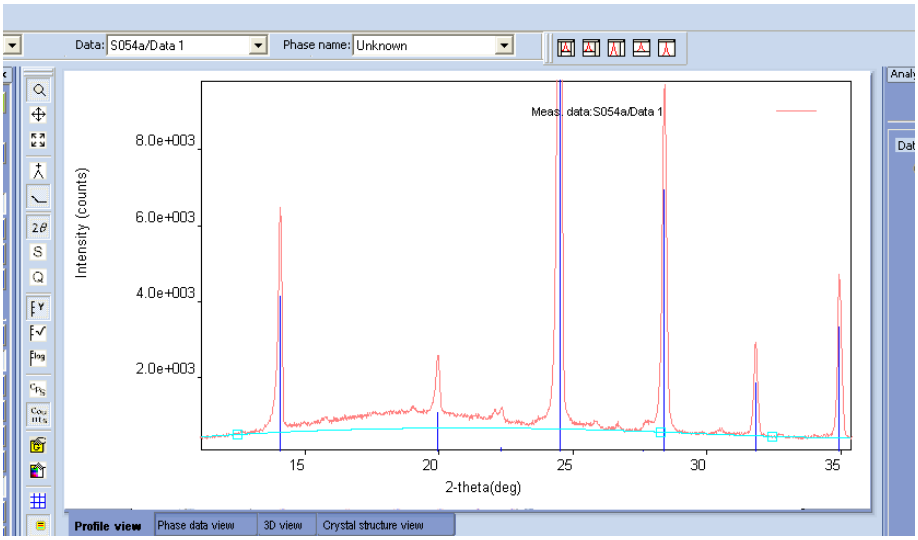
x. Crystallinity

(ALWAYS ALLOW FOR EXTRA DATA COLLECTION RANGE TO ENSURE PROPER BACKGROUND FITTING):

1. Open data. Click on edit background button. Adjust background to form a linear (straight) background. Be sure to allow for amorphous regions (do not model out the amorphous areas by allowing the background to rise up under the crystalline peaks).

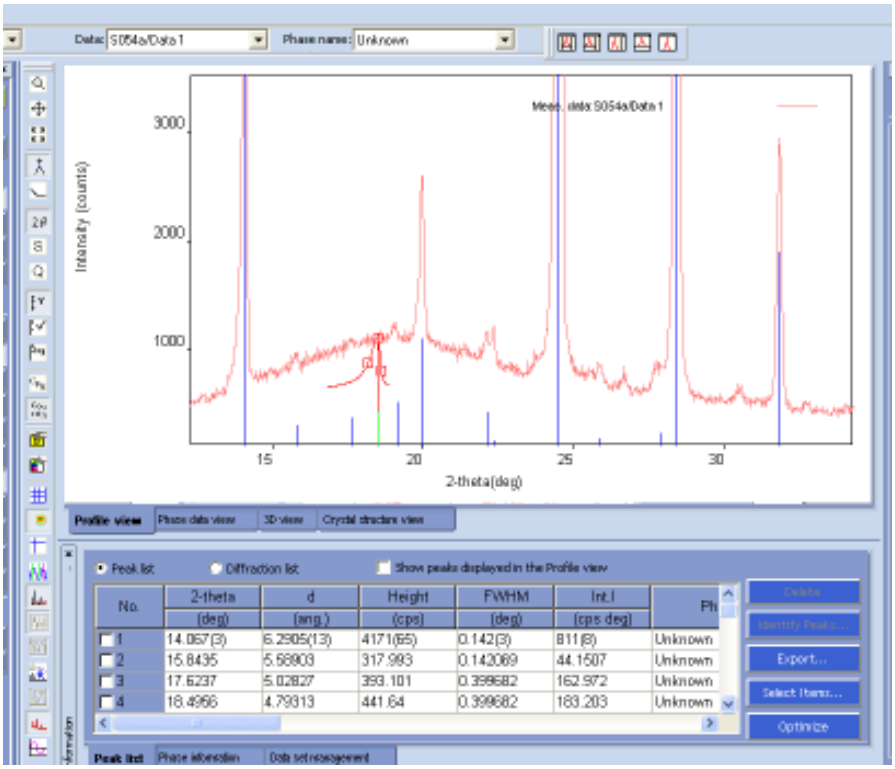


2. Usually a right mouse click on blue squares (when in background edit mode) will remove curvature and allow for a more linear background model….

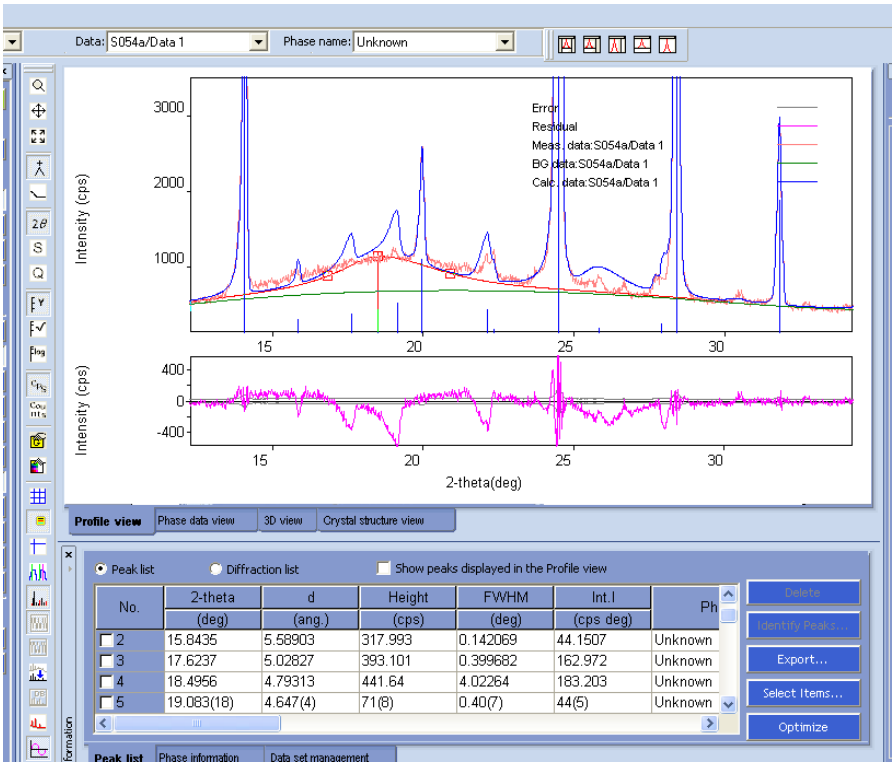


3. Automatic background fitting allows for curvature to remove amorphous regions for Search Match of crystalline phases.

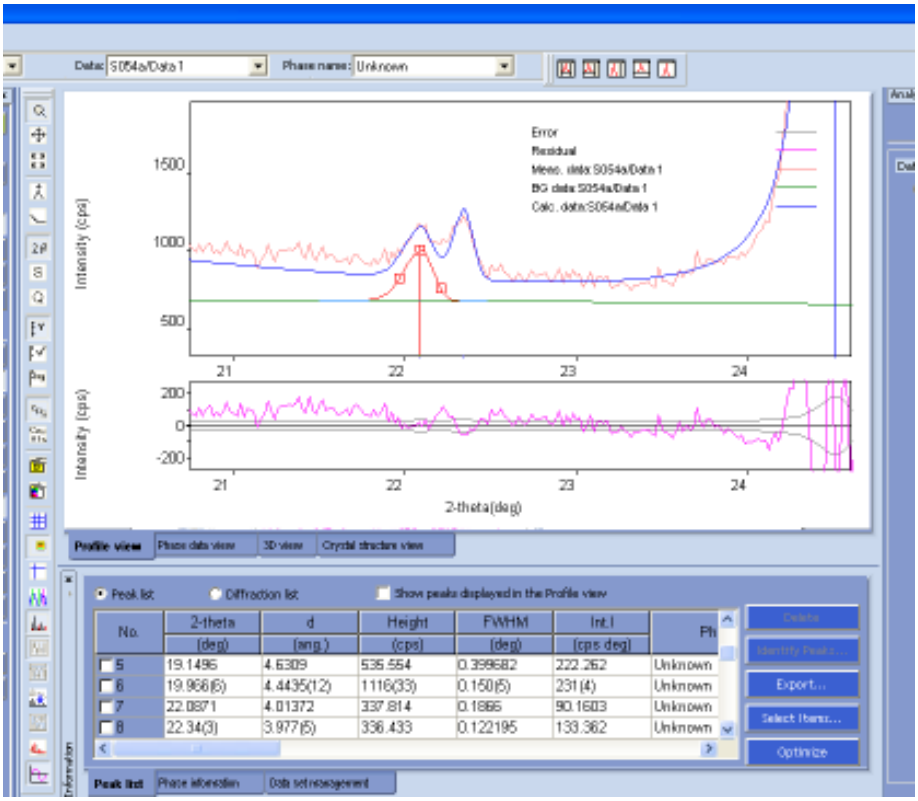
4. The appropriate background model for calculating percent crystallinity uses a linear model. Adjust the current background model to simulate a linear background fit or manually add a linear background. Insert additional peak(s) for amorphous region(s):



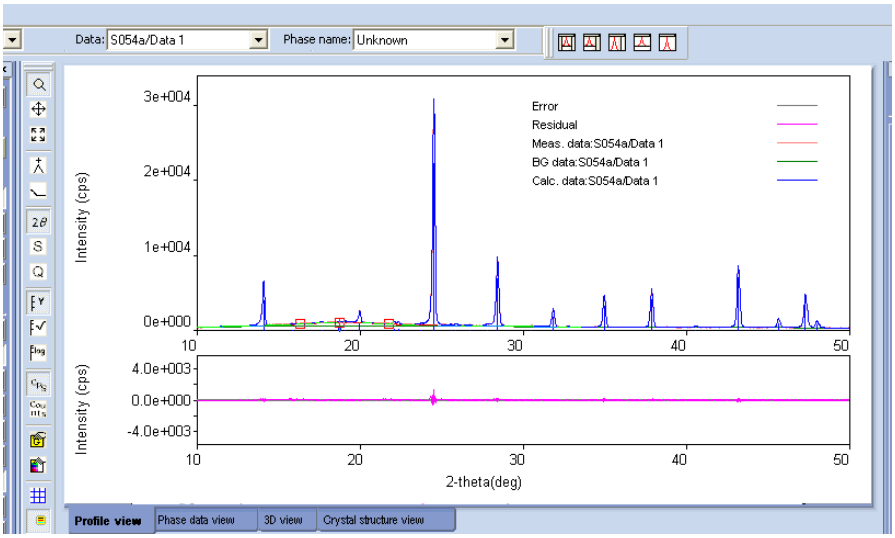
5. Edit all peak position, shape and intensities (if needed). To edit, click on the peak position in peak list (while in Add peak mode) and 3 red squares will appear on the peak (in Profile view). These squares will edit the shape and intensity of the peak. To edit the position, click and hold the peak bar and drag to new location.



6. Edit all peaks until blue difference line matches the experimental data.

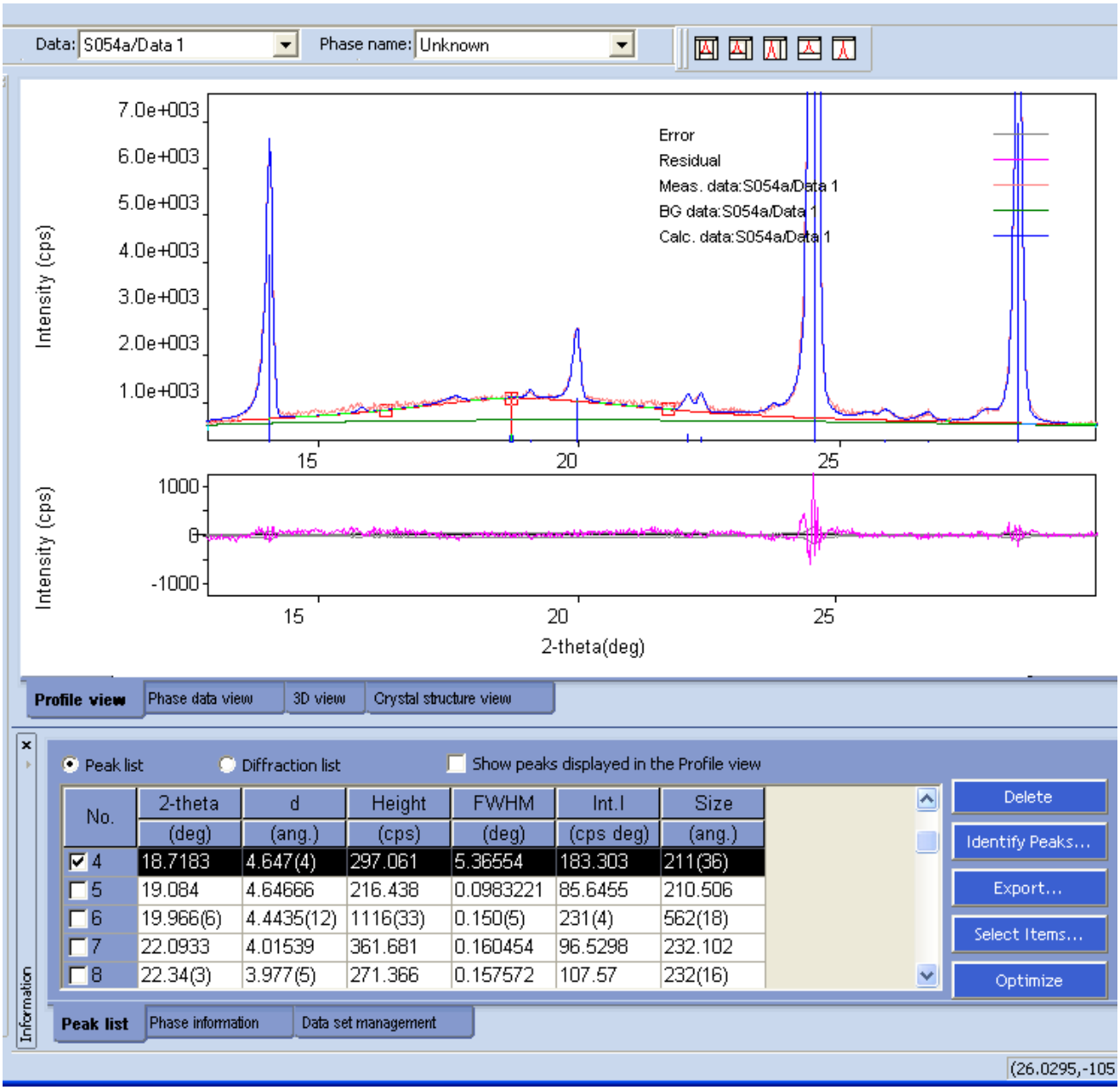


7. Edit all peaks until blue summation line (in Profile View) line matches the experimental data.

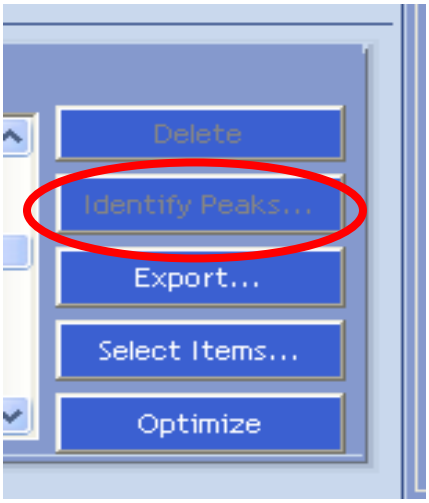


8. Additional confirmation of good fitting can be found on the purple difference located on strip under Profile view. Line should be linear or flat if a perfect fit is achieved.

9. Find **all** amorphous peaks and click on the box preceding the peak on the peak list tab.

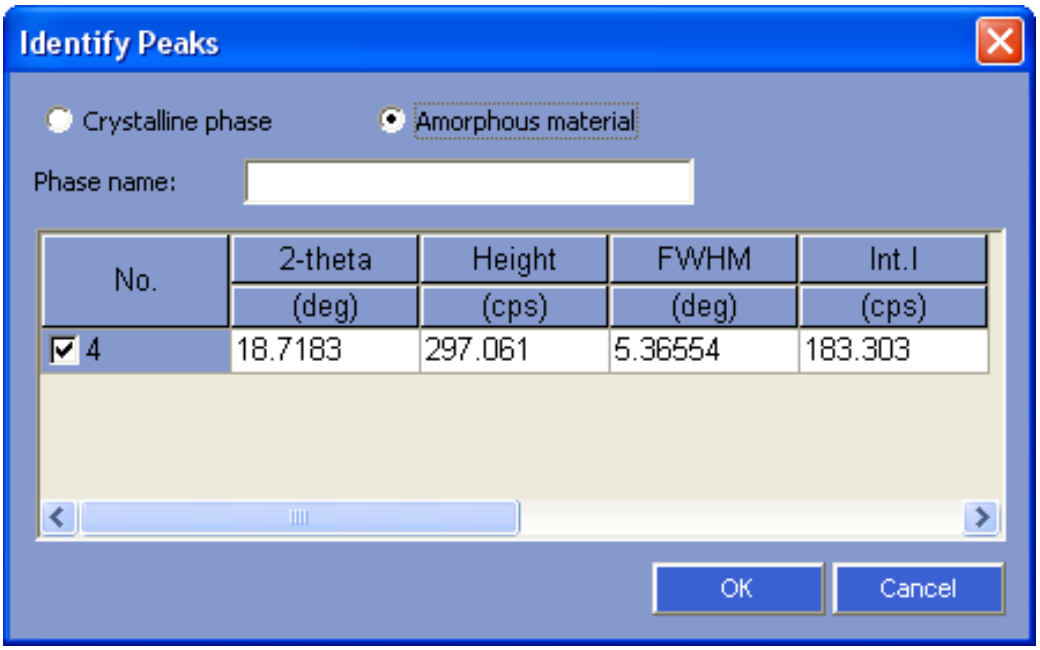


No.	2-theta (deg)	d (ang.)	Height (cps)	FWHM (deg)	Int.I (cps deg)	Size (ang.)
4	18.7183	4.647(4)	297.061	5.36554	183.303	211(36)
5	19.084	4.64666	216.438	0.0983221	85.6455	210.506
6	19.966(6)	4.4435(12)	1116(33)	0.150(5)	231(4)	562(18)
7	22.0933	4.01539	361.681	0.160454	96.5298	232.102
8	22.34(3)	3.977(5)	271.366	0.157572	107.57	232(16)



10. Click on the “Identify Peak” button located on the right of the Information window:

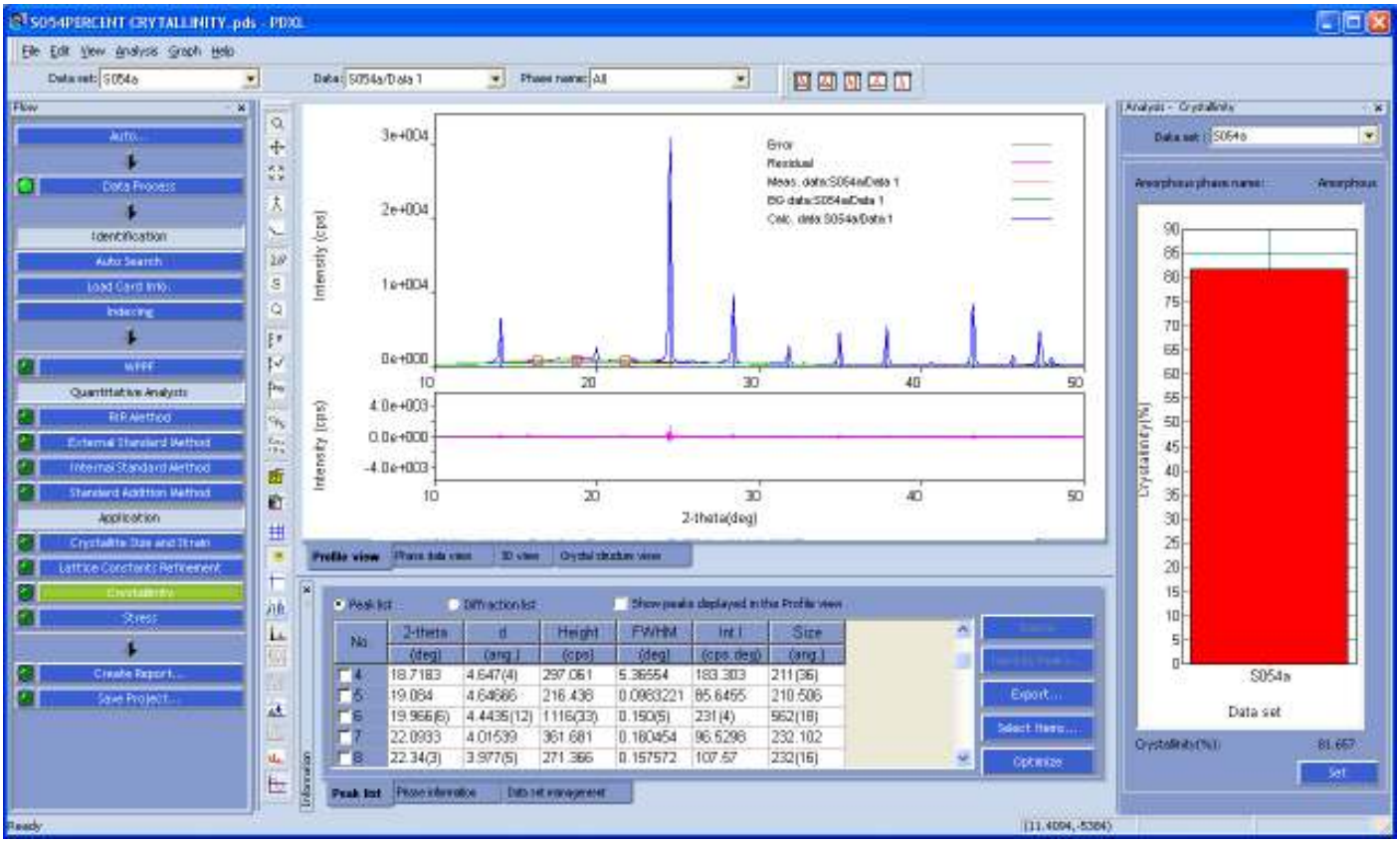
11. Click on Amorphous material button to Tag or identify the peaks as “Amorphous”. Click on the “Ok” button.



12. Usually, by average definition peaks with a fwhm of 5 degrees or greater are typically regarded as amorphous. Definitions vary depending on the industry and the material properties.

13. After the peaks are designated or tagged as Amorphous, click on the flow bar button “Crystallinity”

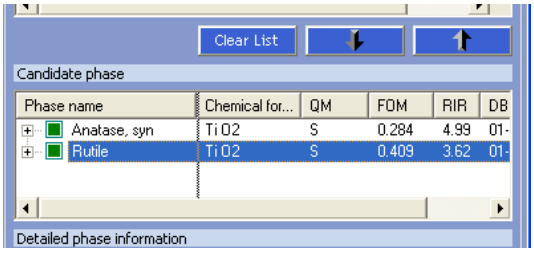
14. Immediately the results should be displayed on the far right side in the analysis window.



XI. Rietveld Refinement or WPPF:

1. Tip: To perform quantitative analysis using WPPF, either a crystal structure information file is needed (*.cif, or *csf) or a RIR (relative intensity ratio from the database file) is needed.

a. Check your phases in the Candidate Phase section to see if a value is listed in the RIR column. If you cannot see the RIR column, move the cursor to the column header line and re-size the column widths until the RIR column is visible.



Use the RIR method in the flow bar if you do not have crystal structures available from ICDD PDF4 or from ICSD /NIST/ FIZ.

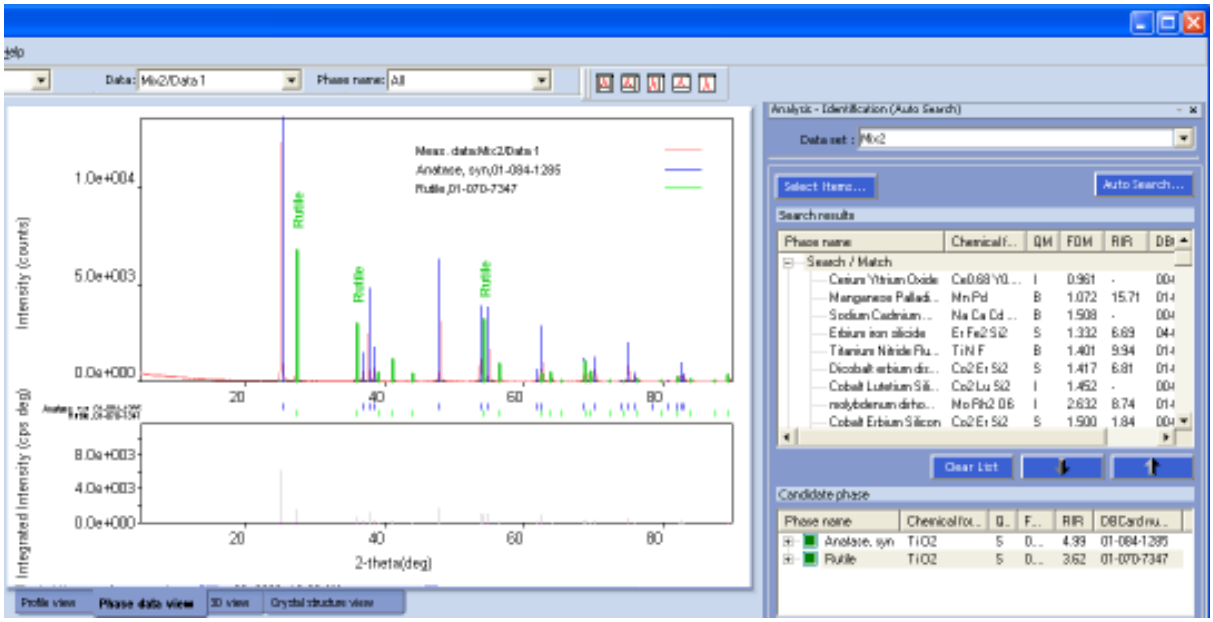
b. To check for crystal structure information, after pressing the set button on the lower right window, go to the center window and click on the “Crystal structure view” tab under the diffraction plot.

c. If your phase does not have RIR value, go to load card info tab on the left and input the same chemical composition as the one in your desired phase. It is highly likely that you will find same phase with RIR.

XII .Setting up the Rietveld Refinement (WPPF):

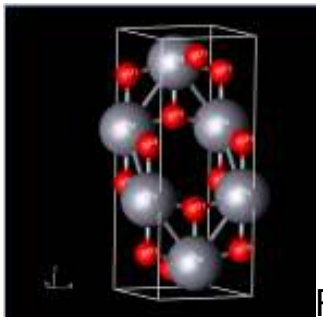
Example from the Demo data in PDXL:

1. Open raw data MIX2.ASC.
2. File is found under C:/Program files/ Rigaku/PDXL/ DemoData/ Quant_Rietveld
3. Process with auto peak search and Auto Search for Phases

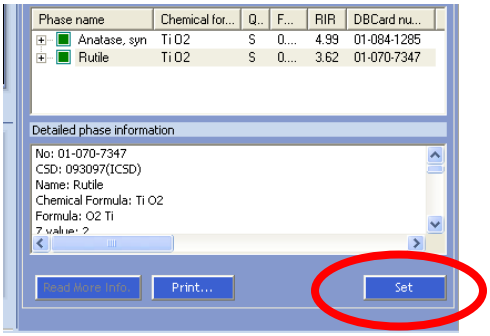


4. Go to the “Phase Data view” tab, to see the fit of each of the candidate phases. Click on phase in Candidate phases list and move cursor to the line markers on the diffraction plot. Roll the mouse wheel up or down to increase or decrease the phase line marker.

5. Go to the “Crystal structure view” tab to see if the phases have crystal structures



Rutile Phase (TiO_2)

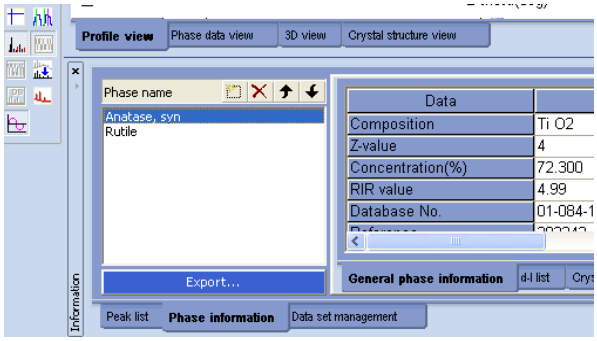


6. Press the “Set” button on lower right window of auto search to ensure phases are locked.

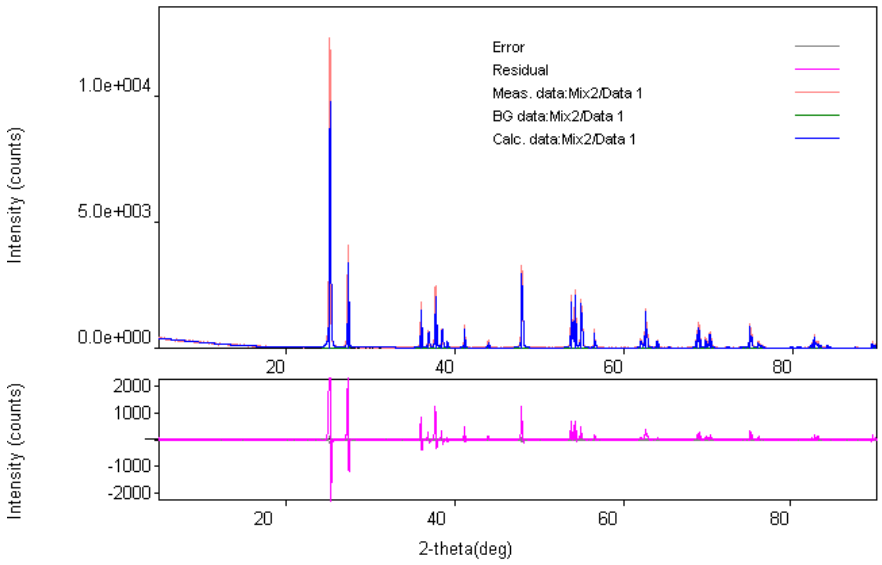
7. Click on WPPF in flow bar of left side menu. A new window will appear on the far right side.

8. In the center window, click on the “Profile view” tab. This window will show the difference or residual plot. This will show results of the simulated pattern versus the raw data and indicate areas that may need additional refinements.

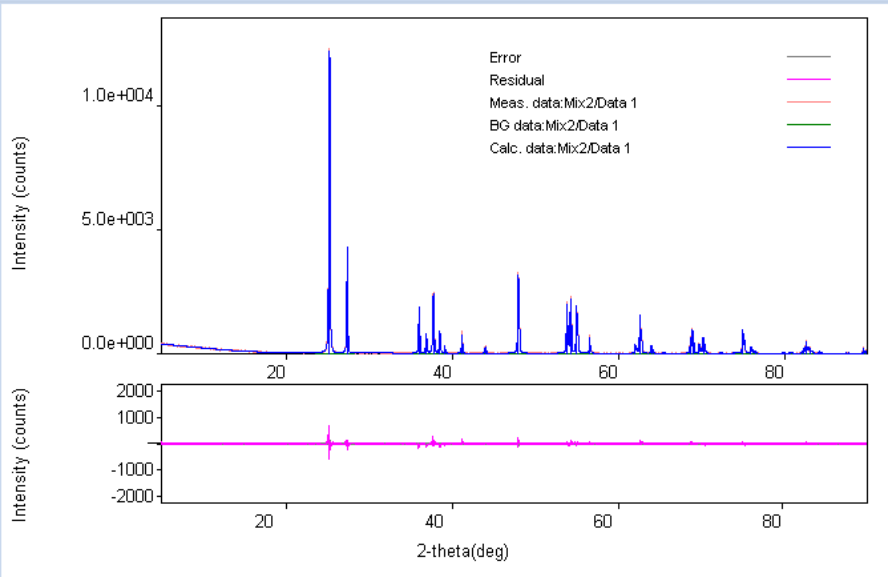
9. Go to the Phase Information tab at the bottom of the center window. Click on the “Unknown” phase and press the delete button (looks like a large “X”).



10. On the lower right side, press the “Simulate” button, then press the “Refine” button and watch the difference plot in the center middle window. The purple difference plot of an ideal refinement would be perfectly flat. When the analysis is completed, press the **“set”** button on the lower right window. This saves all the results of the refinement.



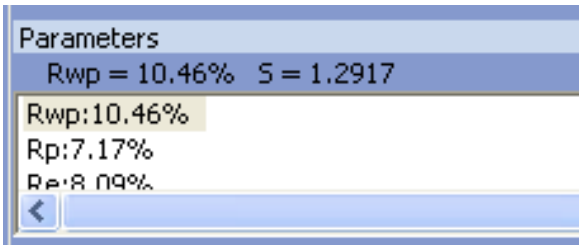
“Simulate” button pressed.



"Refine" button pressed.

11. After the refinement is finished, go to the "Profile view" tab on the upper middle window-"Profile View" window. Compare the goodness of fit (visually) better the Red Raw data pattern and the Blue simulation line. Look for areas where the Blue simulation line does not follow the overall trend of the Red Raw data.

12. Look at the statistics listed on the lower right hand window.



These are good values for a refinement:

Rwp= 10.46% S=1.2917, Rp=:7.17% Re=8.09% Chi^2= 1.6685.

Rwp is the residual whole pattern. A value around "10" is considered to be a fairly good refinement. Rwp residual factor is most important.

Re is the minimum Rwp value reachable using a certain number of refinable parameters. Re is the best expected refinement value based on the raw data quality.

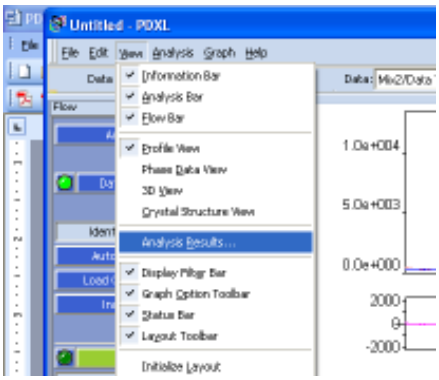
S or the goodness of fit (Gof) is usually defined as a ratio of Rwp over Re. So S cannot be less than 1. Really good refinements gives S lower than 2.

Rp=residual of the profile. The Rp value is usually always better than Rwp.

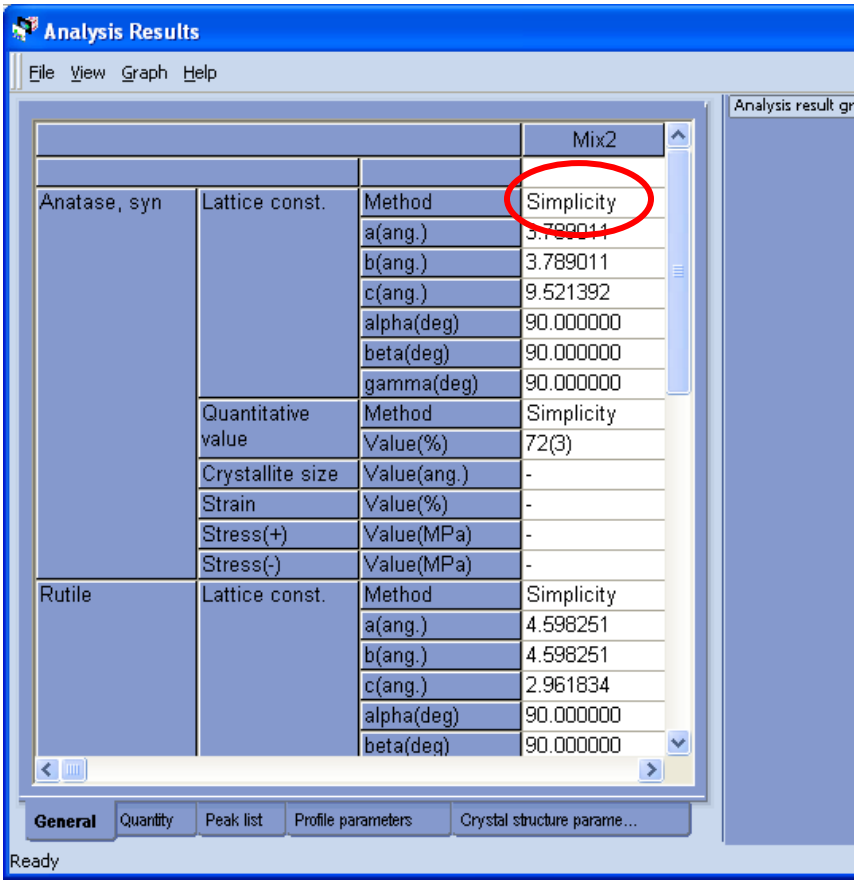
Chi squared for a perfect theoretical refinement should be 1.0. A perfect value is seldom achieved.

13. To see the results of the WPPF quantitative results… go to the menus at the top and click on

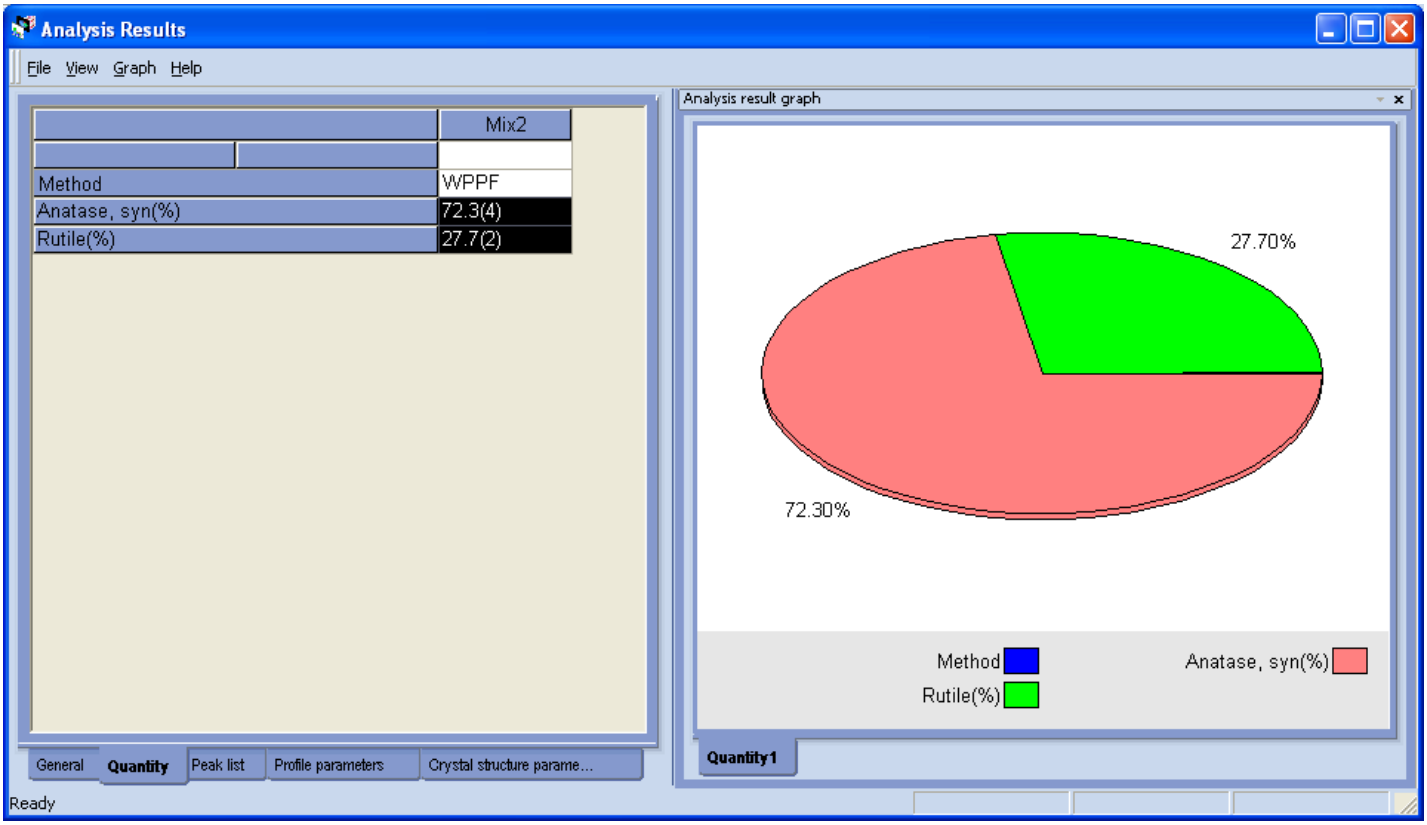
"View" then analysis results…



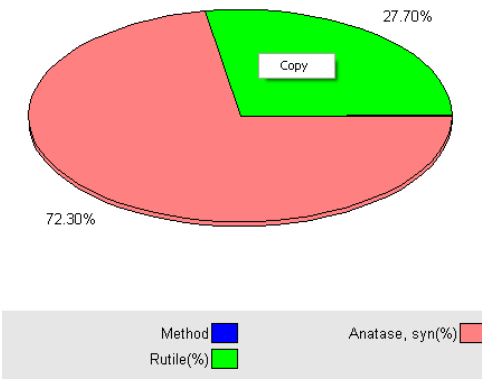
14. If the word "Simplicity" appears on the "General" tab of the view window, then the "set" button described in step 11 was skipped. Close the view window and then press the "set" button. Re-open the view window to see the final results.



15. If the word “WPPF” appears on the “General” tab of the view window, click on the “Quantity” tab. Click once on the WPPF word to highlight the cell and drag down to highlight, then click on the “Graph” menu at the top of the view window and click on the “Pie” chart option. A pie chart should appear in the window to the right…

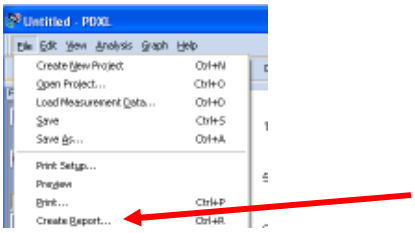


17.



17. Right Mouse click on the pie chart to copy the image and paste into a document

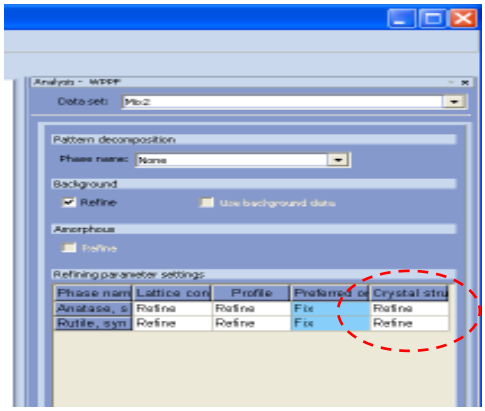
17. Or for an alternative method of reporting, click on the “File” menu and then the “Create Report” option…



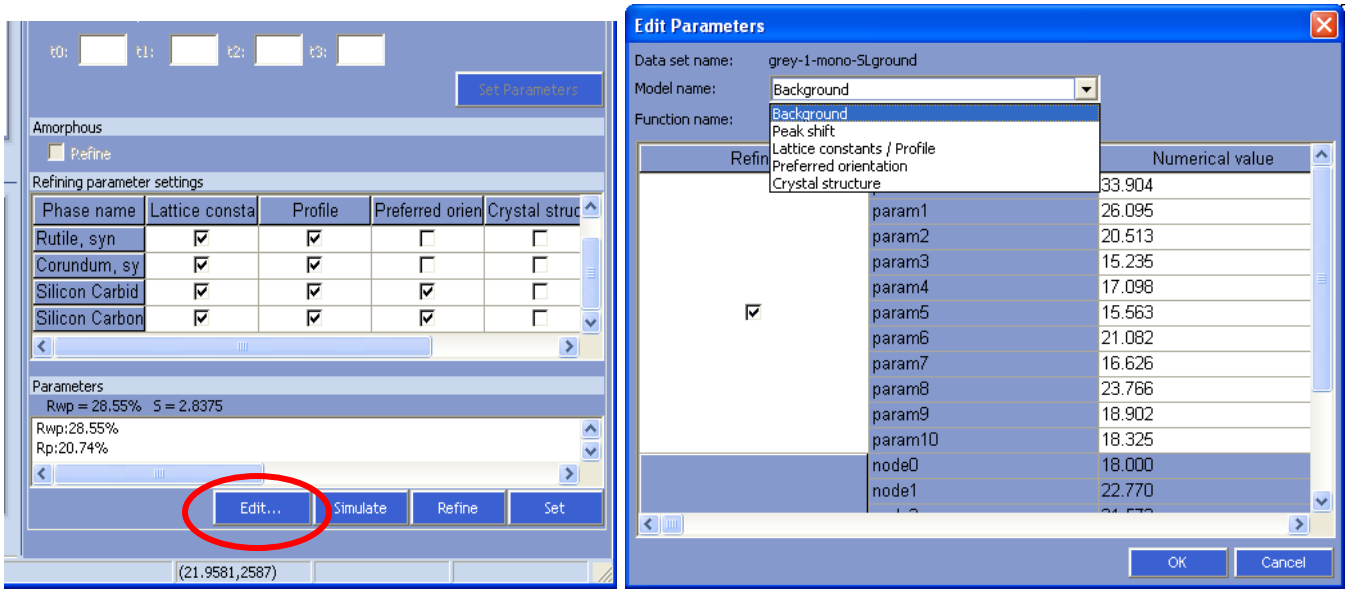
Another menu will open listing all the options for creating and printing an MS word report. This will automatically create a report with a much or as little data as selected.

Advance WPPF Features :

18. You can refine crystal structure provided there is enough data (data/parameter ratio is 5 or more). In that case one should check bond distance and angles to make sure if they make sense (from the perspective of literature values) and esds associated are low.



19. To adjust for preferred orientation or a skew in the intensity fitting, click on the Edit button in the WPPF (on the lower left side). A window will appear.

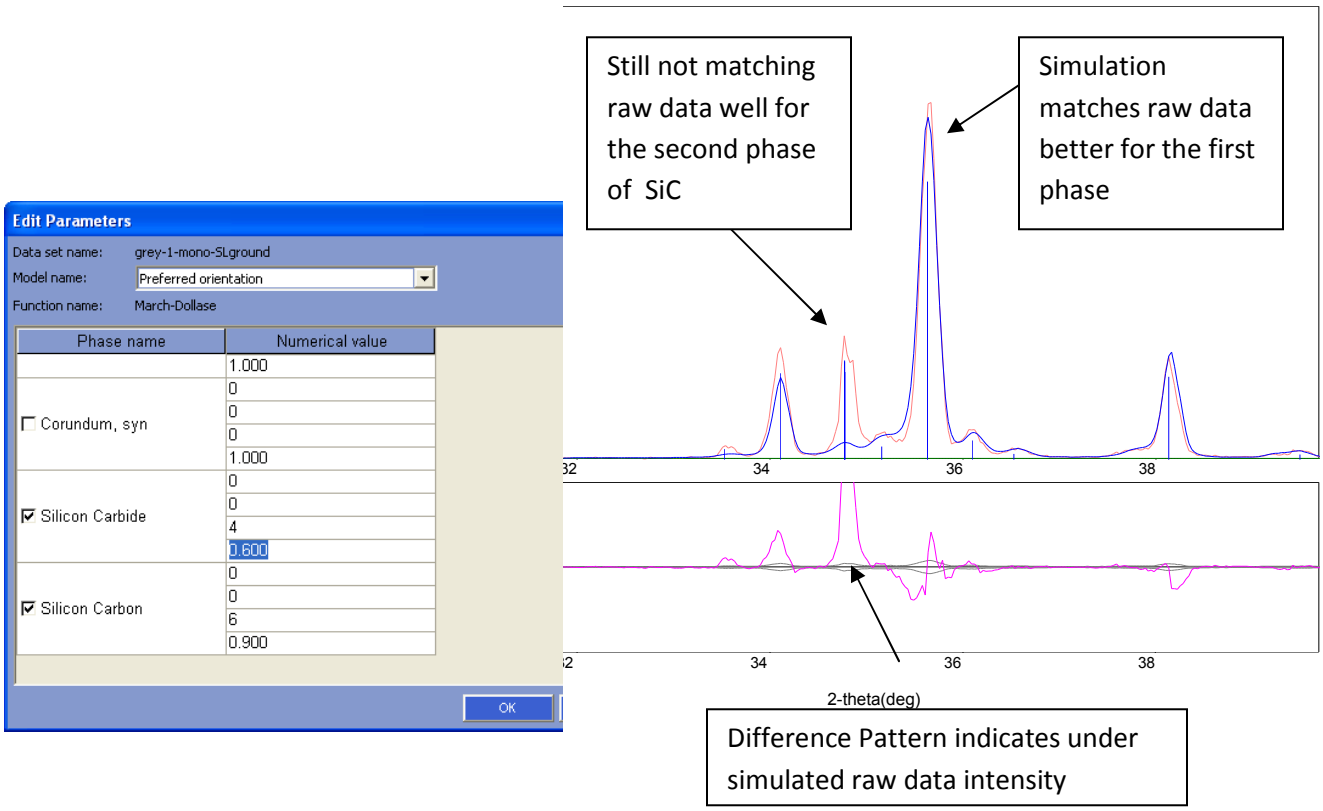


20 (a). In the drop down menu, choose the Preferred Orientation option under Model Name:

20 (b). Check the box in front of the phase or phases that have preferred orientation. Identify the direction in reflections or miller indices of the orientation (exaggeration of intensity). Enter the number of the reflection in individual components (i.e. if oriented on the (002)) enter “0”under h and “0” under k and “2” under l).

20(c). Then by trial and error determine a good starting orientation vector for the March coefficient. For March coefficient the smaller the number, the larger the degree of orientation—1.0 is random and has no additional orientation—0.9 is a very smaller degree of orientation—0.5 is a high degree of orientation.

20 (d). Start with a value of 0.8 for the March coefficient on the phase that has low intensity on a peak. Press the “okay” button. Press the “simulate” button back on the WPPF window and observe the goodness of fit (visually) between the red and blue lines of the “Profile View” Window. Are the intensities going in the correct direction or are they getting worse? Make more adjustments until the “simulation” is going in the right direction. Press the “Refine” button in the WPPF window when you are getting closer. Has the Rwp and Rp value changed for the better? (Gone to lower or smaller numbers?).

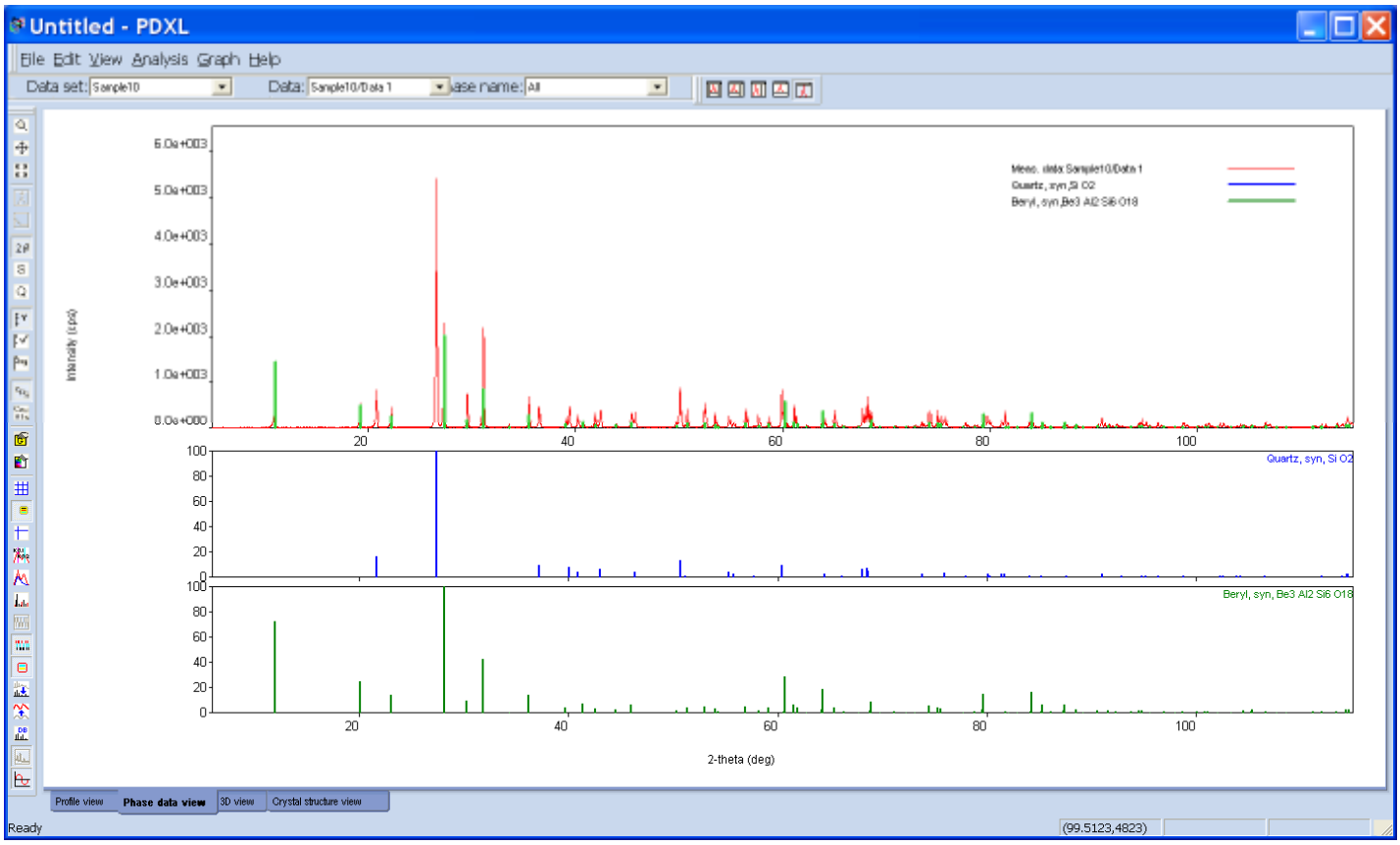


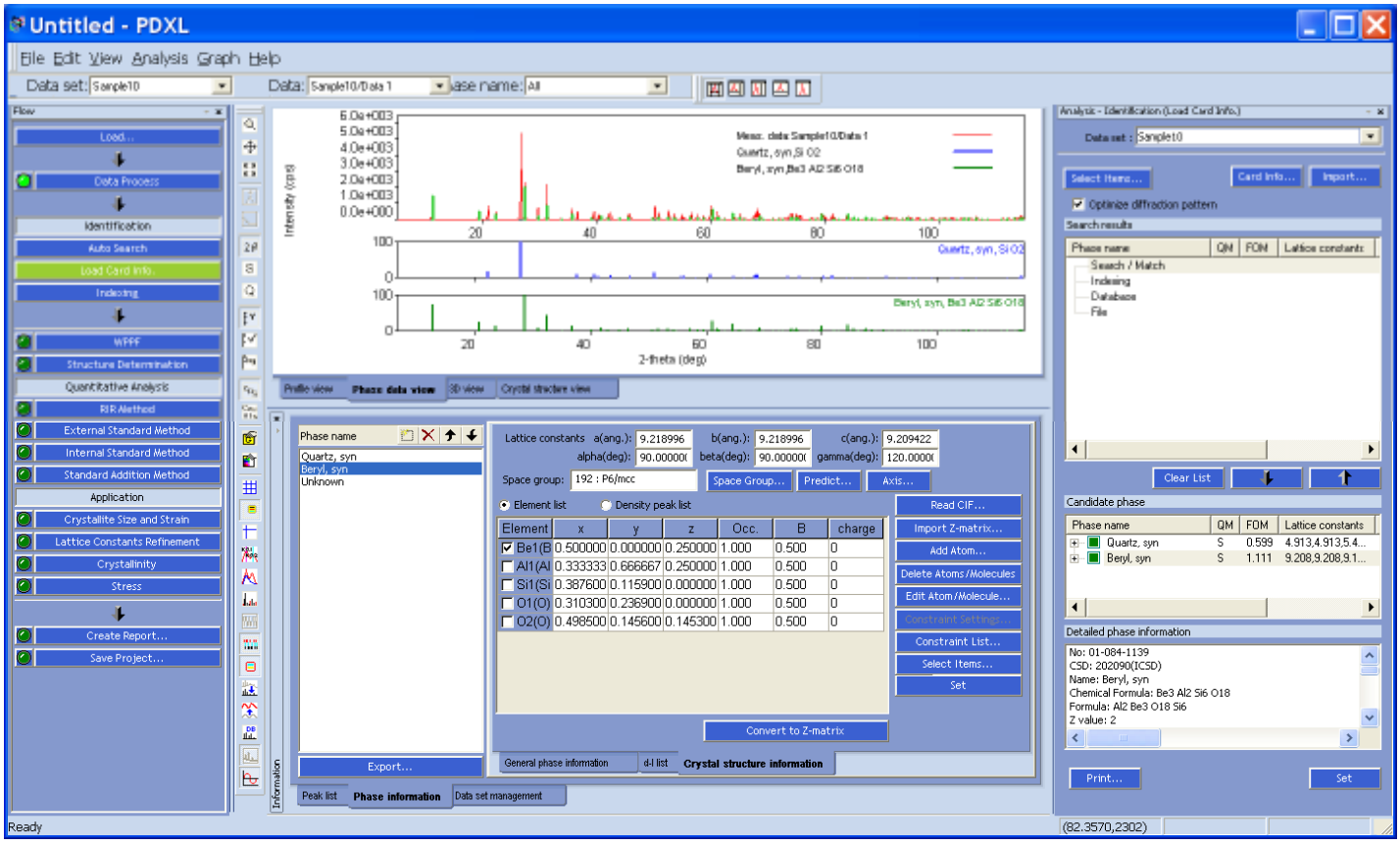
21. If you have a solid solution or if your phase has a different chemical composition but the space group is same. You can modify the database entry and refine the occupancy values to get the best-fit model.

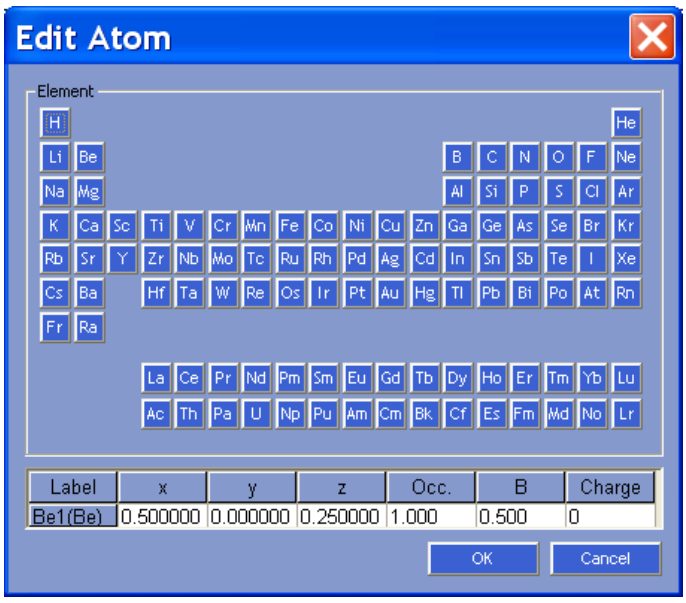
Example: The XRD pattern shown below is a mixture of Quartz (SiO_2) and Beryl ($Be_3Al_2Si_6O_{18}$) However for a synthetic Beryl prepared in lab may have additional elements (different composition, the elemental composition can be determined by any elemental; technique such as XRF, SEM, ICP etc). A possible combination of formula for Beryl to begin with based on elemental analysis can be (Be2.978 Li0.022) Al2 Si6 O18.

Assuming space group is same (this can be checked if all the peaks are present, their intensity and position may change). It is clear that the atomic site to make changes is one of Be. Next few screen

shots shows how to change the composition of Beryl from $Be_3Al_2Si_6O_{18}$ to Be2.978 Li0.022) Al2 Si6 O18 (caution, it has been determined by powder pattern that space group has not changed). Note the multiplicity of the site is 3 so we divide each occupancy by 3. This part will be disucces more in lecture.




Untitled - PDXL
File Edit View Analysis Graph Help
Data set: Sample10
Load...
Data Process
Identification
Auto Search
Load Card Info.
Indexing
WPPF
Structure Determination
Quantitative Analysis
RIR Method
External Standard Method
Internal Standard Method
Standard Addition Method
Application
Crystallite Size and Strain
Lattice Constants Refinement
Crystallinity
Stress
Create Report...
Save Project...
Intensity (cps)
2-theta (deg)
Quartz, syn
Beryl, syn
Unknown
Export...
Lattice constants
Space group: 192 : P6/mcc
Element list
Density peak list
Read CIF...
Import Z-matrix...
Add Atom...
Delete Atoms/Molecules
Edit Atom/Molecule...
Constraint List...
Select Items...
Set
Convert to Z-matrix
General phase information
Crystal structure information
Peak list
Phase information
Data set management
Search results
Candidate phase
Detailed phase information
Clear List
Print...
Ready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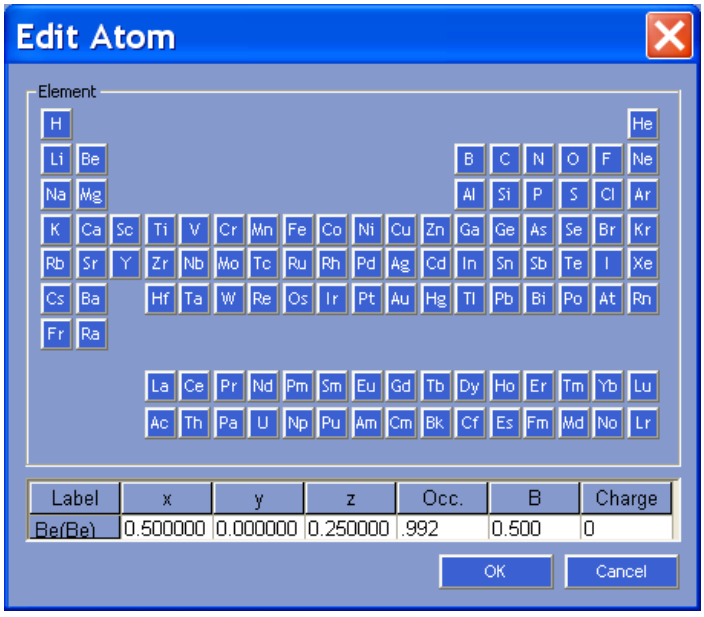
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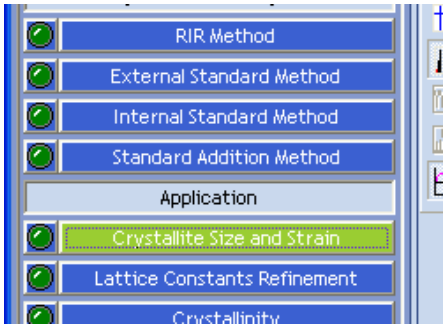
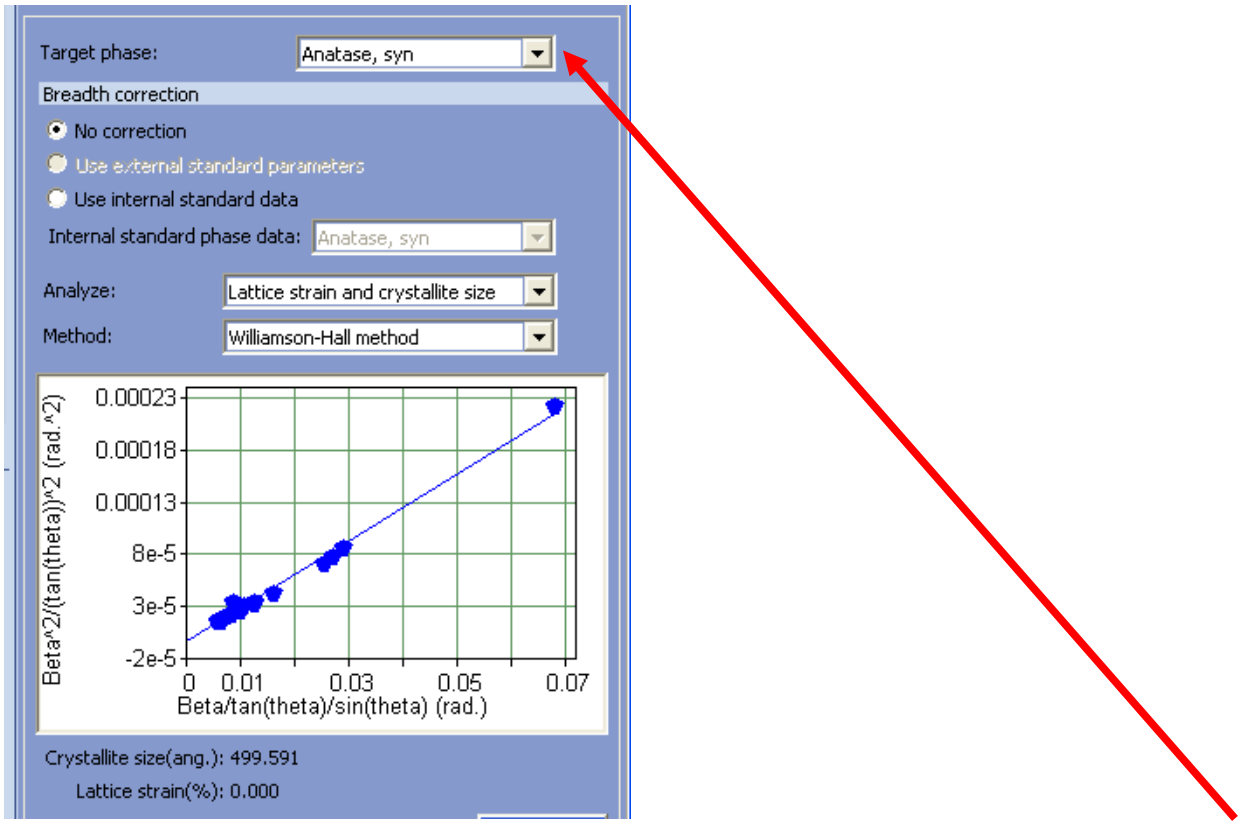
XII. Crystallite Size Module:

1. Click on the “Crystallite Size” button on the flow bar (left side).



2. A window will appear on the right hand side with a graph showing the individual points connected with a line. The average of all the points is the average crystallite size as calculated by the Willamson-Hall method



3. If more than one phase was identified (and “Set”), the crystallite size will be listed by phases. To see the crystallite size for other phases, click on the “Target phase:” drop down menu at the top.