XRD DIFFRAC.TOPAS: Quick Start Guide

V372 | December 2017

Introduction

This guide is a quick introduction to the DIFFRAC.TOPAS software and how to use the program for profile fitting and Reitveld Refinements. For detailed information and an in-depth guide refer to DIFFRAC.TOPAS User Guide & Technical Reference available in hard copy in the XRD lab or online in Cumulus.

DIFFRAC.TOPAS is not a phase identification software. If you do not know the minerals present, first identify the phases using DIFFRAC.EVA and then use DIFFRAC.TOPAS to quantify those minerals.

Part 1: Opening a Diffractogram in DIFFRAC.TOPAS

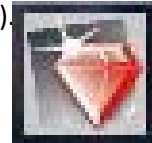Select the DIFFRAC.TOPAS icon on the desktop to open the software (*Figure 1*).

Figure 1. DIFFRAC .TOPAS icon

To open a diffractogram for evaluation:

1. Click **File** > **Load Scan File**
2. Select the Scan you want to open.
3. The scan is now loaded into the main screen.

To open a pre-existing TOPAS File:
1. Open the DIFFRAC.TOPAS software
2. Click **File** > **Load Project File**
3. Select the appropriate .pro file

Getting Acquainted

The main screen has the following areas (Figure 2):

1. Menu Bar – Load and save scans here
2. Tool Bar – identify peak locations, show calculated curve, set axis'
3. Parameter Window – scan trees, select features to refine
4. Fit Window – run refinement
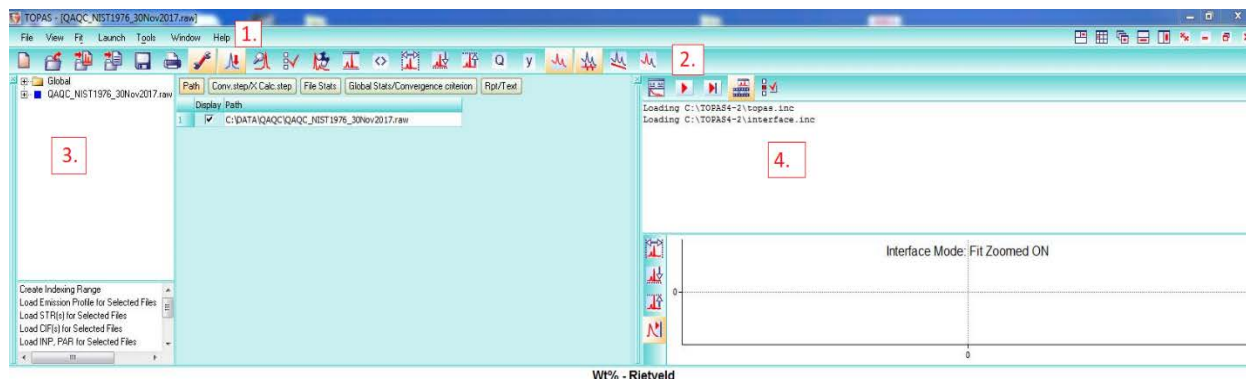5. Main Work Area – work with scan, identify peaks

Figure 2. Main window of TOPAS with Scan loaded. 1. Menu Bar 2. Toolbar 3. Parameter Window 4. Fit Window 5. Main Work Area

When a scan is loaded in it appears in the Parameter Window as the title of the scan (Figure 3). 'Global' is always present.

In DIFFRAC.TOPAS a scan is referred to as a 'Range'. Multiple scans can be loaded and refined simultaneously.
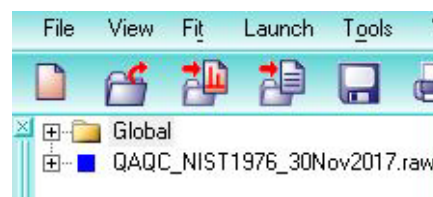

Figure 3. Parameter window after scan is loaded

Part 2: Identify Peaks

Peaks can be defined either automatically or manually inserted.

Automatically Insert Peaks

1. On the Toolbar select the 'automatically insert peak' icon (Figure 4)

2. A 'Peak Search' window will appear (Figure 5). Here adjust the peak width, noise threshold, and K-Alpha 2 removal. Each vertical line is a detected peak and the scan updates as the parameters change, increasing or decreasing the amount of detected peaks. When satisfied with the peak selection select 'Add Peaks'.

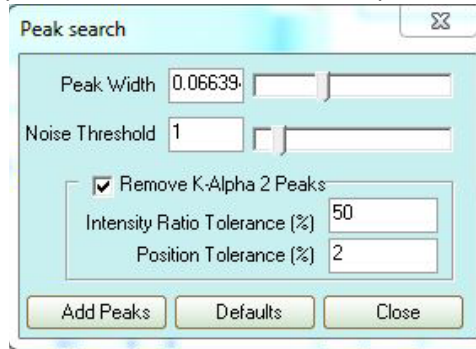Figure 4. Automatic-ally insert peak icon

Figure 5. 'Peak Search' Window

3. The peaks now appear in the Parameter window within the range (scan) under 'Peaks Phase' (Figure 6). To delete any peaks right-click on the peak and select 'delete nearest peak'.
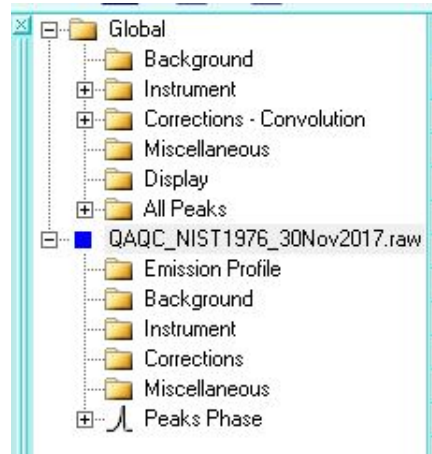
Manually Insert Peaks

Figure 6. Parameter Window featuring Peak Phase at the end of selected range

1. Select the magnifying glass icon located on the toolbar (Figure 7).

Figure 7. Peak details icon

2. The 'Peak Details' window pops up (Figure 8). The Peak Details window controls the displayed peak label information. The left column has 5 different fit models: 'FP', Fundamental Parameters, is the only model that uses instrument parameters; the other models are purely mathematical fits assuming nothing about the instrument. The middle column changes what information is displayed: peak position, area, crystal size, and strain. The right column determines how the information is displayed, ie a numerical value, a fixed or refined text label, measurement error. Set the peak details parameters.

3. With the mouse click on the center of each peak you want to identify directly in the scan window. The peak details window can be pulled up again to change the peak values.
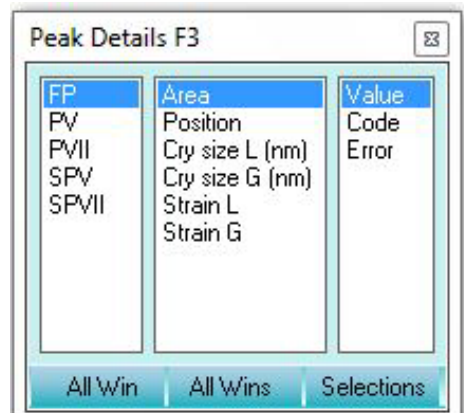

Figure 8. Peak Details Window

Part 3: Set Refinement Parameters

Refinement parameters are set in the 'Parameter Window' and changed in the data grid. In the Parameter Window there are two parameter definitons: **Global** and **Range**. Global parameters apply to all scans in the parameter window. Range parameters will only apply to that specific scan.

To select a parameter, check the box in the 'Use' column in the parameter row (Figure 9). A parameter can then either be **fixed** or **refined**. Switch between 'fix' and 'refine' under the 'Code' column. Click inside the box next to your parameter. Select the drop down menu and choose between 'fix' or 'refine'. Note that a fixed parameter remains black and a refined parameter turns red. A fixed parameter can also be denoted with a '!' and a refined parameter with an '@'
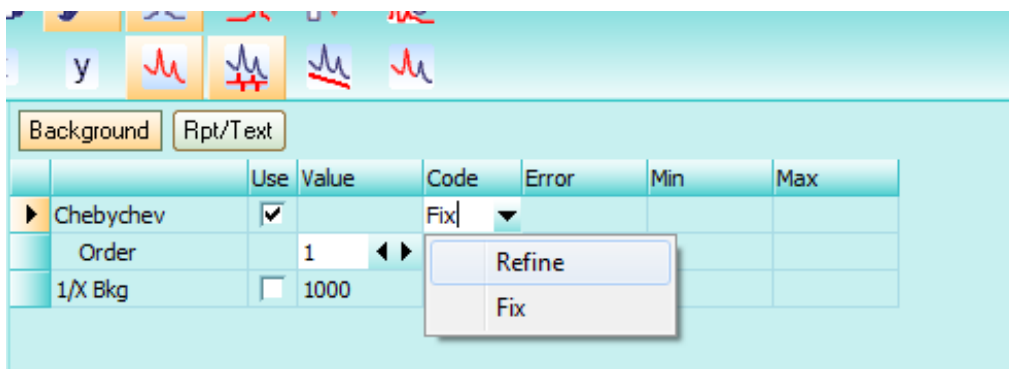

Figure 9. The Chebychev polynomial selected to be used and toggled between fix and refine.

Emission Profile: Wavelengths of K-Alpha 1 and K-Alpha 2 for a copper tube. This value is constant.

Background:

>*Chebychev*: Chebychev polynomial can be set to any order but do not increase the order too much or peaks may be modeled as background.

>*1/X Bkg*: aims to reduce air scatter at low angles.

Instrument: The refinement can account for instrument settings. The settings loaded in are accurate for the system used on board. It is your decision to include it or not include it in your refinement. Instructions on how to load are below in 'Load instrument Parameters'

Corrections:

>*Peak Shift*: Zero Error refers to the starting position of the goniometer. It is only needed if there is doubt the Goniometer has an accurate starting location. Sample Displacement will correct for shift in peaks to the right or left.

>*Intensity:* LP factor is only used if a monochromator is in the system. **Uncheck LP factor the JR does not have a monochromator**

>*Sample Convolutions:*

>*Zero Error*: Corrects the zero position of 2-theta. Only necessary if there is thought to be a positioning error

>*Surface Roughness*: Can refine for intensity corrections due to surface roughness based on one of two equations

Miscellaneous: Conv. Steps refers to how many iterations the refinement should go to before converging on a solution.

Load Instrument Parameters

Correct for instrument sources of error and load the instrument parameter file for the D4 Endeavour.

1. Right click on 'Instrument' and select 'Load Instrument Details'.
2. Navigate to Local Disk C > TOPAS 4-2 > Instrument details > D4EndeavourParameters.par
3. Select the file and click 'open'

Part 4: Run Refinement

When the parameters are set run the refinement.

1. Go to the Toolbar inside the 'Refinement window and select the 'play' button (Figure 10). In the window you will see a graph showing the refinement converge on a solution.
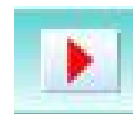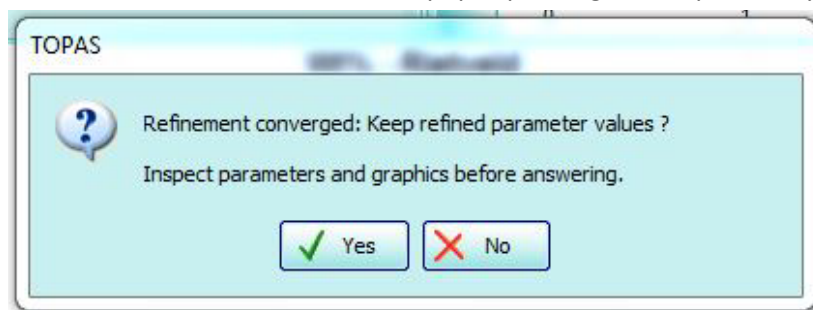2. When the refinement finishes a window pops up asking to accept or deny the

Figure 10. Play button

refinement (Figure 11). It is recommended to first examine the calculated curve.

3. Examine the measured scan versus the calculated scan. The blue line is the measured values and red line is the calculated result. Ideally the red line lies over the blue line with the peak position in the middle (Figure 12).
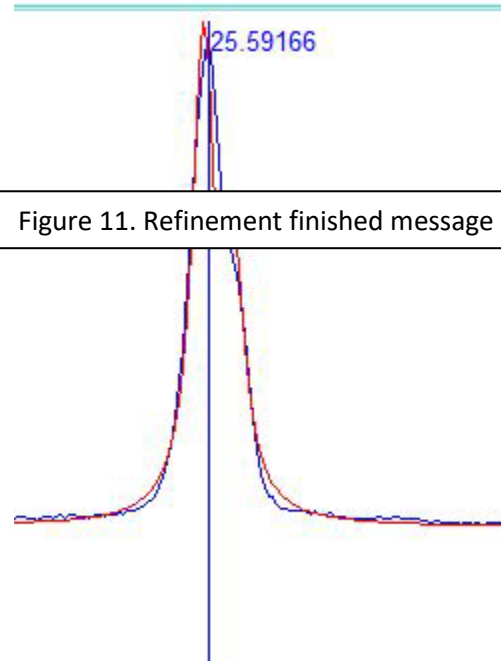

Figure 11. Refinement finished message

Figure 12. Example of calculated curve over measured curve

4. If the calculated line looks good select 'Yes' in the window to accept the refinement. If the calculated line does not look good select 'No' to reject the refinement and then go back to the parameter window and tweak the parameters. When a refinement is rejected the calculated curve will remain there until another refinement is run.

Part 5: Find Area Under Curve

1. In the parameter window go to your scan tree and select 'Peaks Phase' (Figure 13).

	Type	Use	Position	Area	Cry size L (n	Cry size G (n	Strain L	Strain G
1	FP	✔	6.013978	28641.63	1.0	200.0	0.1	0.1
2	FP	✔	12.50425	524.3353	58.2	200.0	0.1	0.1
3	FP	✔	18.81645	49.67658	219.4	200.0	0.1	0.1
4	FP	✔	19.89417	386.3037	22.6	200.0	0.1	0.1
5	FP	✔	20.86609	781.6675	111.4	200.0	0.1	0.1
6	FP	✔	21.99411	161.7508	116.8	200.0	0.1	0.1
7	FP	✔	26.65009	4123.634	132.0	200.0	0.1	0.1
8	FP	✔	27.96959	1409.4	51.2	200.0	0.1	0.1
9	FP	✔	29.41835	782.8218	66.4	200.0	0.1	0.1

Figure 13. Peaks Phase Grid

2.   Under the 'Values' tab look at the 'Area' column. This lists the area under each peak in total counts.
3.   These values can be copied and pasted into a personal spreadsheet.

Part 6: Weight Percent Analysis

A structure file contains crystallite size and indice values for minerals. Add structure files of each mineral present for TOPAS to calculate the weight percentage and/or the crystallite size. If evaluating multiple scans and using the same minerals to quantify each this can be done in 'Global' tree. If doing one scan or using different minerals for each scan, add structures scan by scan.

One Scan
Add Structure Files
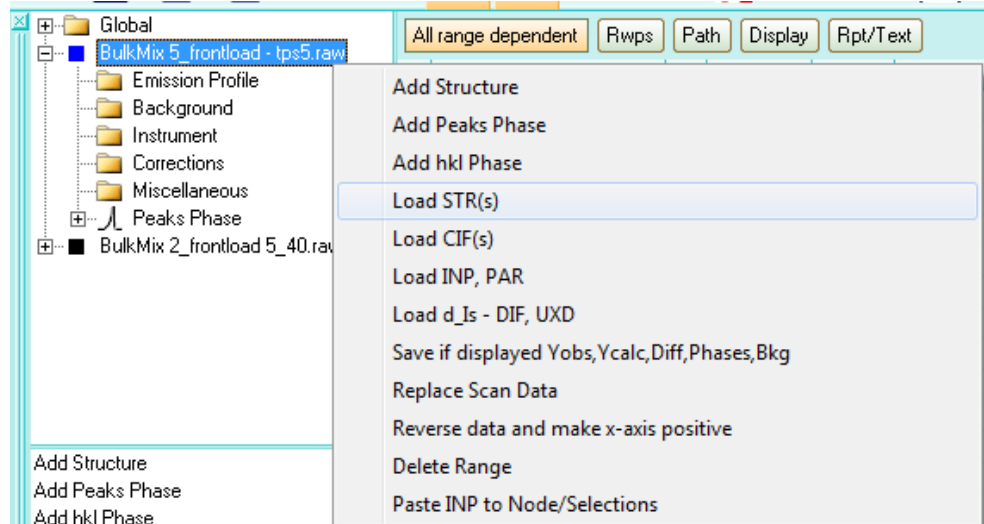1.   Right click on the desired scan and select 'Load STR(s)' (Figure 14)

Figure 14. Load STR(s) pathway

2.   Select the minerals from the pop up window (Figure 15). You can search through the mineral groups or start typing in the mineral and it will appear in the bar.
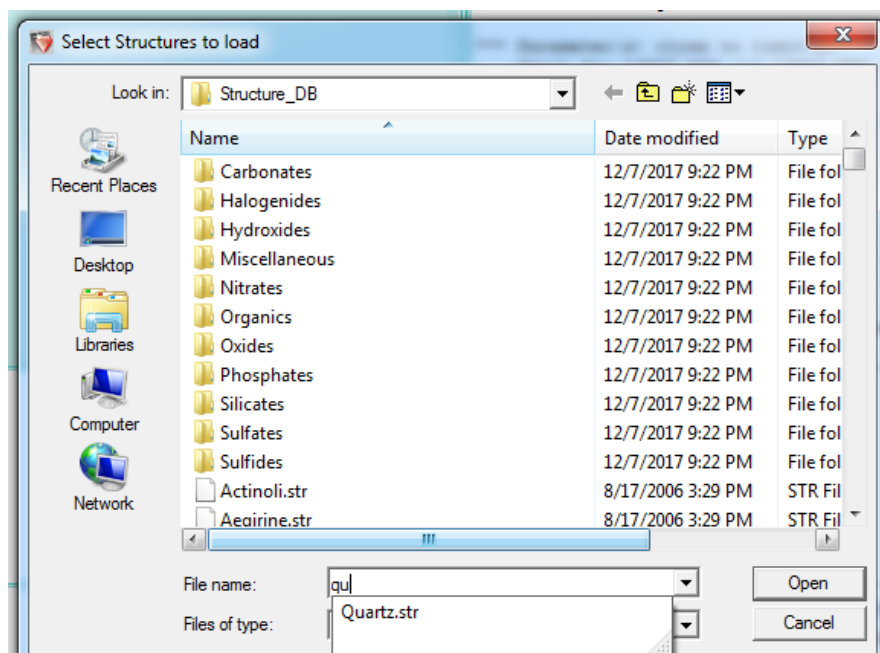
Figure 15. STR Library

Run Refinement

1. Go to the Fit Window and select the 'Play' button as done earlier.
2. When the Refinement finishes a message box shows up asking if the solution is accepted or denied. Look at the solution before saying yes or no.
3. The weight percent for each mineral shows up in the top right corner of the scan window. View the areas under each curve as noted in 'PART 5'. You will notice the area values change if run with/without structure files.
4. If you agree with the results, select 'accept'. If you do not agree deny the refinement and go back to your parameter window and re-evaluate your structures and parameters.

Multiple Files

Add Structures

1. In the Parameter Window select 'Global'. Then in the display window hit 'ctrl' and select your scans, highlighting them as seen below in Figure 16.
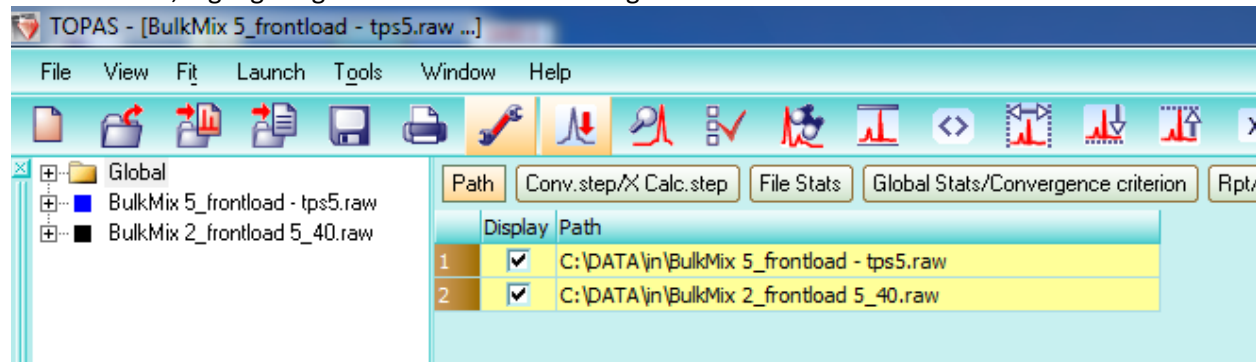

Figure 16. Add STR files to multiple scans at once

2. Back in the Parameter Window, right click on 'Global' and select 'Load STR(s) for Selected Files' (Figure 17).
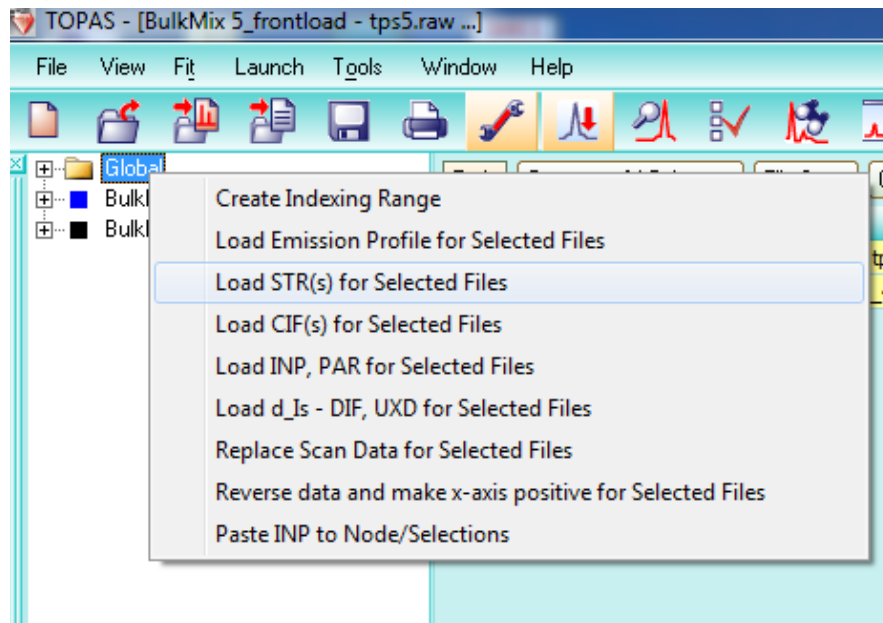

Figure 17. The 'Global' pathway to Load STR(s)

3. Select the minerals from the pop up window (Figure 18). You can search through the mineral groups or start typing in the mineral and it will appear in the bar
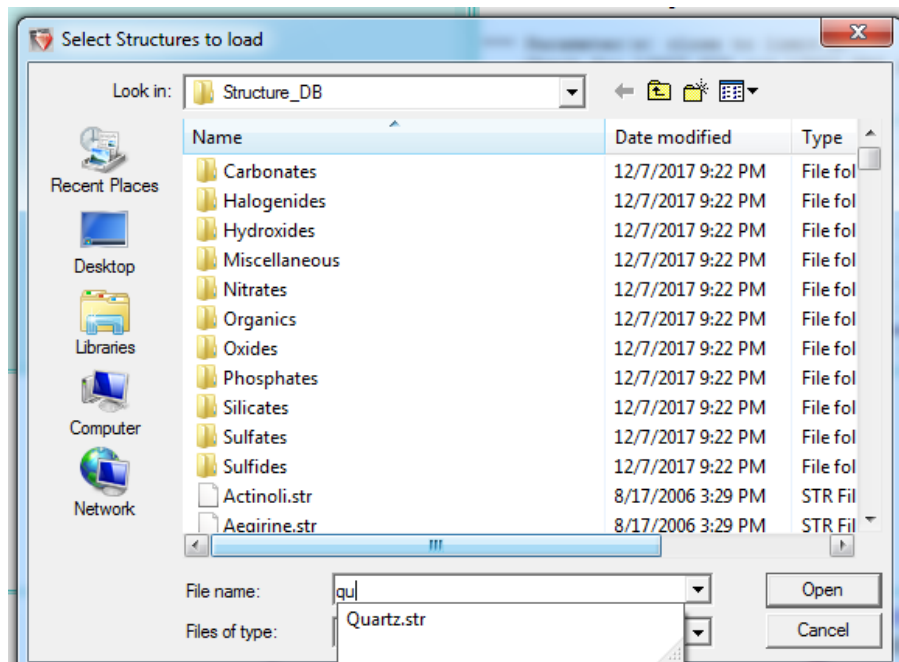

Figure 18. STR Library

4. Now when the scan is expanded in the parameter window a mineral structure is included at the bottom (Figure 19).
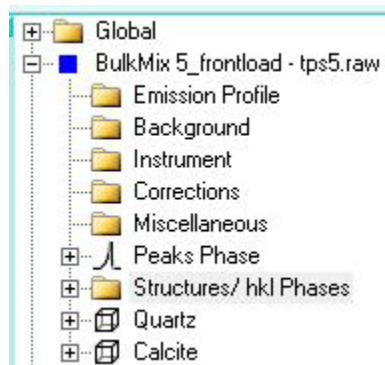

Figure 19. Structures present in parameter window under range tree

Run Refinement

TOPAS can run this refinement if multiple scans are loaded in, but there can only be one scan active at a time or the refinement will run the most recent scan and apply that data for all scans.

1. Go to the parameter window and look at all the scans loaded in.
2. The colored box next to the scan indicates that a scan is active. Clicking on the box alternates it between 'active' and 'inactive'. In Figure 20 there are two active scans, quartz is blue and Illite is black. In Figure 21 the white box next to Illite illustrates that this scan is now inactive.

Figure 20. Two Active Scans

Figure 21. One Active Scan and one inactive scan

3. One scan can be run at a time so click on the colored boxes next to all but one scan. After this there should only be one scan seen on the graph.
4. Go to the refinement window and select 'Play'.
5. The message will appear stating the refinement converged and asking to accept or deny the refinement. Check the data before selecting an answer.
6. In the upper right corner, the weight percent of each mineral appears. Check the areas by following instructions listed in Part 5.

Part 7: Save and Export Image

Save a File

1. Select 'File' on the Menu Bar and go to 'Save project As…' (Figure 22)
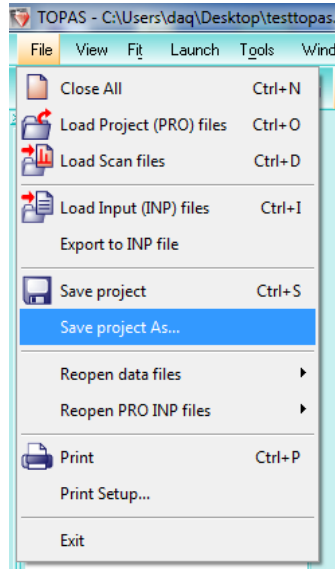2. Navigate to a place on the desktop on server and save the file.

Figure 22. Save Project Pathway

Export or Print Graph

To print or export the graph as image follow the steps below.

1. Select the printer icon on the toolbar

2. This brings up the 'Chart Options' Box. Select 'Print / Print Setup' (Figure 23)
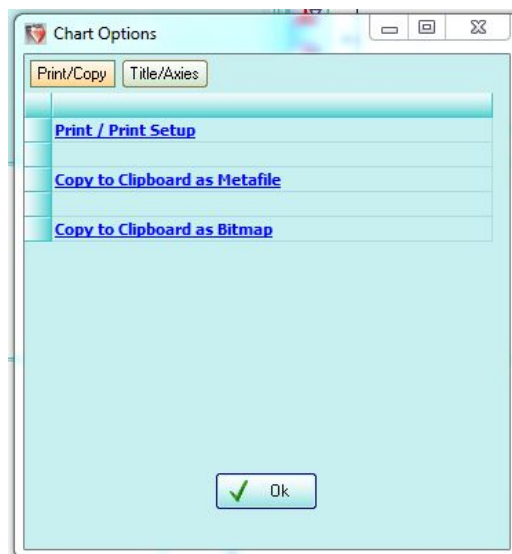

Figure 23. Print Setup Window

3. Select the dropdown menu to select a printer. Choose 'Microsoft XPS Document Writer' to get a digital image of the graph (Figure 24). Then select 'Print'.
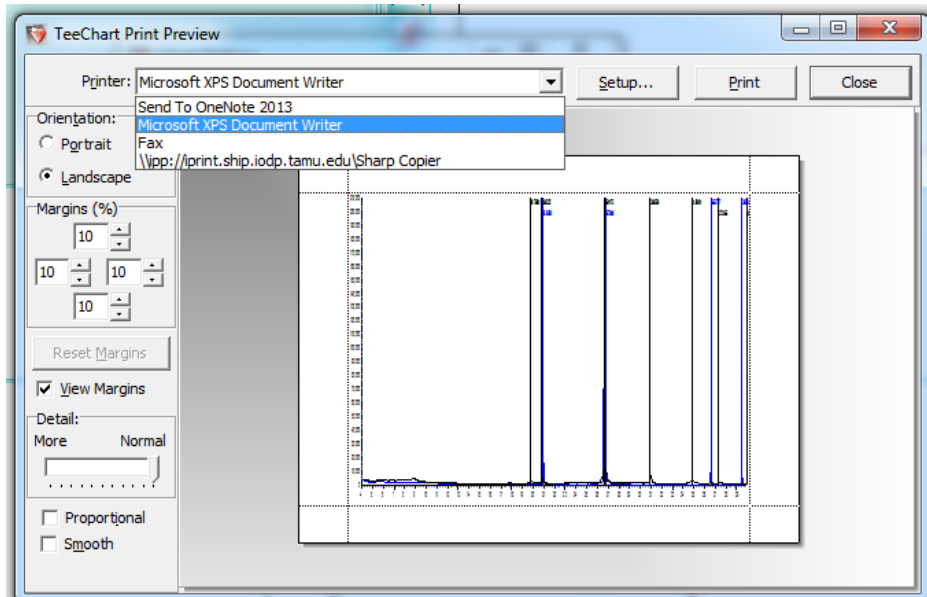

Figure 24. Print Window with Printer dropdown highlighted