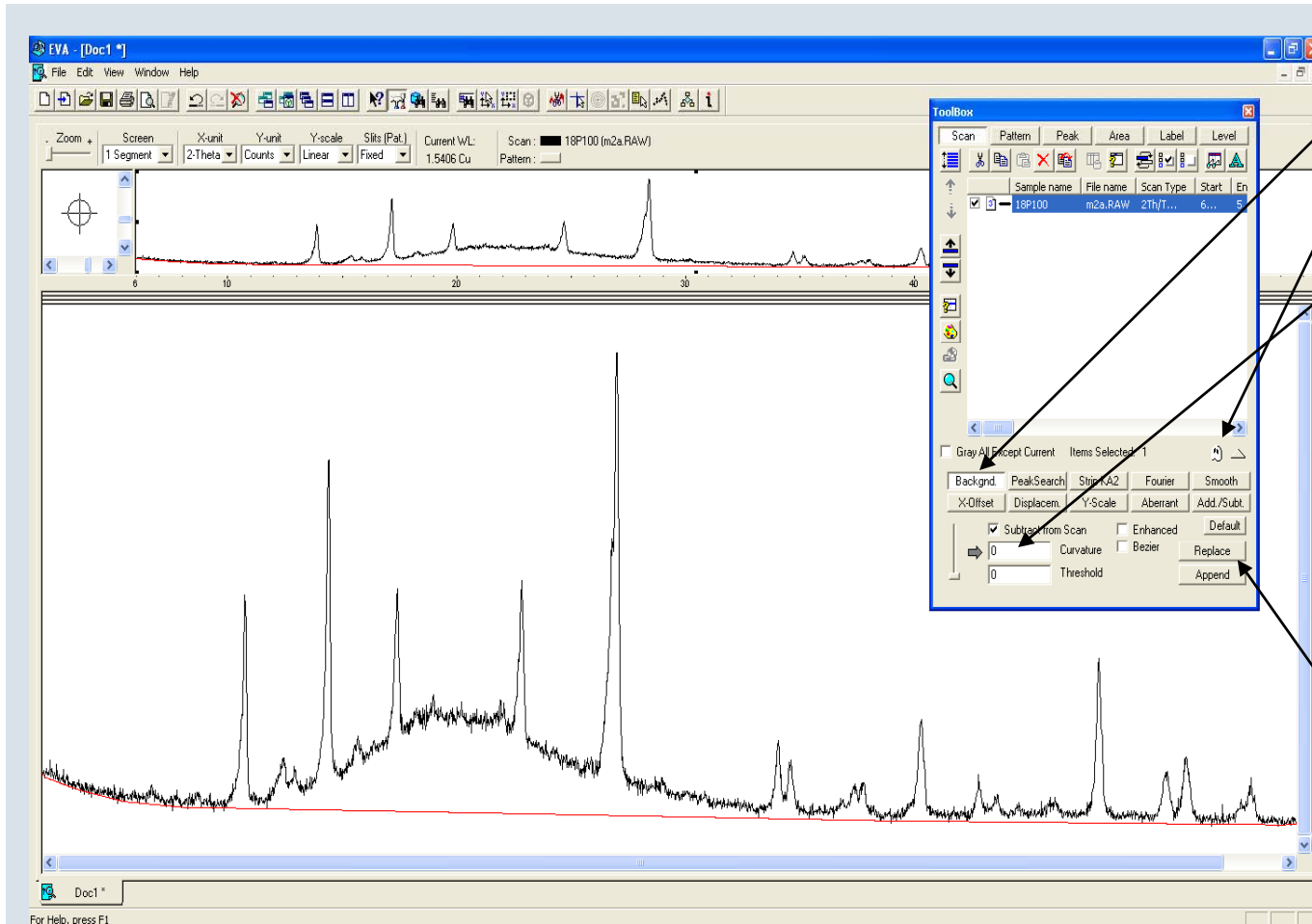




Percent Crystallinity EVA

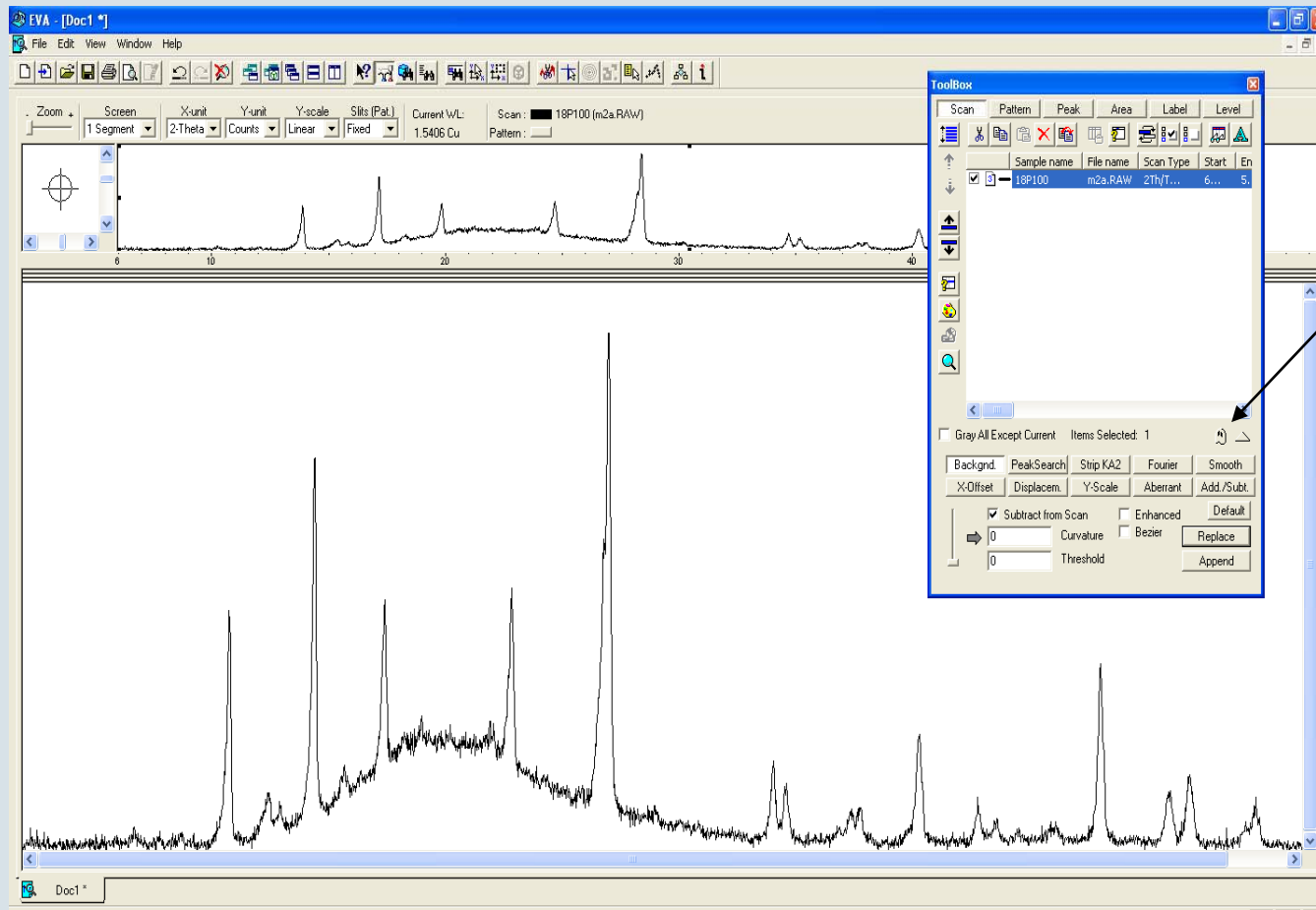
- Example scan file is from the Topas tutorial folder:
 - C: \Topas4\Tutorial\DOC\m2a.raw

Step 1



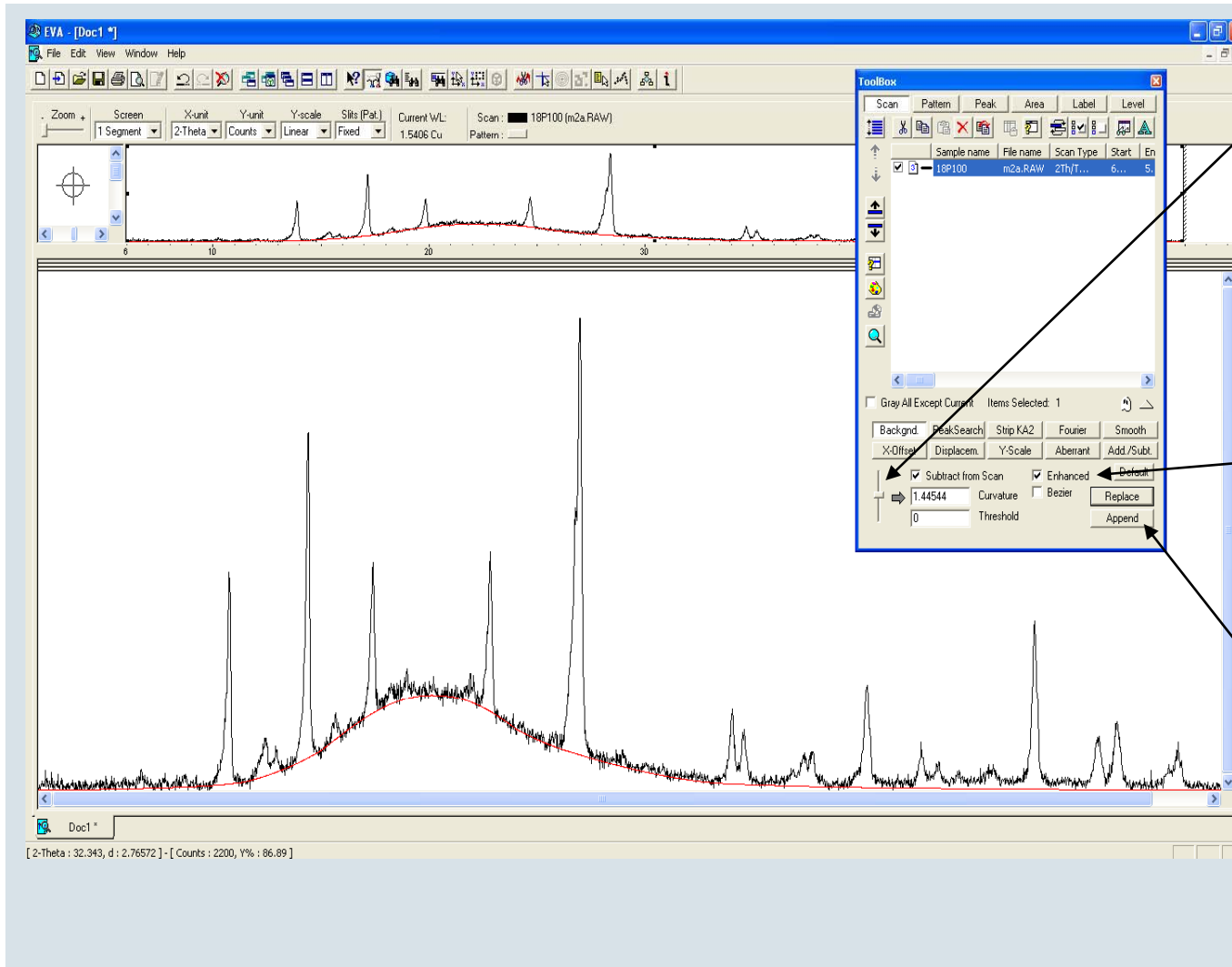
- Select the Background button.
- Activate the preview ghost.
- Set the Curvature value to 0.
- This background represents Air scatter, incoherent scatter, etc. and is not a part of the amorphous scatter.
- Click the "Replace" button to remove it.

Step 2



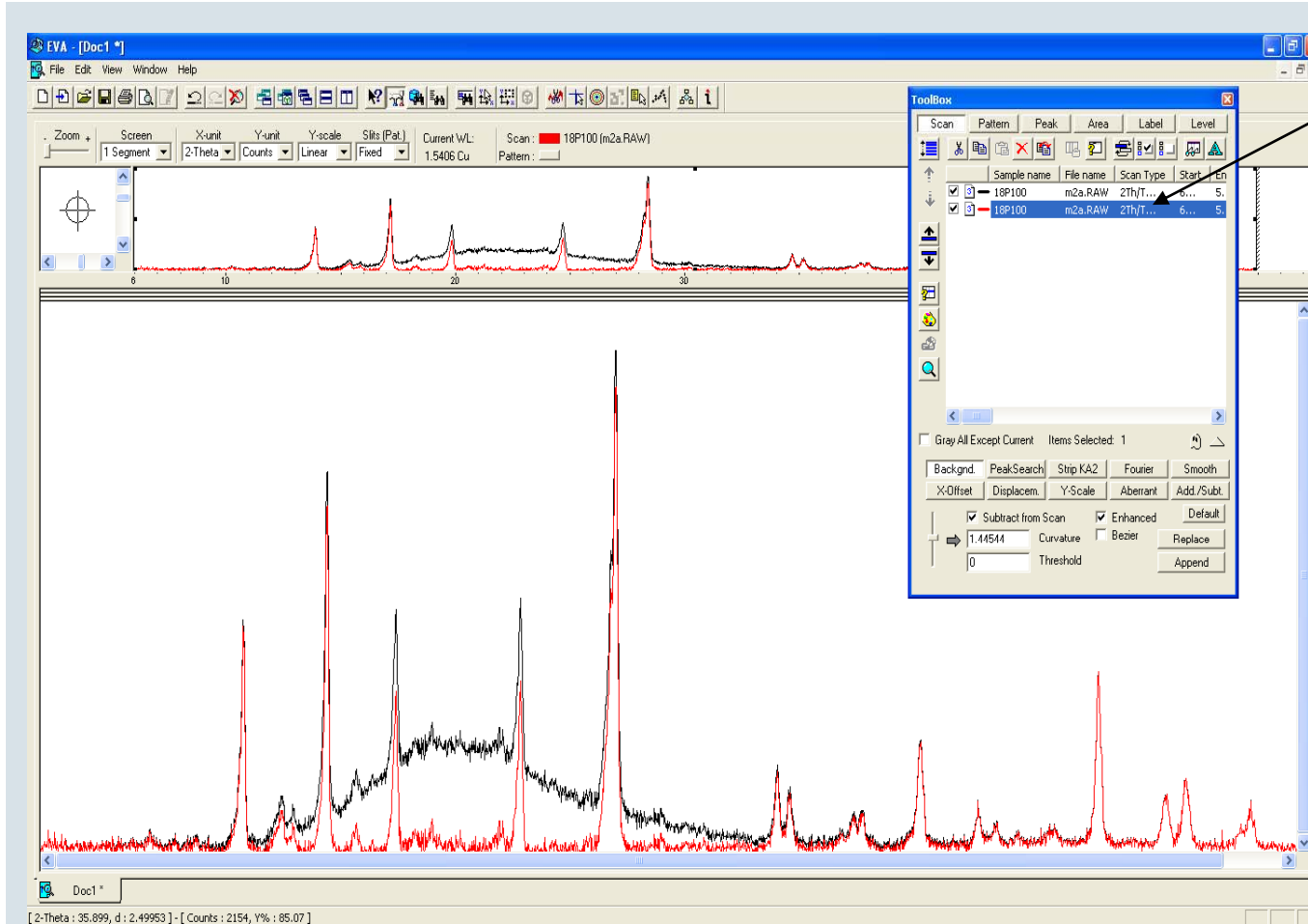
- What remains is scatter from crystalline and amorphous contributions only.
- With the background tool button selected, click the preview ghost again.

Step 3



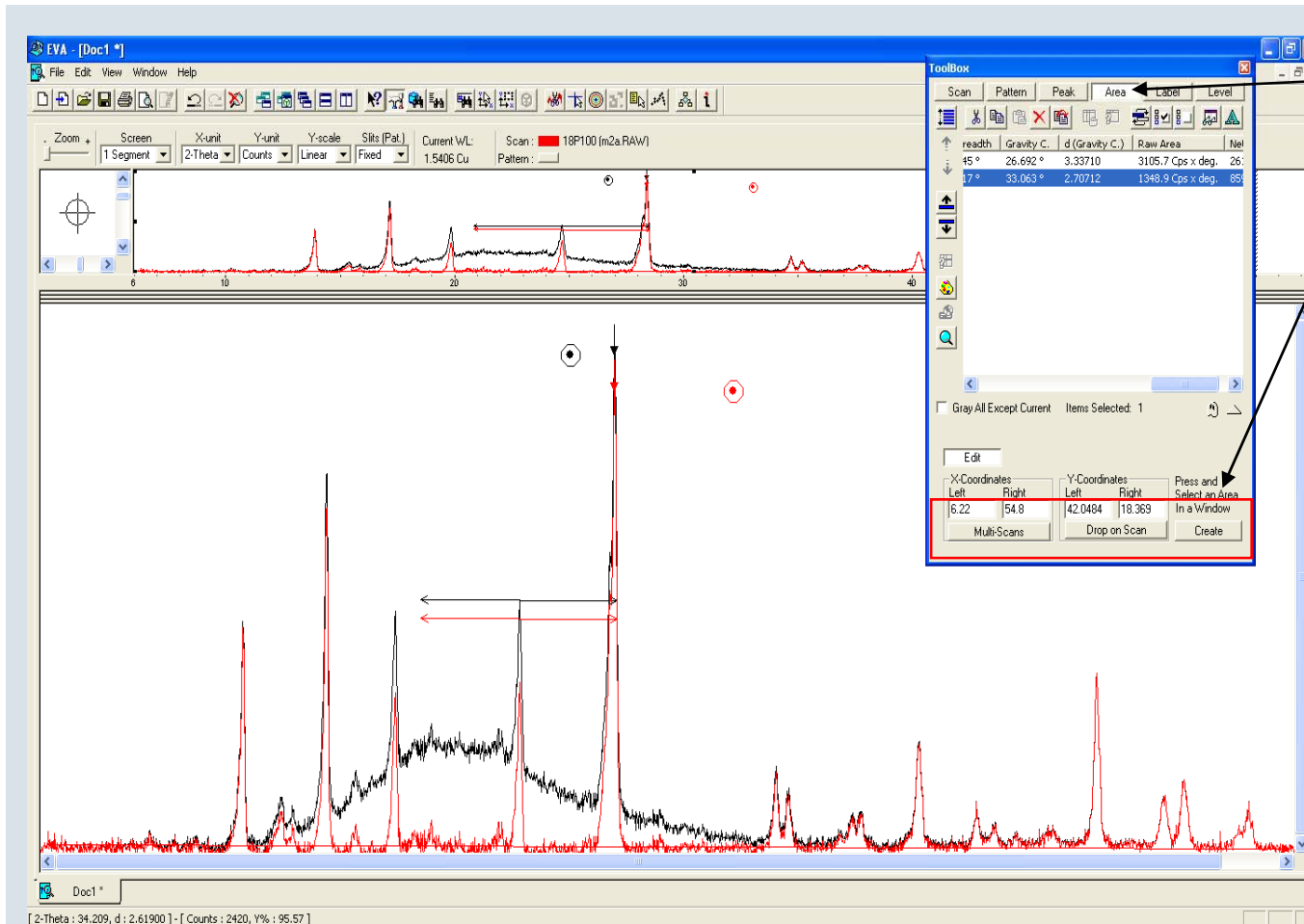
- Adjust the curvature value with the slider until suggested background appears to account for all of the diffuse scatter without cutting into the crystalline peaks.
- You may need to activate the Enhanced checkbox to achieve a good background model.
- When done, click the Append button and choose "Yes" to removing a background again.

Step 4



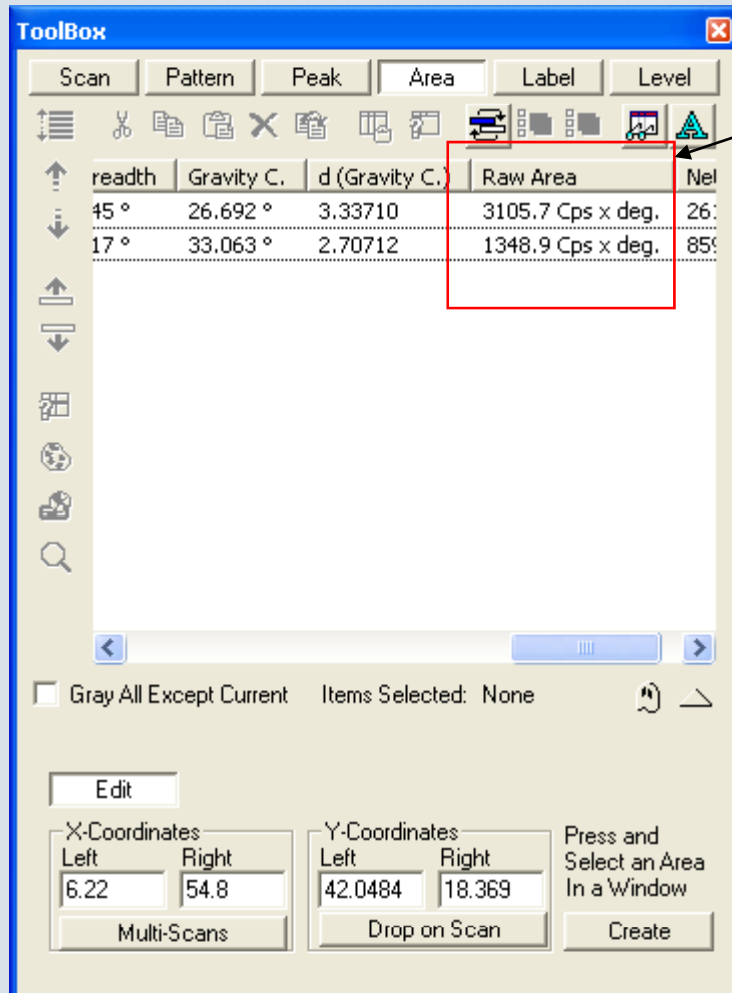
- This will add a range to the display that represents the crystalline only contribution to the scatter.

Step 5



- Now select the "Area" button.
- Click the 3 buttons, "MultiScans", "Drop on Scan" and "Create".
- With the cursor, click on the left-most portion of the scans and drag to the right-most portion and release. This will create an area region for both scans.

Step 6



- In the ToolBox window, scroll to the right until you see the column titled "Raw Area".
- These values represent integrated intensities of the amorphous + crystalline contribution and the crystalline-only contribution, respectively.
- Percent crystallinity can be calculated from this ratio:

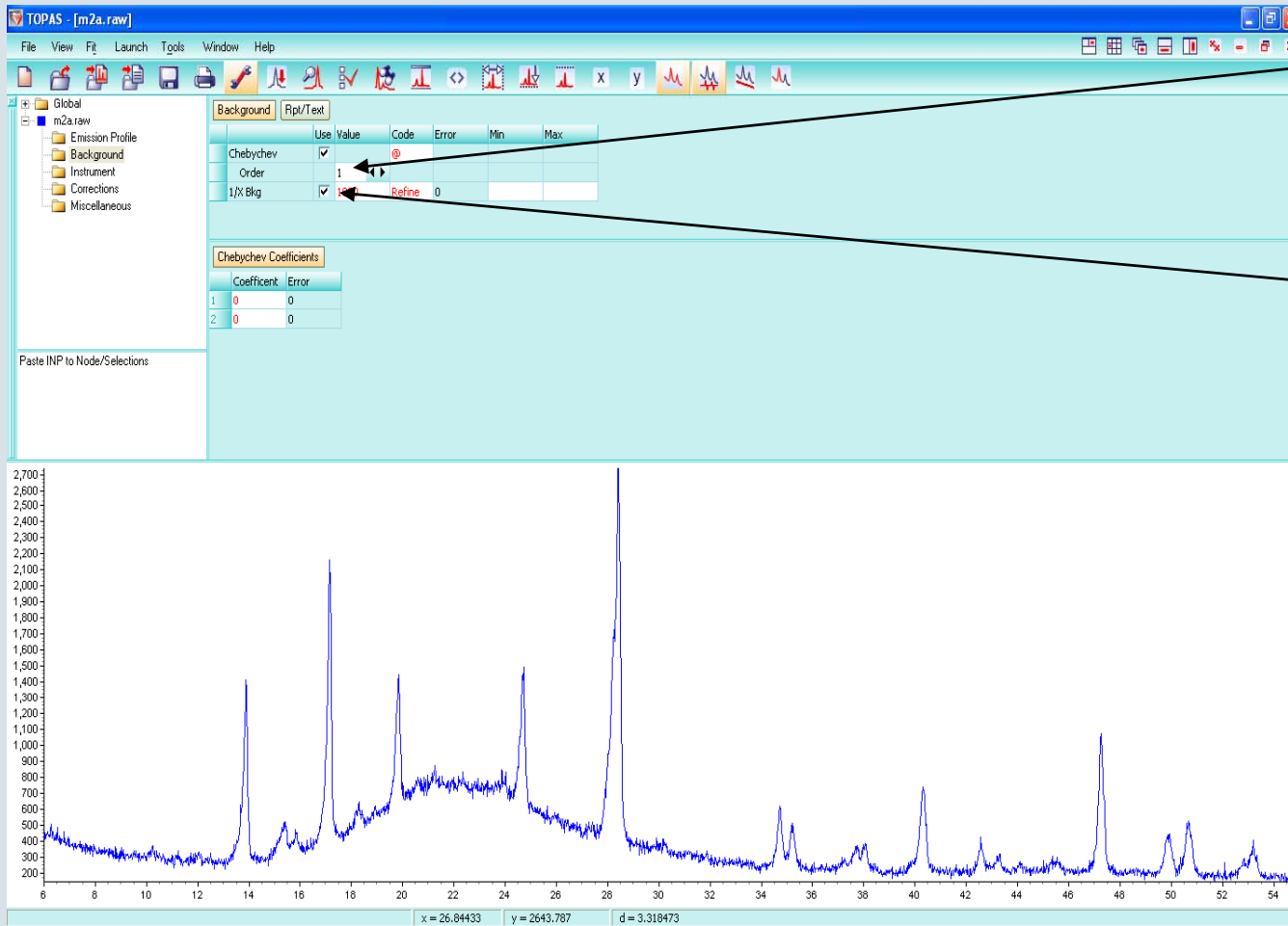
$$\begin{aligned} \% \text{crys} &= 1348.9 / 3105.7 \\ &= 43.4 \% \end{aligned}$$



Percent Crystallinity TOPAS

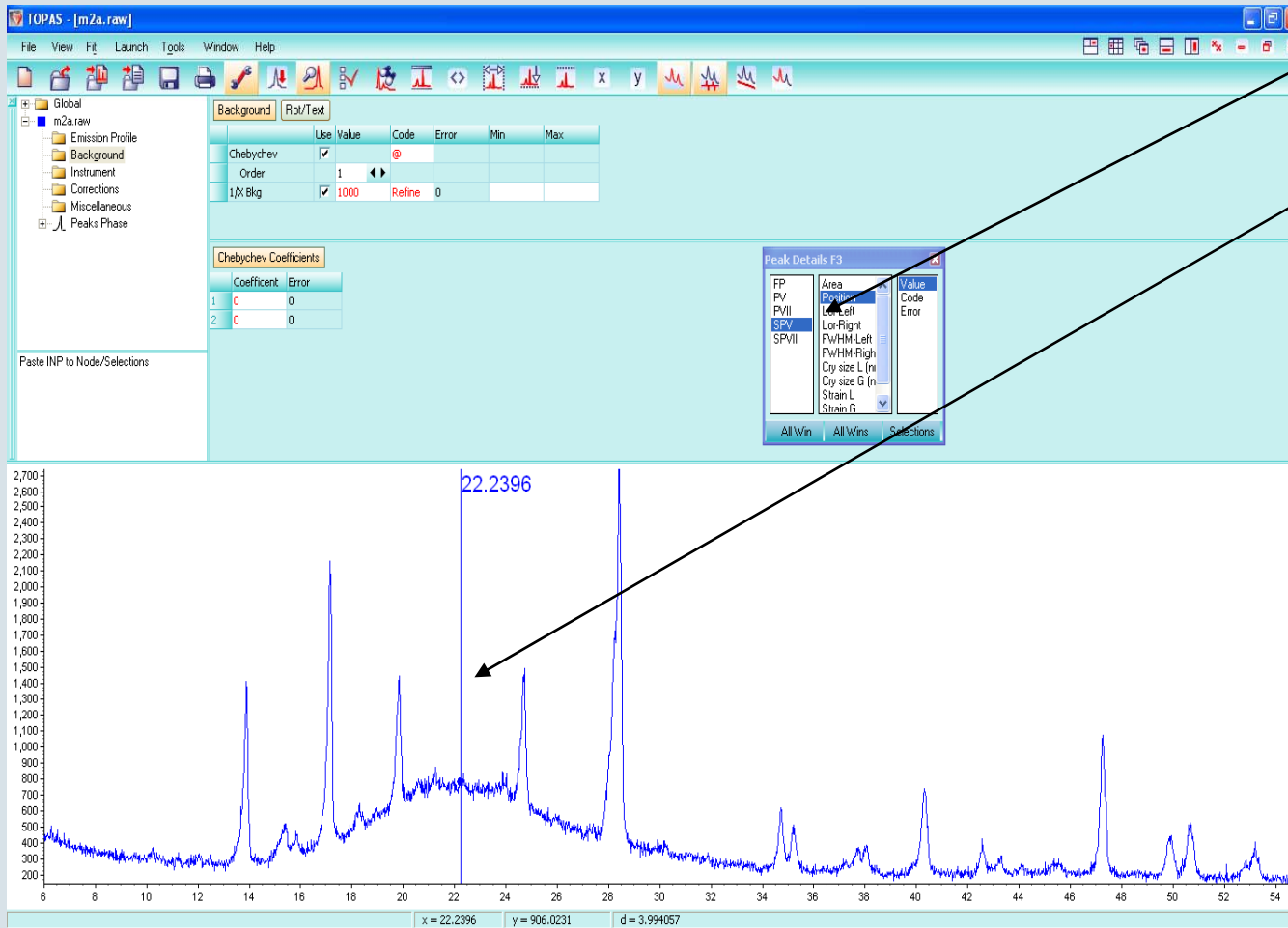
- Example scan file is from the Topas tutorial folder:
 - C: \Topas4\Tutorial\DOC\m2a.raw

Step 1



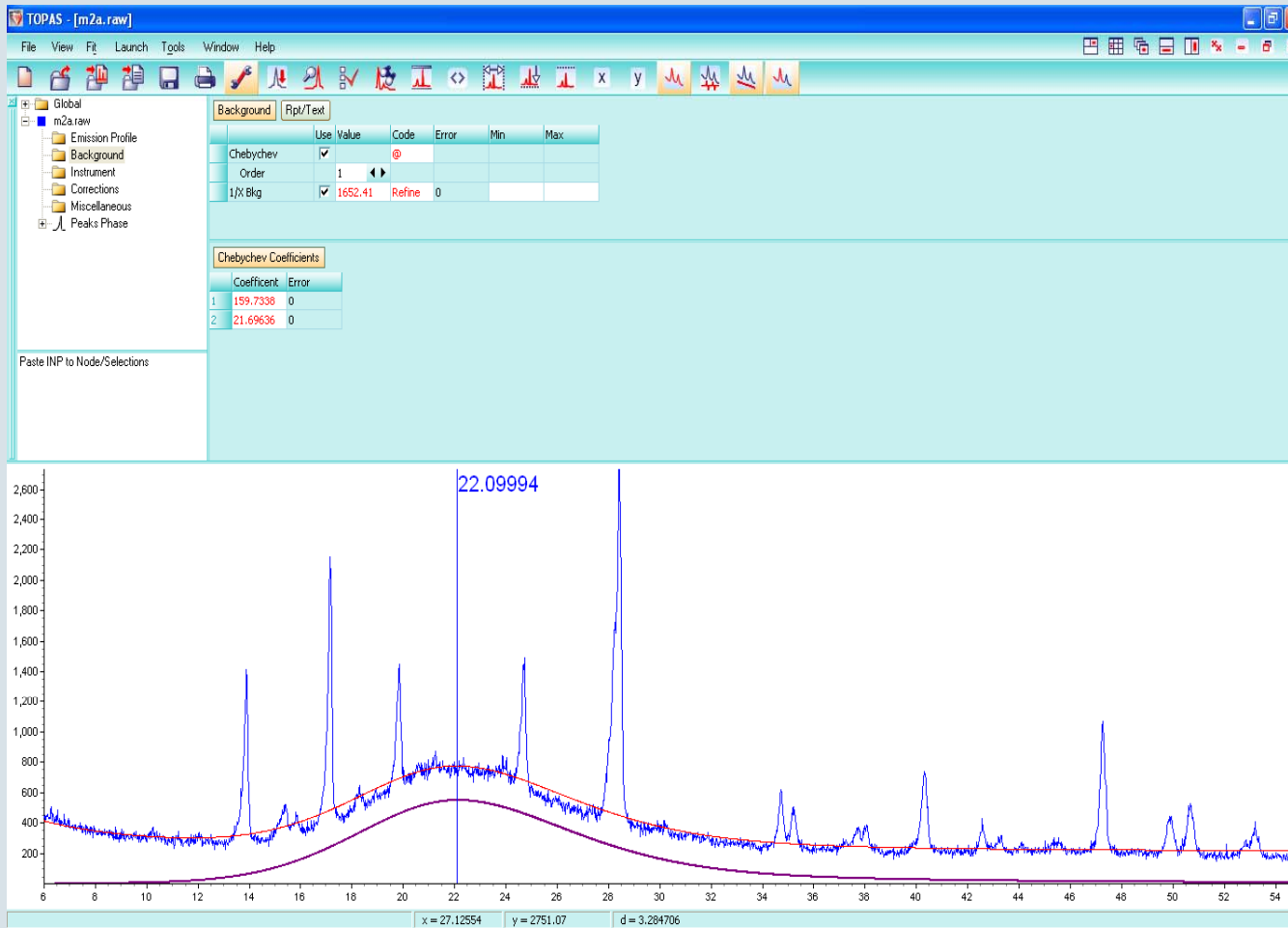
- In the parameters window, select an order 1 Chebyshev polynomial.
- Check the Use box next to the 1/X Bkg

Step 2



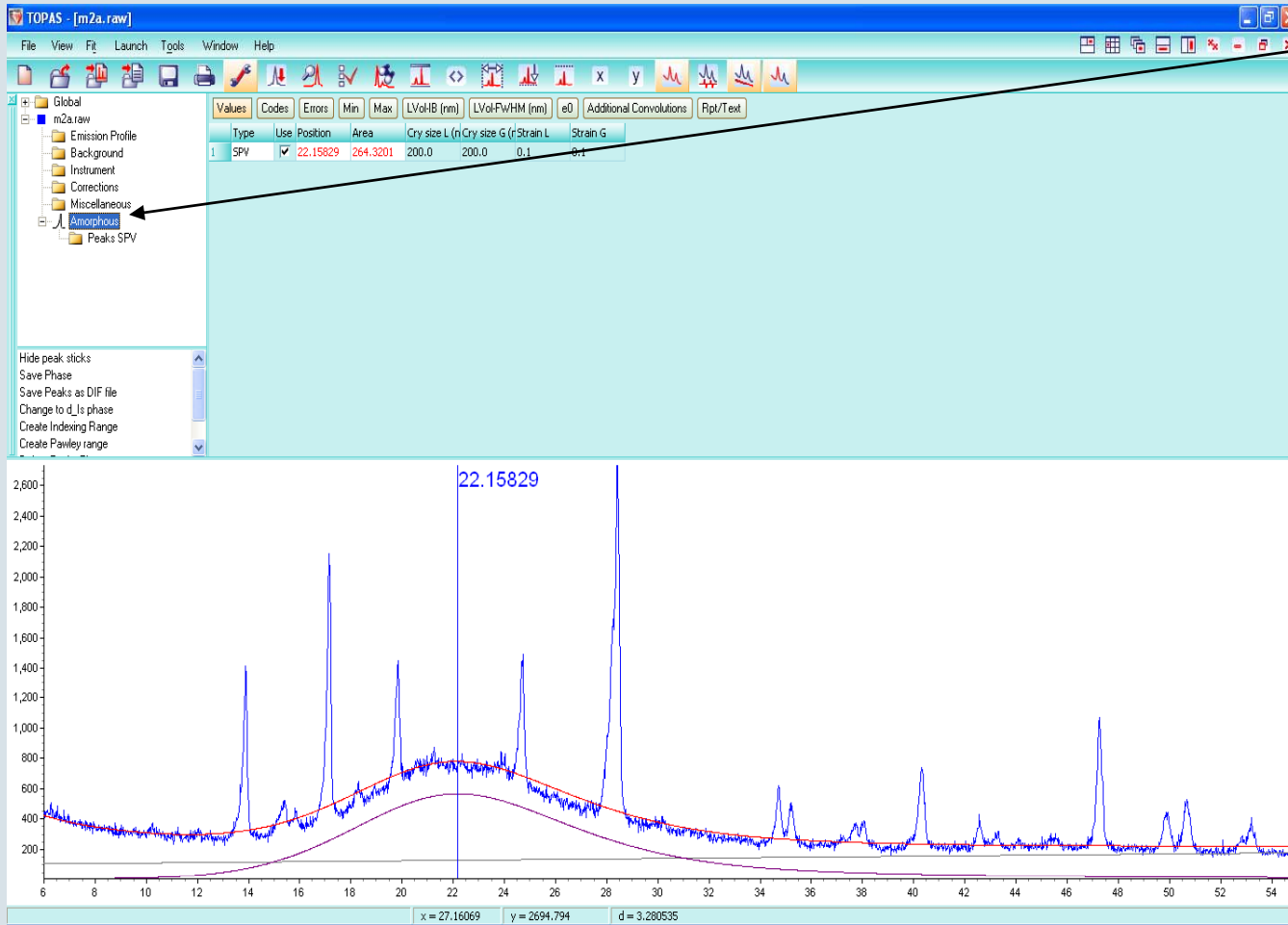
- Hit F3 and select the SPV peak type.
- Insert a peak at the approximate center position of the diffuse amorphous scatter.

Step 3



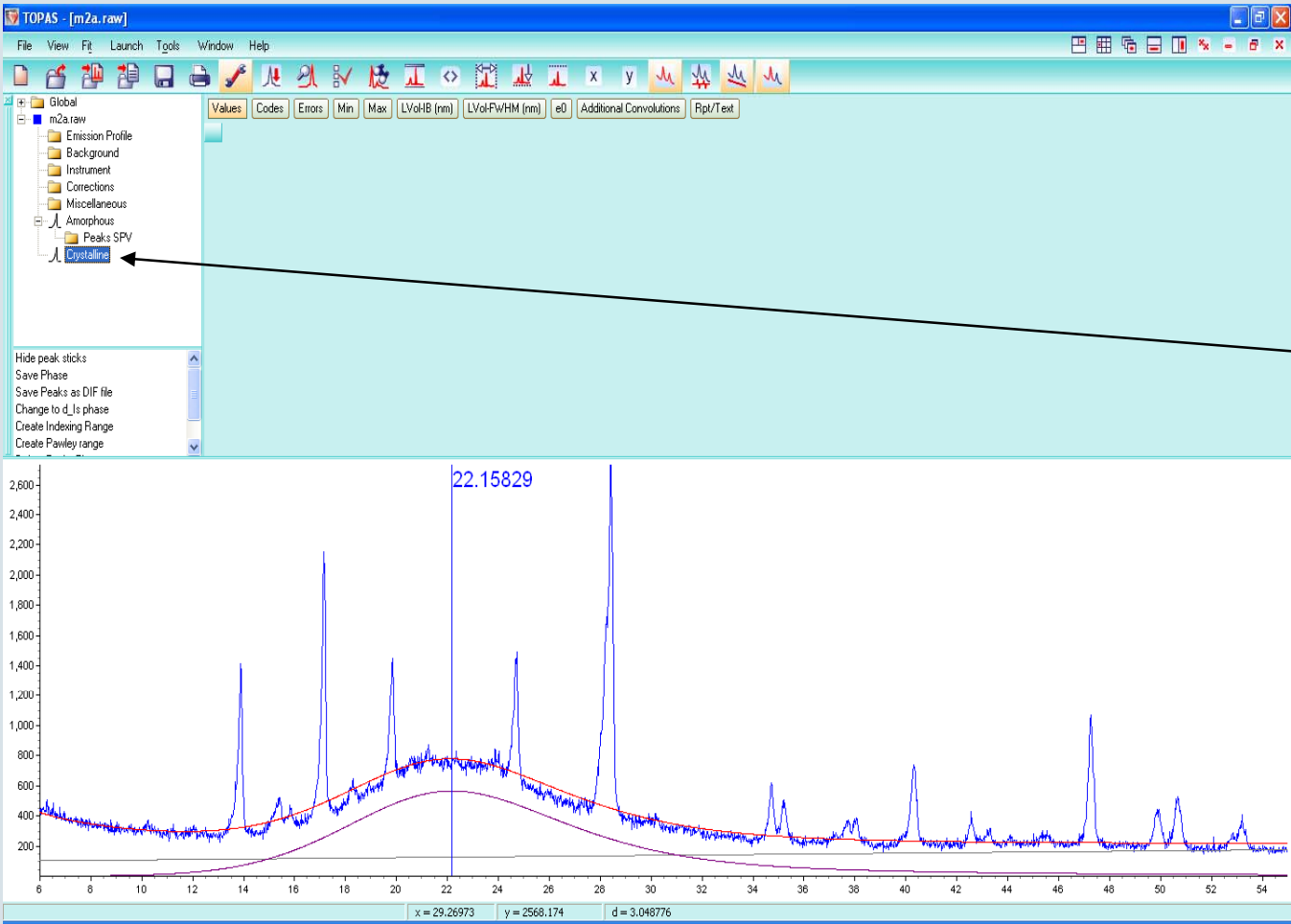
- Start a refinement by pressing the F6 button.
- The simulation should approximate the amorphous scattering only.

Step 4



- Rename this peaks phase "Amorphous" or something similar.

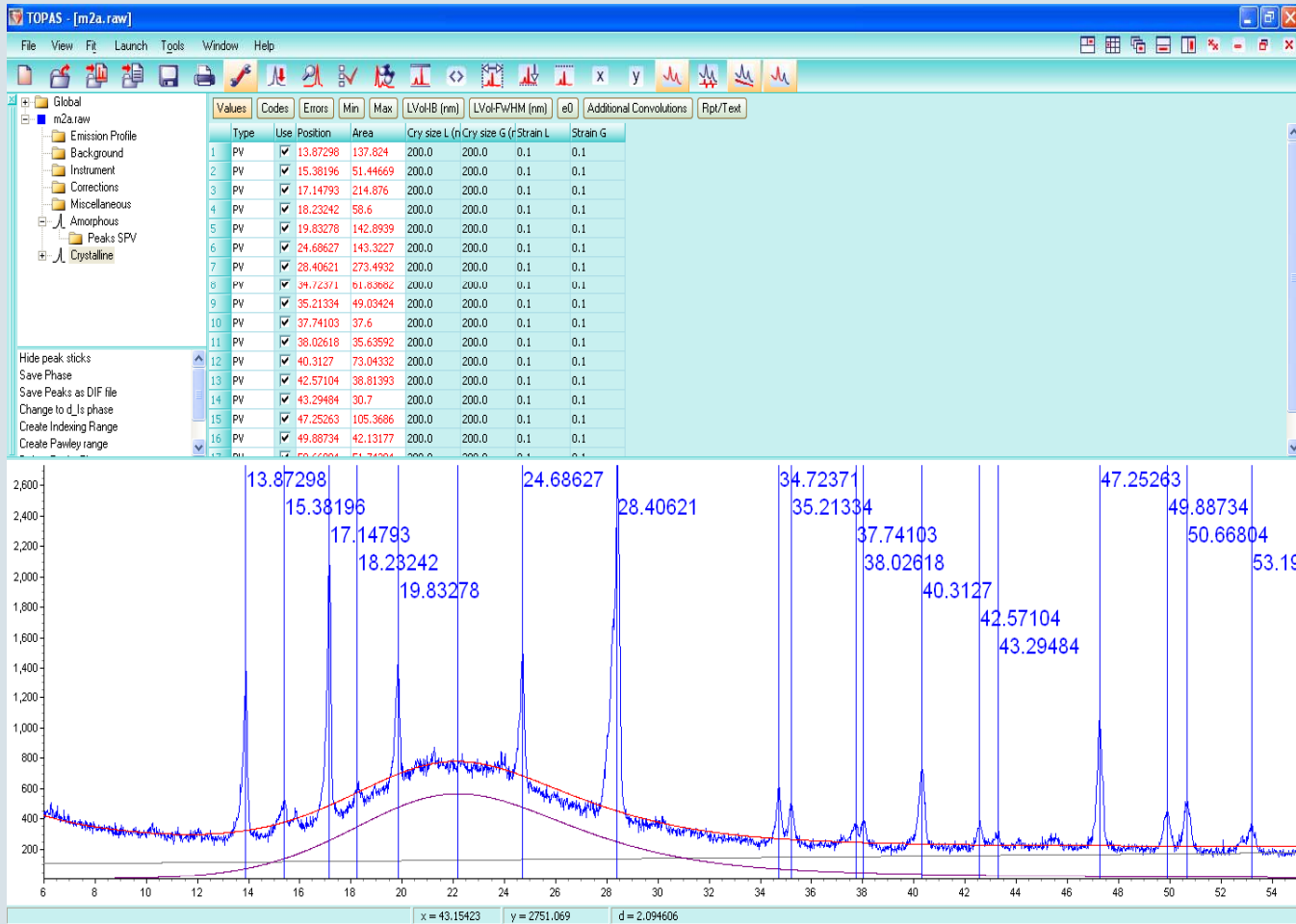
Step 5



The screenshot shows the TOPAS software interface. The left sidebar contains a tree view with folders for 'Global', 'm2a.raw', 'Emission Profile', 'Background', 'Instrument', 'Corrections', 'Miscellaneous', 'Amorphous', 'Peaks SPV', and 'Crystalline'. An arrow points from the 'Crystalline' folder to the 'Add peaks phase' button in the main window. The main window displays a plot of intensity versus 2θ, with a peak at 22.15829. The plot shows a blue line for the experimental data, a red line for the fit, and a purple line for the background. The x-axis ranges from 6 to 54, and the y-axis ranges from 0 to 2,600. The status bar at the bottom shows the coordinates x = 29.26973, y = 2568.174, and d = 3.048776.

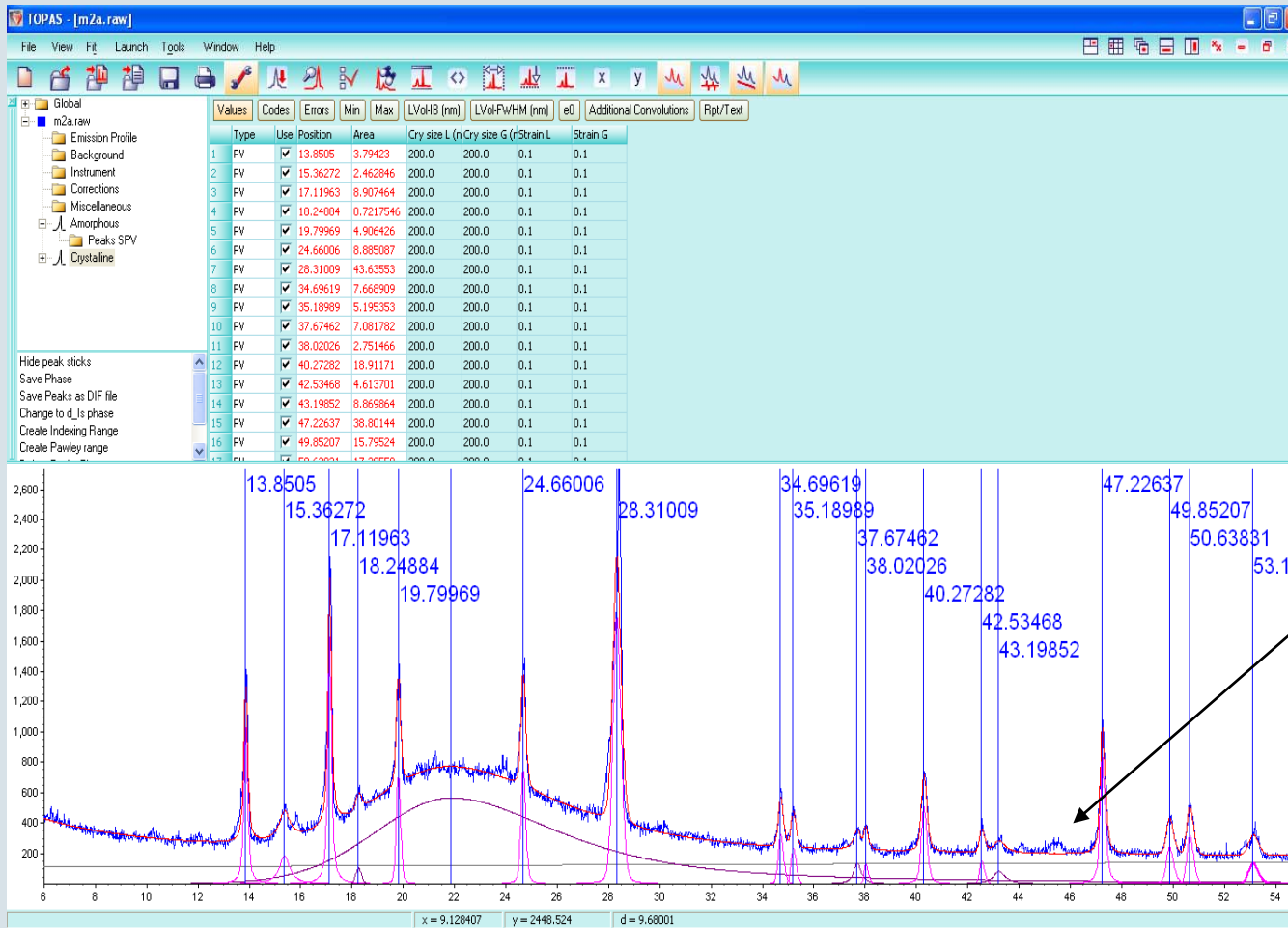
- Add another peaks phase by selecting the range and then choosing “Add peaks phase” in the window below.
- Name it “Crystalline” or something similar.

Step 6



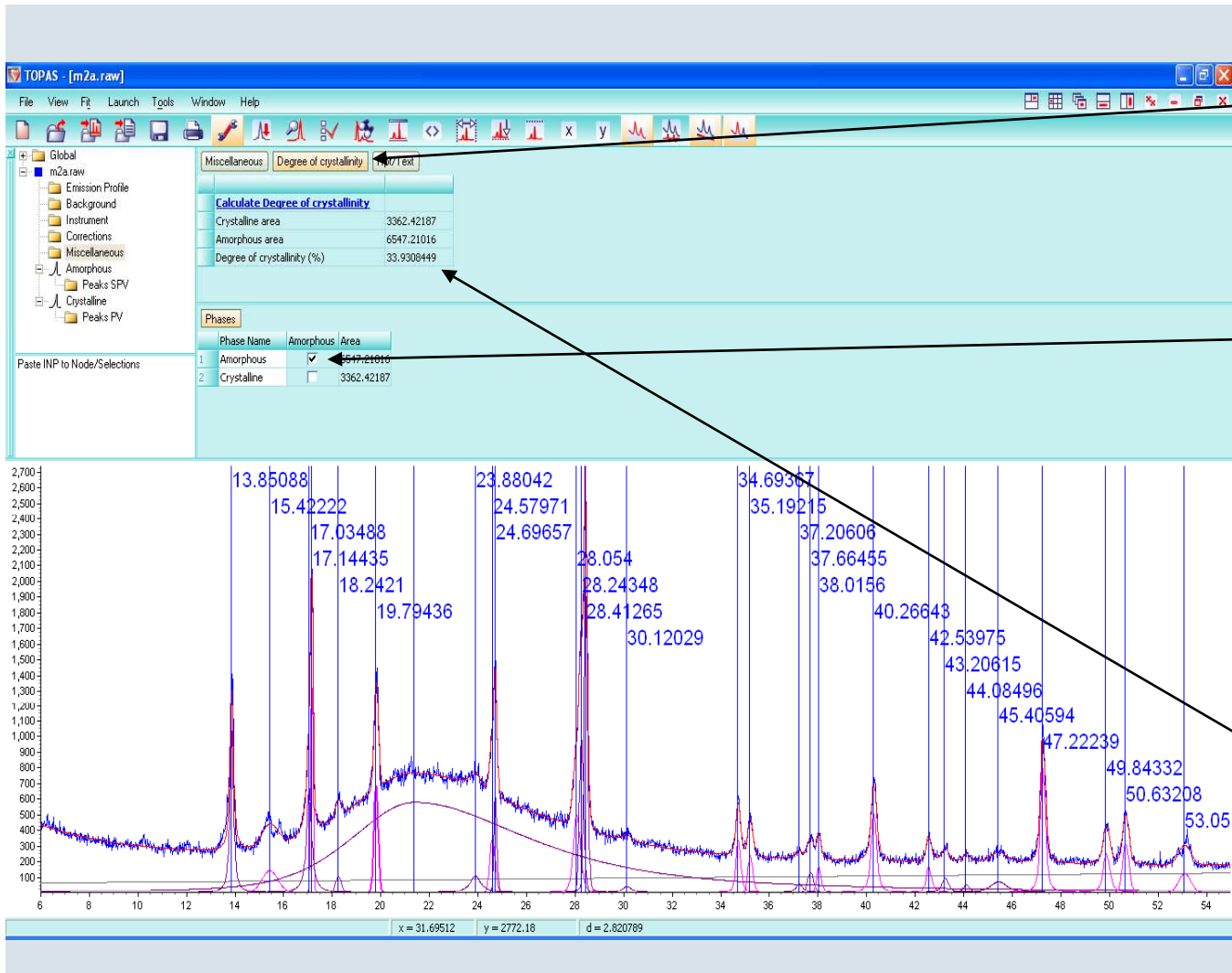
- Insert peaks for all major crystalline peak positions.
- It is recommended to use FP peaks if you know the experimental conditions. Otherwise, pick an analytical peak shape such as PV.
- Start another refinement by pressing F6.

Step 7



- Both the crystalline and amorphous phase scattering should now be mostly simulated.
- Add in additional crystalline peaks if necessary, to account for weaker crystalline peaks (for example)
- Refine again with F6

Step 8



- Highlight the Miscellaneous item in the list and click the "Percent Crystallinity" button.
- Choose the peaks phase(s) that represent the Amorphous contribution.
- Click the "Calculate Degree of crystallinity" link.
- Here degree of crystallinity is about 34%.

Comments

- A weakness of the EVA method is that it requires a user to pick the background representing the amorphous scatter and therefore introduces some subjectivity to the calculation.
- The TOPAS method fits the amorphous scattering and the crystalline scattering simultaneously so if a similar fitting process is followed, the value for percent crystallinity should be more repeatable for different users.
- Percent crystallinity values for EVA and TOPAS may differ from one another due to differences in the way the background and amorphous scattering are determined. Therefore it is recommended to use one method or the other unless it can be clearly demonstrated that both techniques result in the same values.