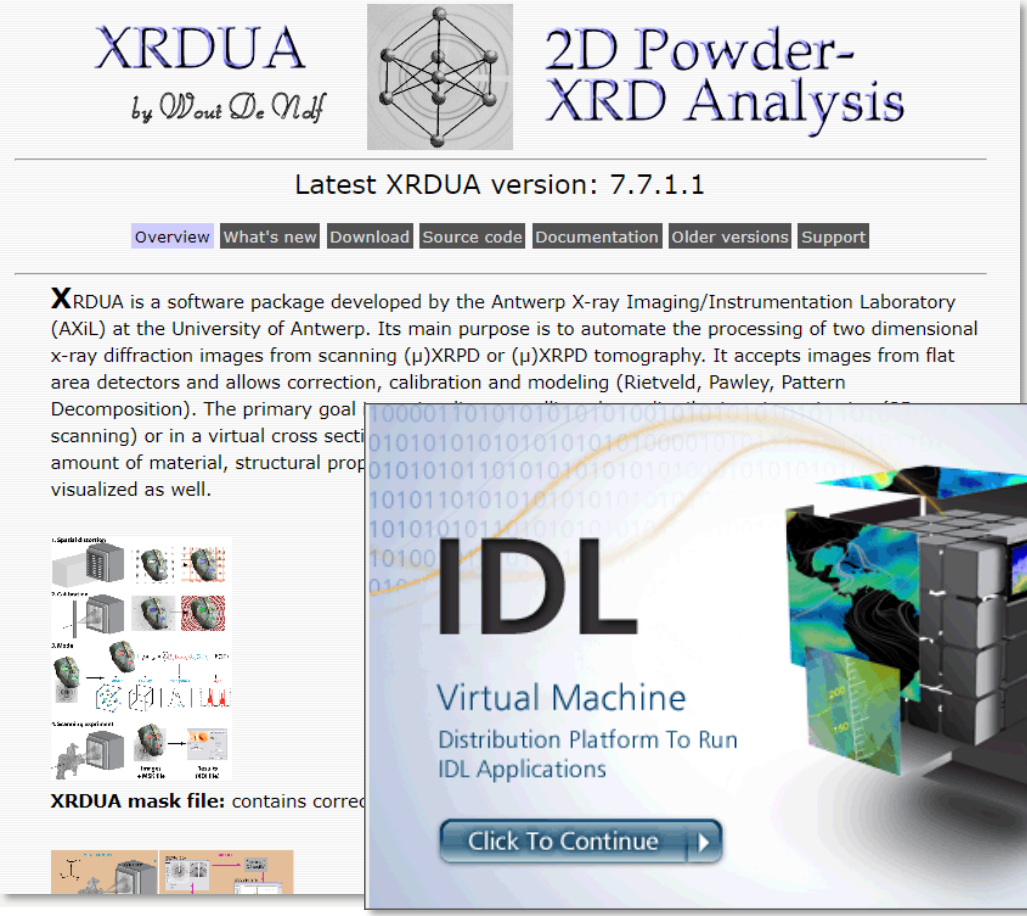
XRDUA – How to process (ID13) XRD imaging data

Frederik Vanmeert

03/12/2024



Starting with XRDUA

1. Install IDL 8.3 or higher (no license required).
2. Download XRDUA from https://xrdua.sourceforge.net/
3. Unzip the xrdua_vx-x-x-x.zip package.
4. Double-click "xrdua.sav" (**Windows**) or type "idl -vm=xrdua.sav" in a shell (**Linux/MAC**).

What to expect

- General introduction to XRDUA
- Most useful options to help you get from ID13 EDF integrated data to compound-specific distribution maps
- Some tips on how to use the software with example workflows
- (Strong) bias to pigments and paint samples

What not to expect

- Exhaustive overview and understanding of XRDUA
- Many useful/advanced options that we won't have time for
- Blindly follow to success

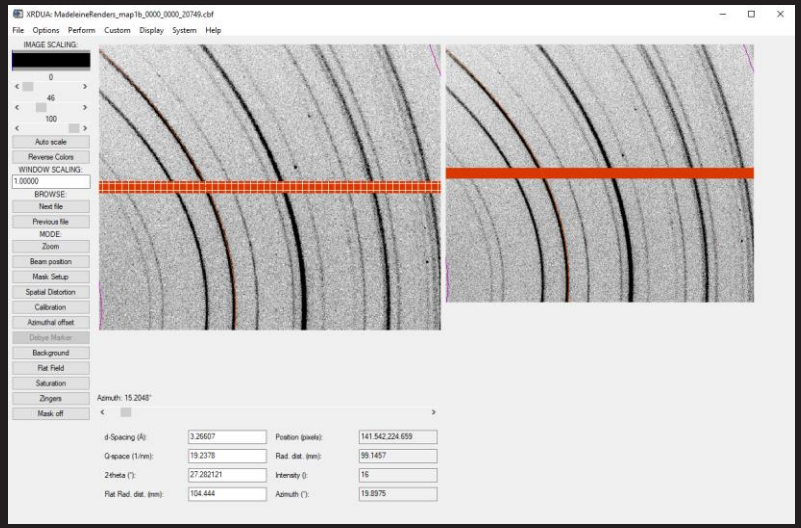
XRDUA
by Wout De Nolf

2D Powder-XRD Analysis

Universiteit Antwerpen

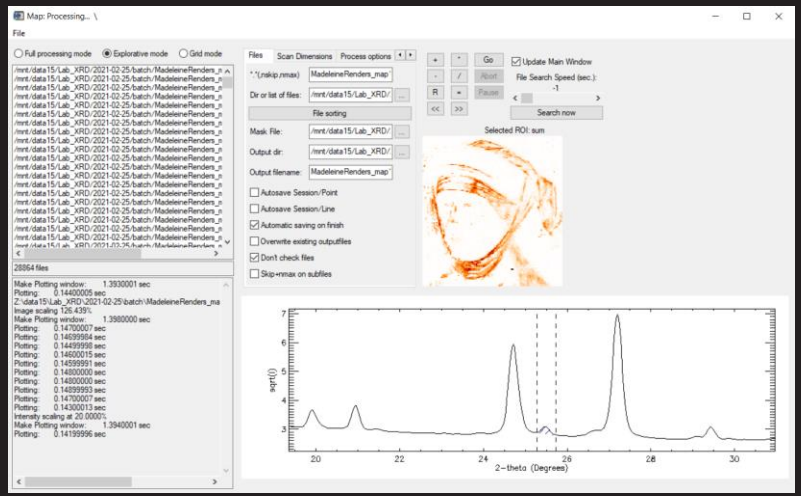
https://xrdua.sourceforge.net
https://sourceforge.net/projects/xrdua/

Dr. Wout de Nolf
(ESRF)



From raw diffraction data to phase distributions

- 2D diffraction image corrections
  - Spatial distortion / Flat field / Saturation / Masking / …
- Calibration
- (batch) Azimuthal integration
  - Mean / median / …
- ROI imaging
- Reference matching
- Autonomous whole pattern fitting
  - Rietveld, Pawley, PD
- Visualization (based on scaling factors, weight fractions, …)
- …



De Nolf, W., et al. (**2014**). *"XRDUA: crystalline phase distribution maps by two-dimensional scanning and tomographic (micro) X-ray powder diffraction."* Journal of Applied Crystallography 47(3): 1107-1117.

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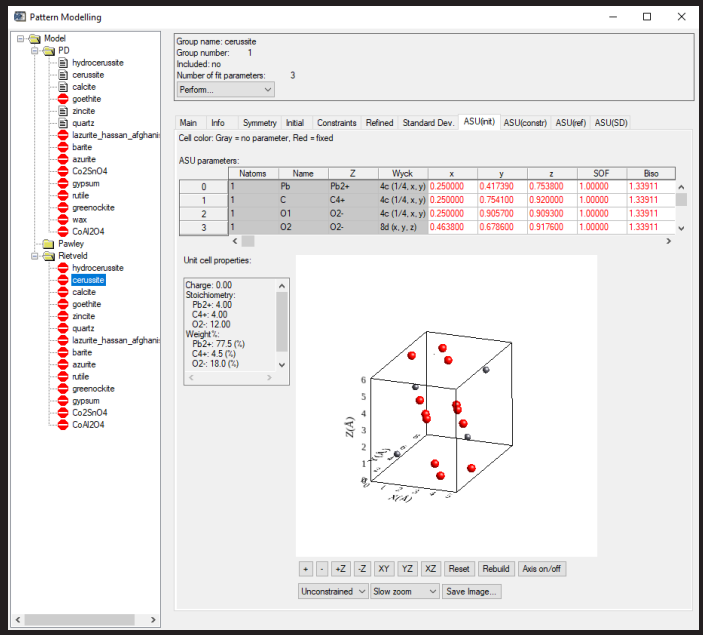

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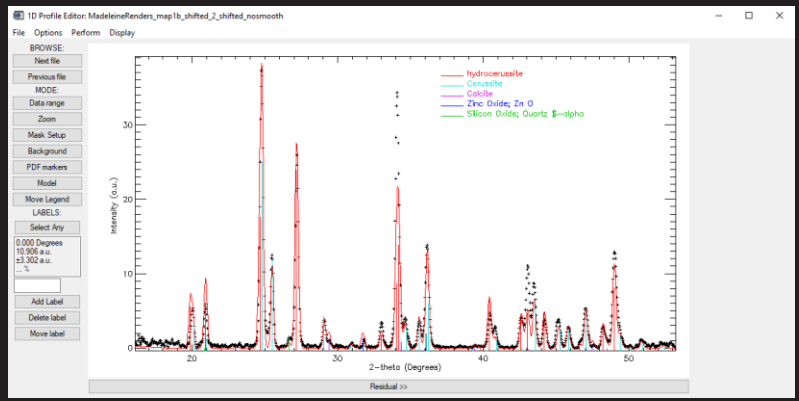

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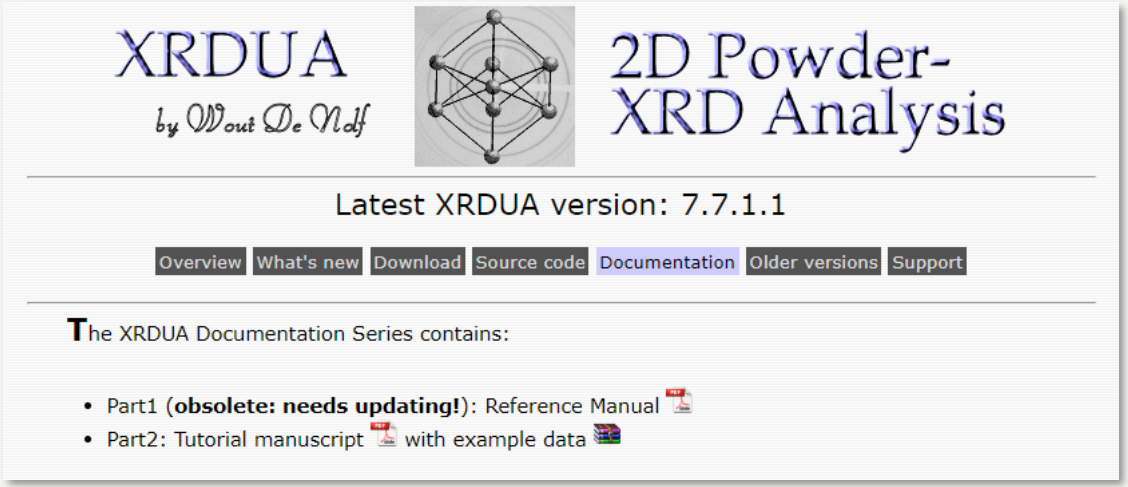
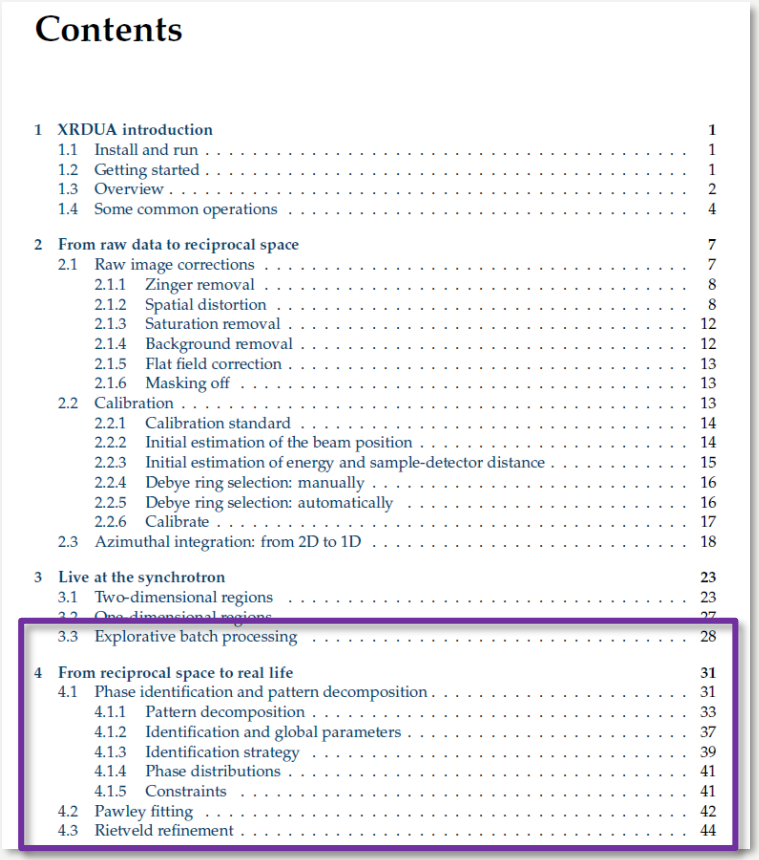


A more elaborate tutorial with manual and example data is available on the XRDUA website: [XRDUA Distribution Page]



Contents

Official XRDUA tutorial

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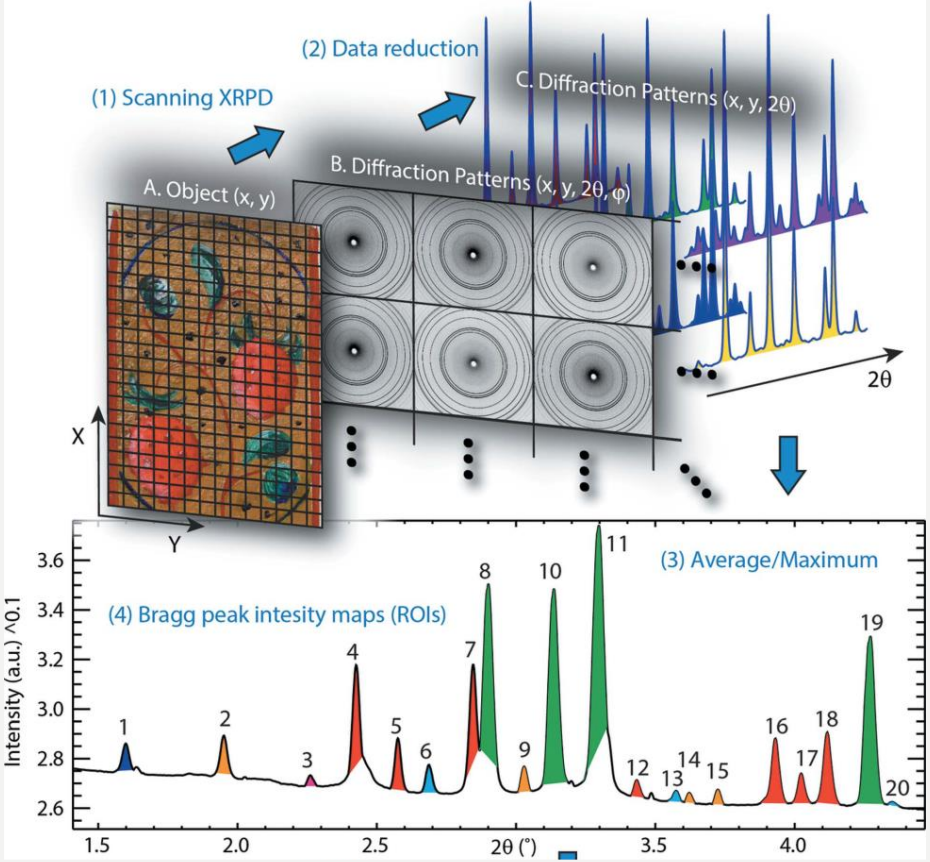

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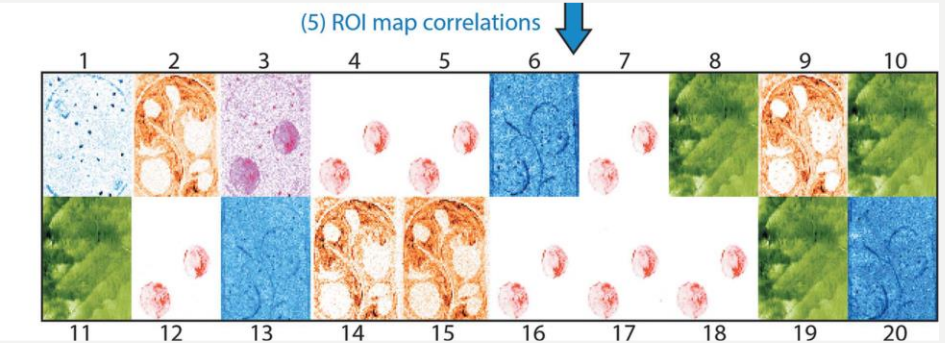

XRDUA Workflow

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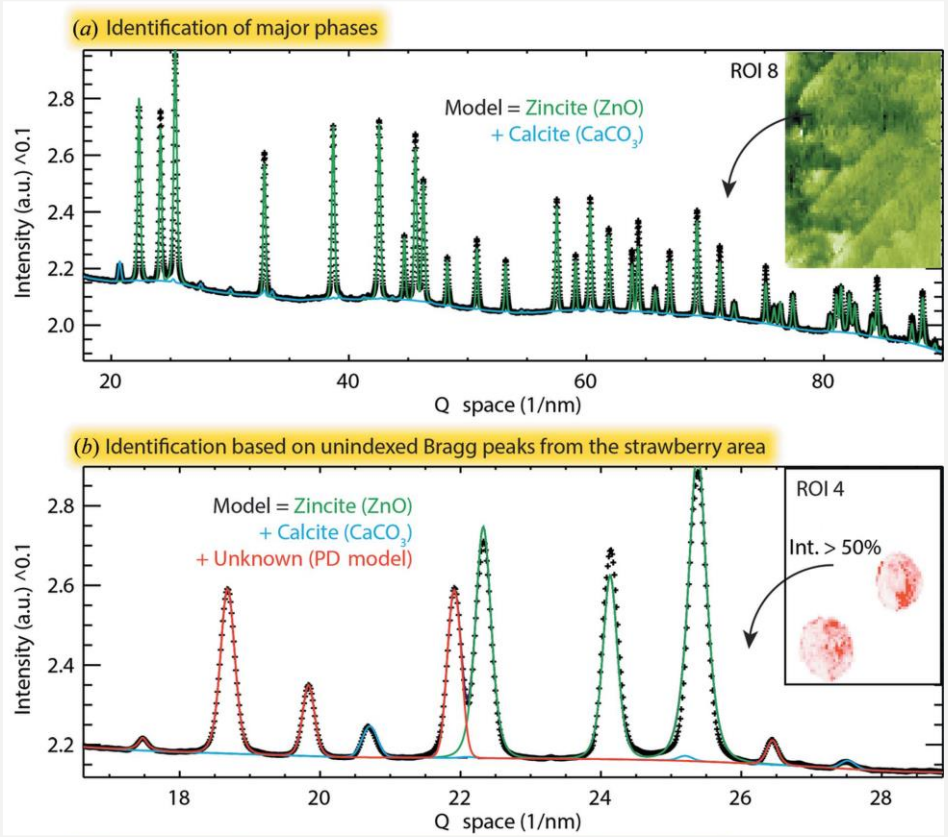

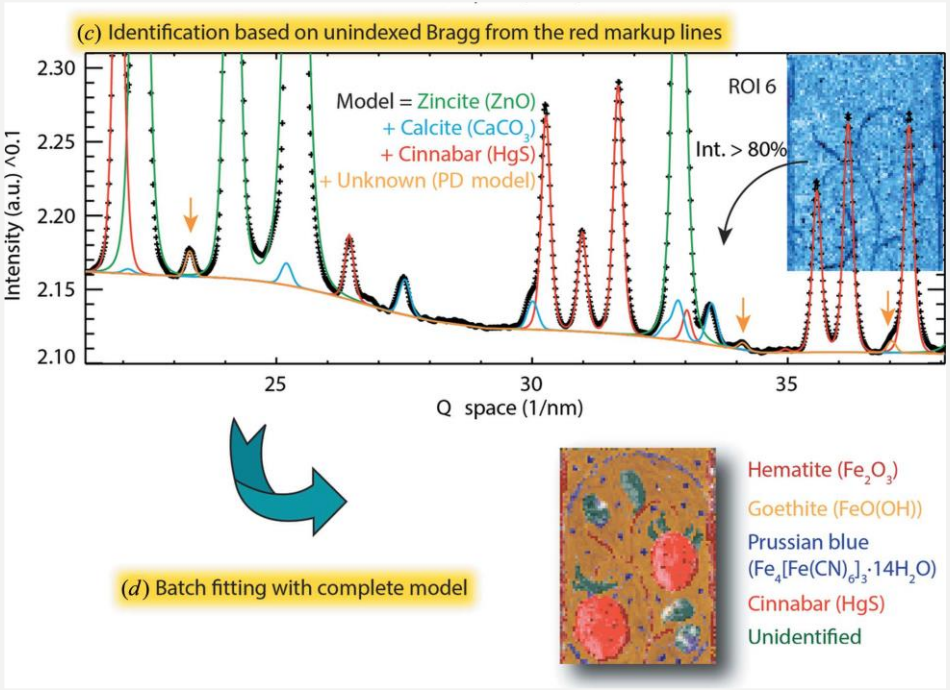

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XRDUA Workflow

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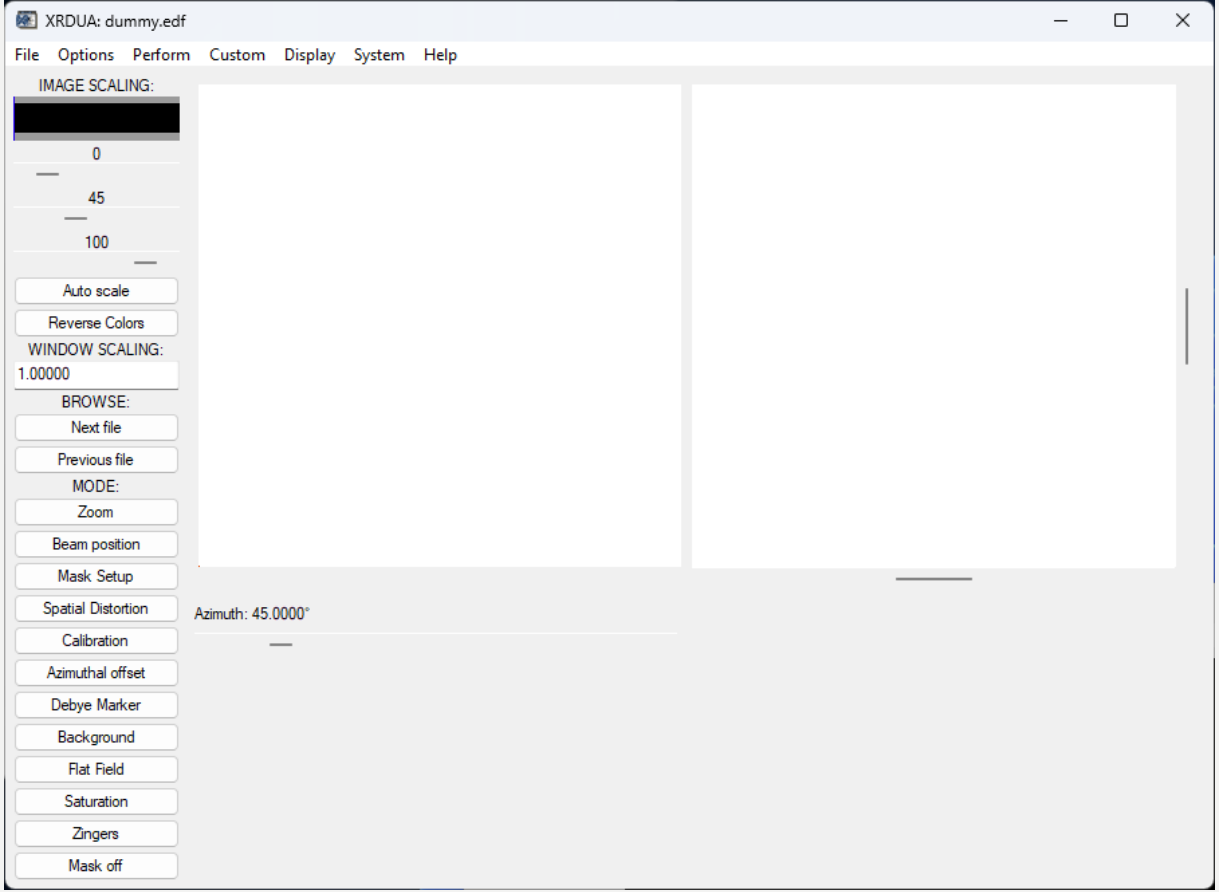
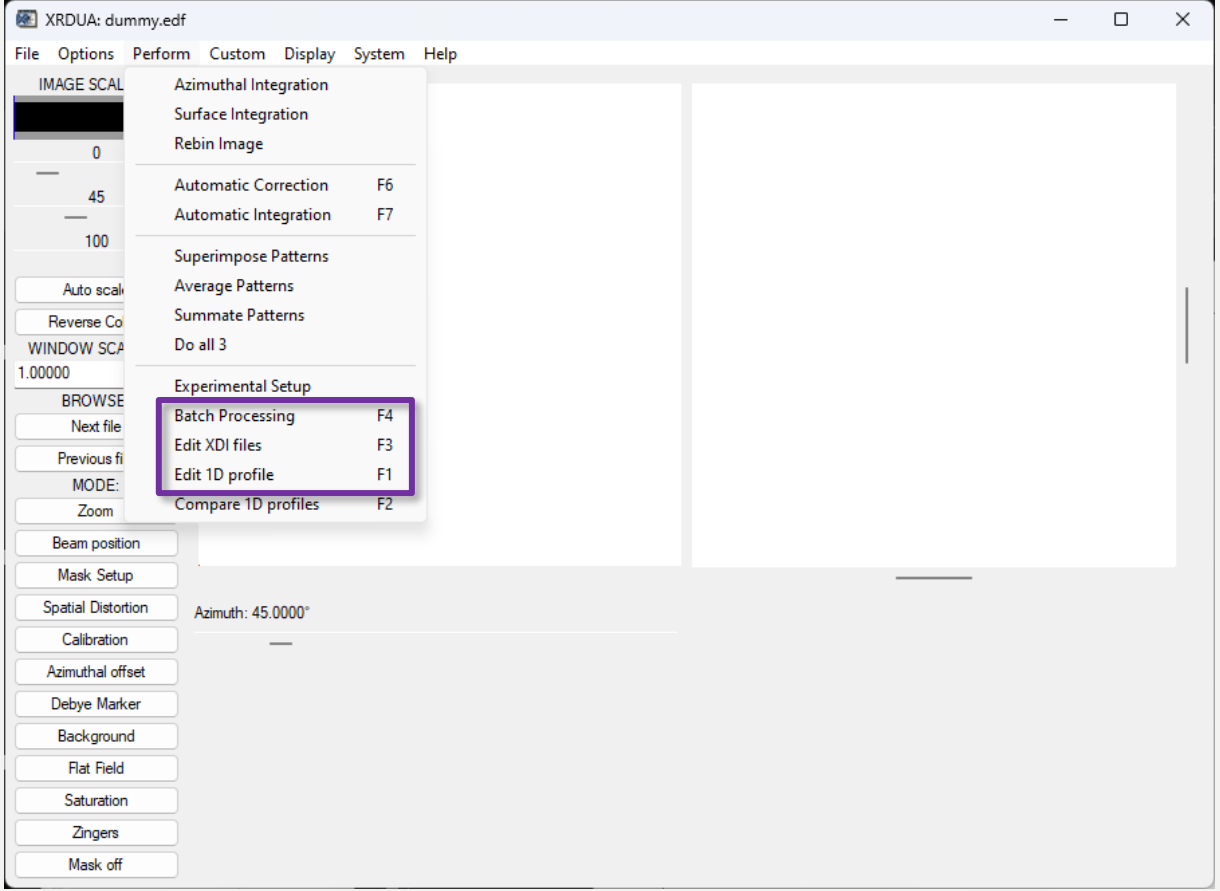


XRDUA 2D

- visualizing 2D patterns
- 2D pattern corrections
- geometry calibration
- defining settings for azimuthal integration
- …

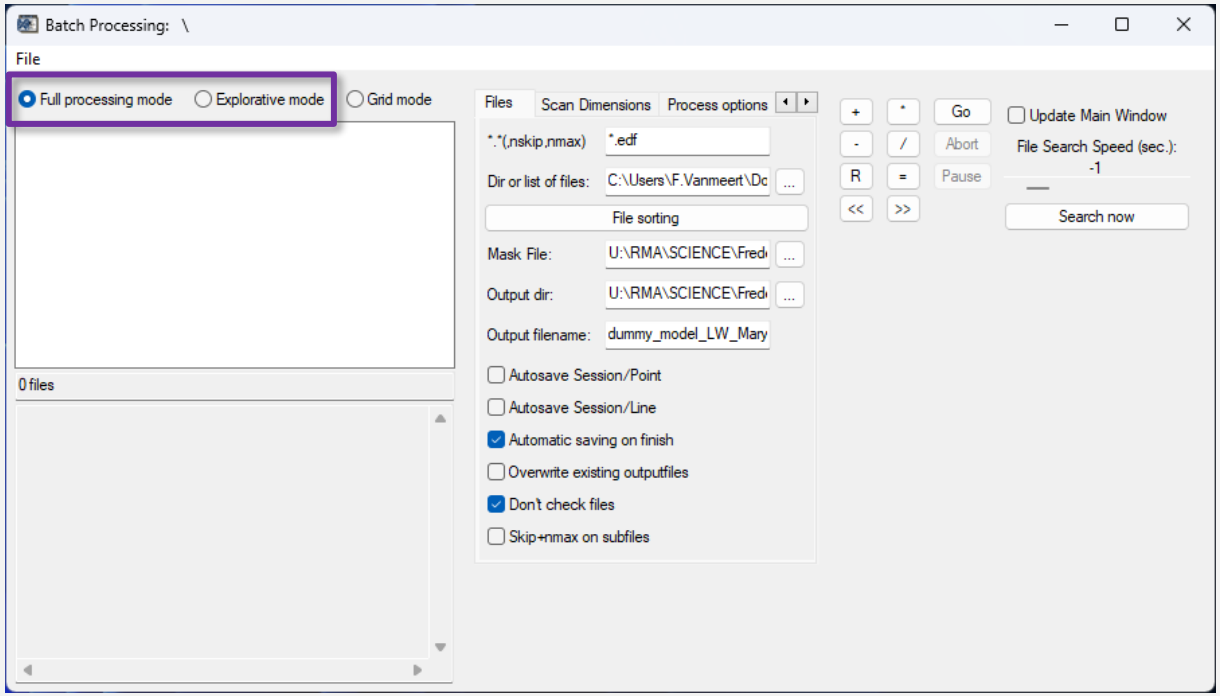
Main windows: **XRDUA 2D**



XRDUA 2D

Perform ->

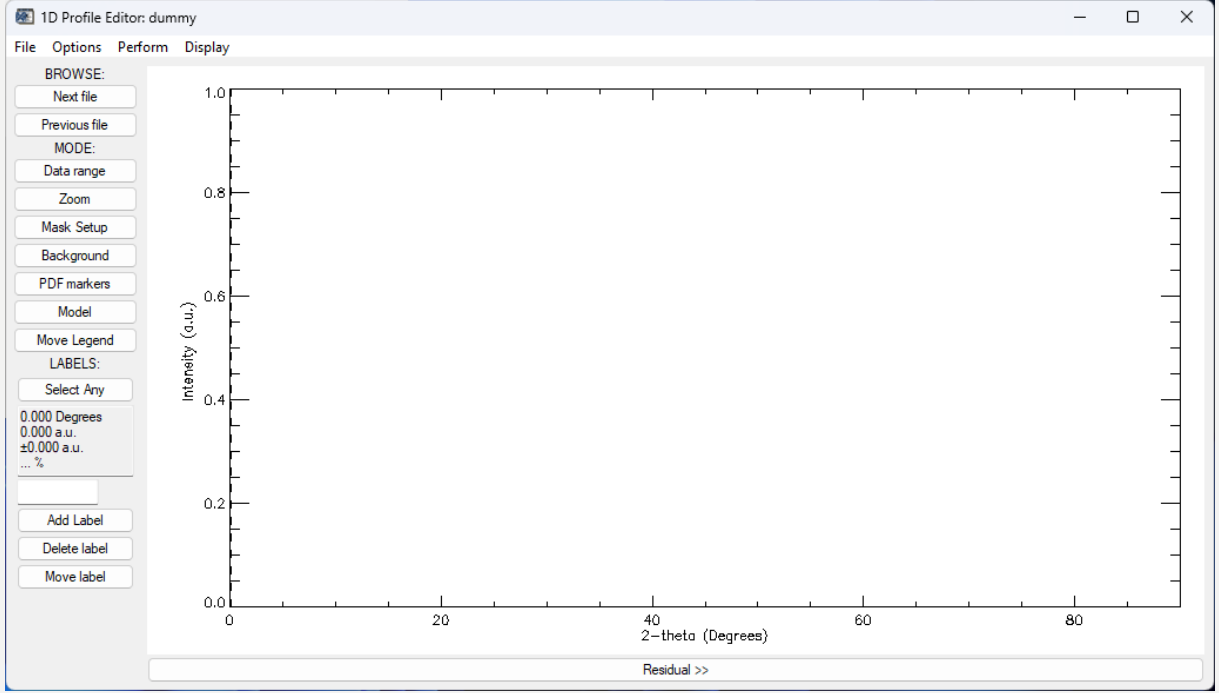
- *Batch Processing*
  - Perform batch processes
  - 2D -> 1D
  - 1D -> distribution maps
- *Edit 1D profile*
  - 1D diffractograms
  - Fitting model
- *Edit XDI files*
  - Results



XRDUA BP

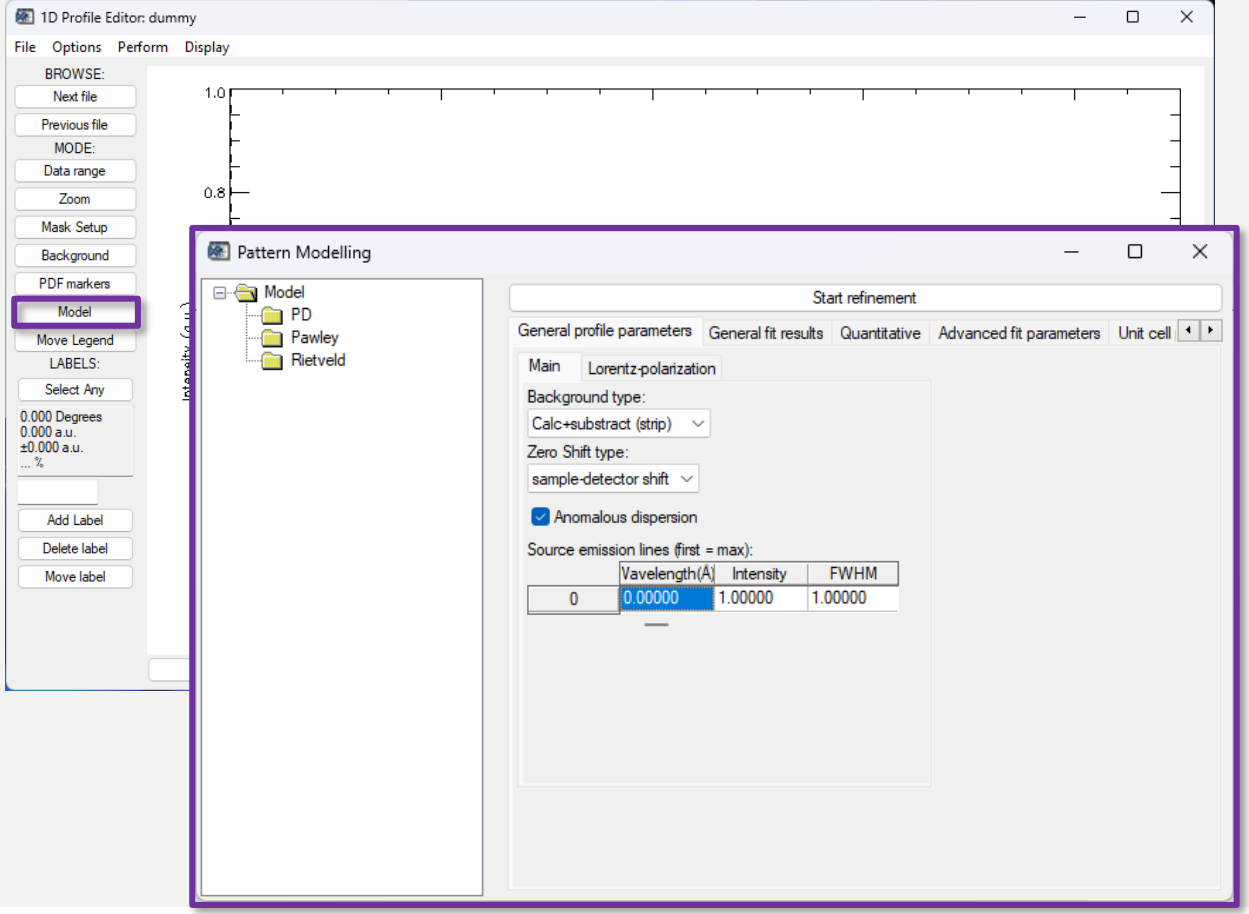
- *Explorative Mode*
  - Perform batch azimuthal integration (2D to 1D)
  - Explore data
  - ROI imaging
  - Extract 1D diffractograms
- *Full Processing Mode*
  - Perform batch fitting (1D to distribution maps)

Main windows: **XRDUA BP**



XRDUA 1D

- *Main Window*
  - Shows 1D diffractograms
  - Background subtraction
  - Overlay PDFs
  - …
- *Model*
  - Create fit models



XRDUA 1D

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Main windows: **Modelling**

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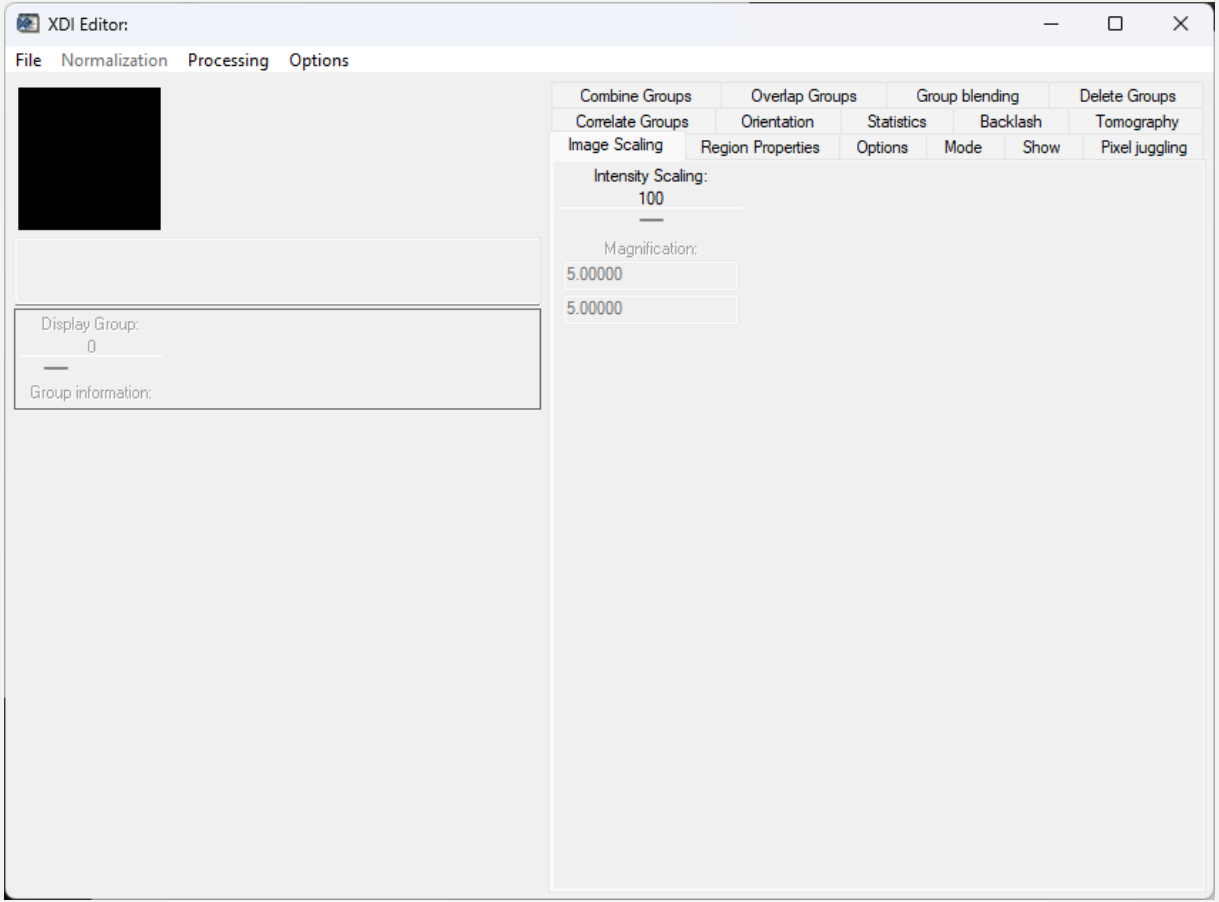
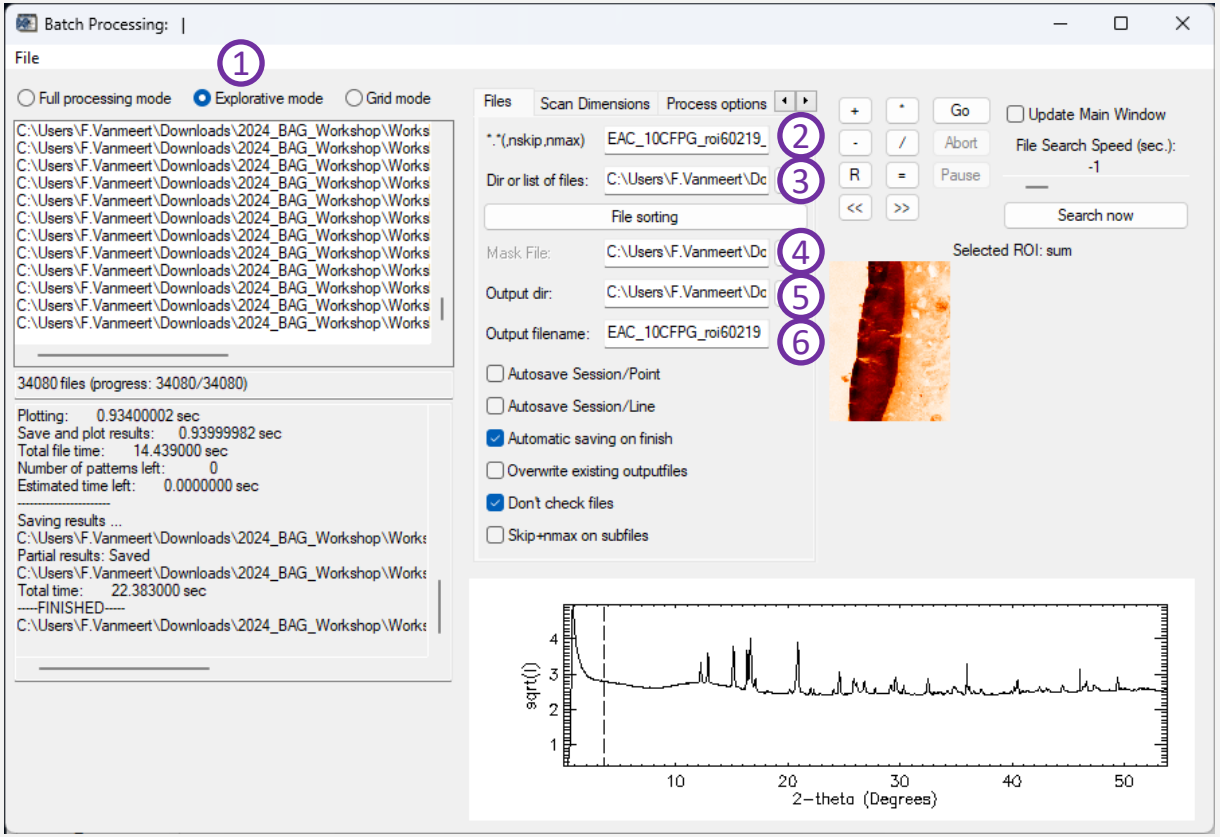


XRDUA XDI

- *Results (groups)*
  - (Intensity) Scaling
  - Edit/combine results
  - Resize results
  - …
- *Tomography*
  - Single slice tomography reconstruction



XRDUA BP

Files

1. Explorative Mode
2. Filename of edf file
3. Directory of edf file
4. Mask file (dummy_calib.msk)
5. Output directory
6. Output filename

CAREFULL!

- Always press ENTER when manually inputting data!
- Changing the *mask file*, also changes the *output directory* and the *output filename*!

1. Loading ID13 EDF data

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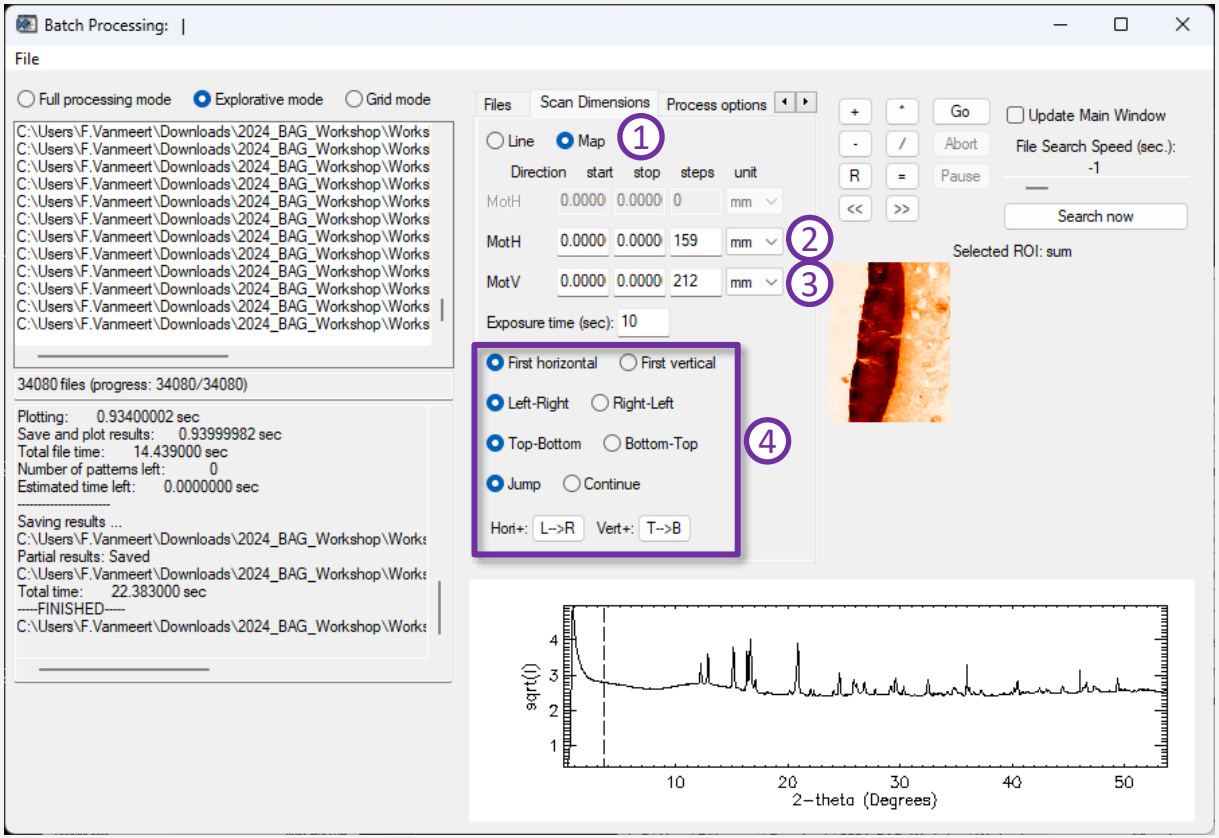

XRDUA BP

Scan Dimensions

1. Map
2. MotH steps: #pixels - 1
3. MotV steps: #pixels - 1
4. Options to change the orientation of the map

1. Loading ID13 EDF data

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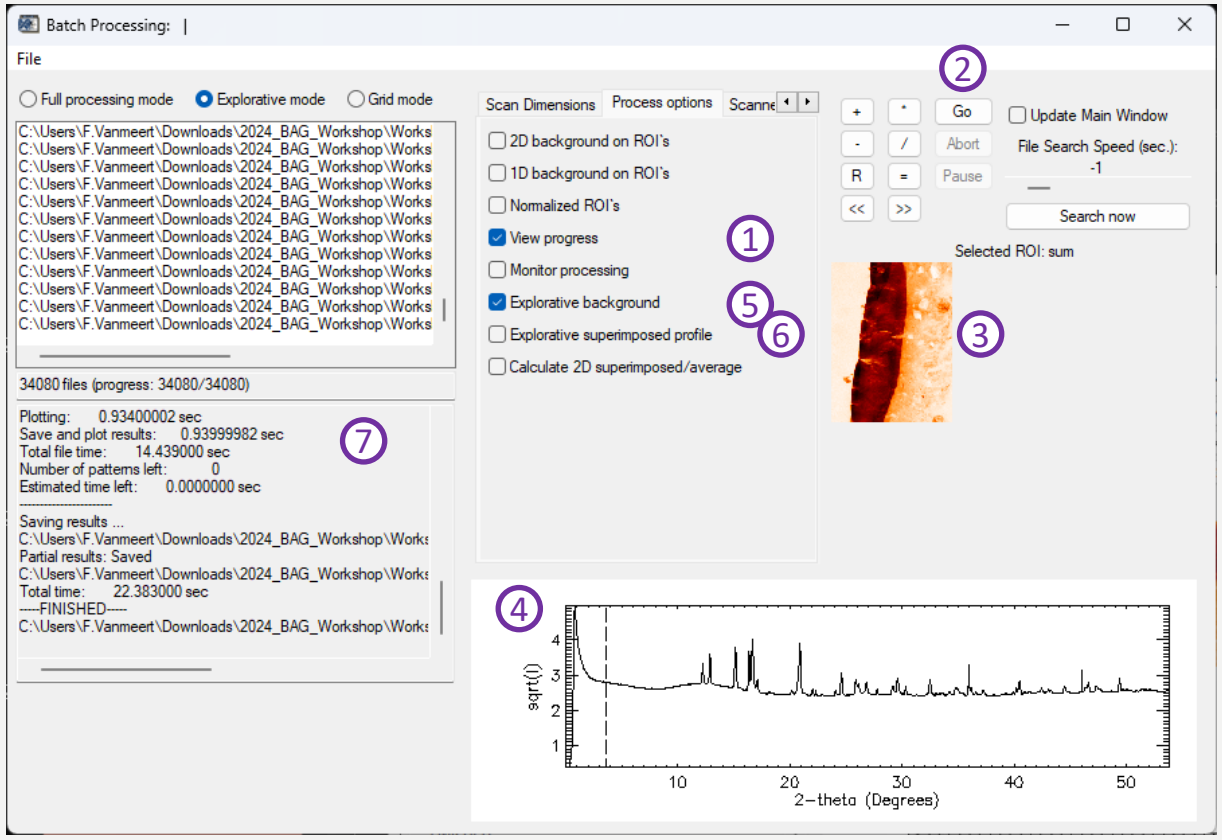
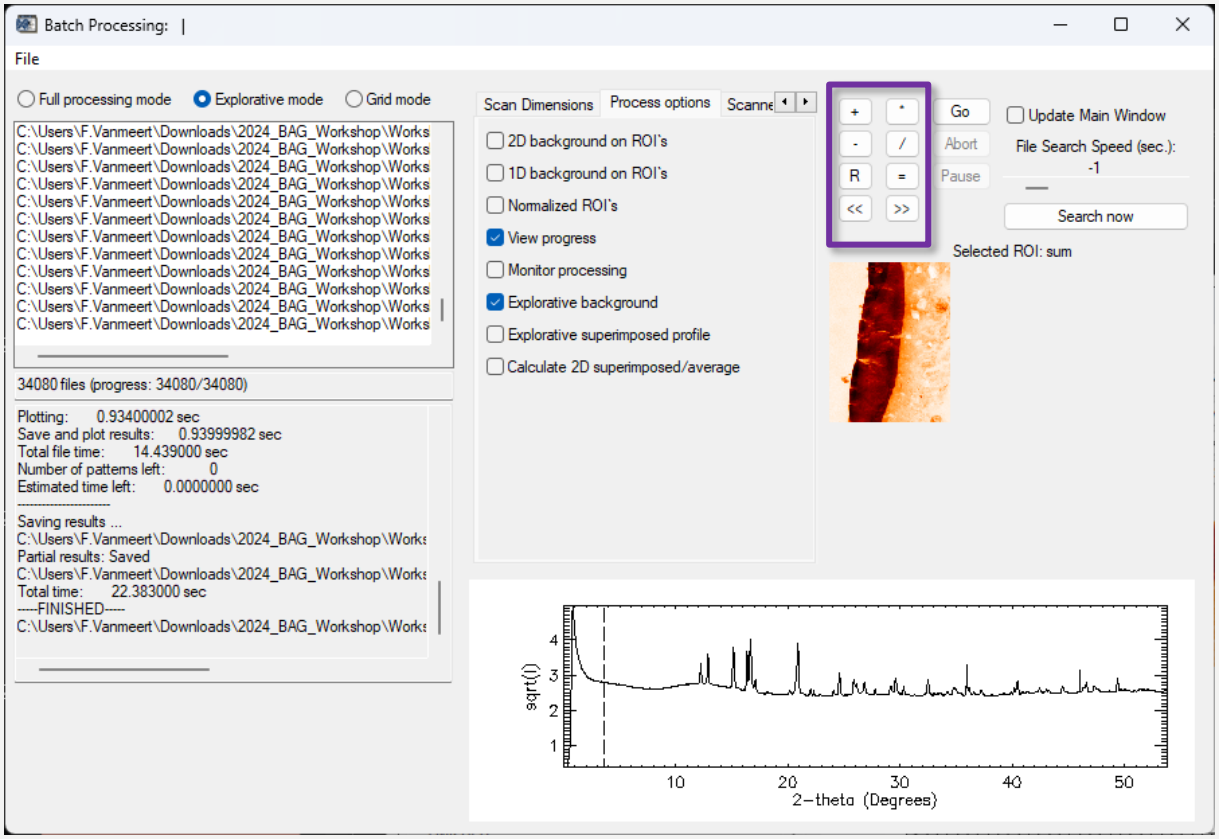


XRDUA BP

Process options

1. View progress to view the ROI image
2. **Go!**
3. ROI Image
4. Average (default) profile of the entire map
5. Explorative background applies a linear background under the ROI
6. Show superimposed profile instead of average profile
7. Some useful output
   - BP session, 1D tiff and XDI are automatically saved

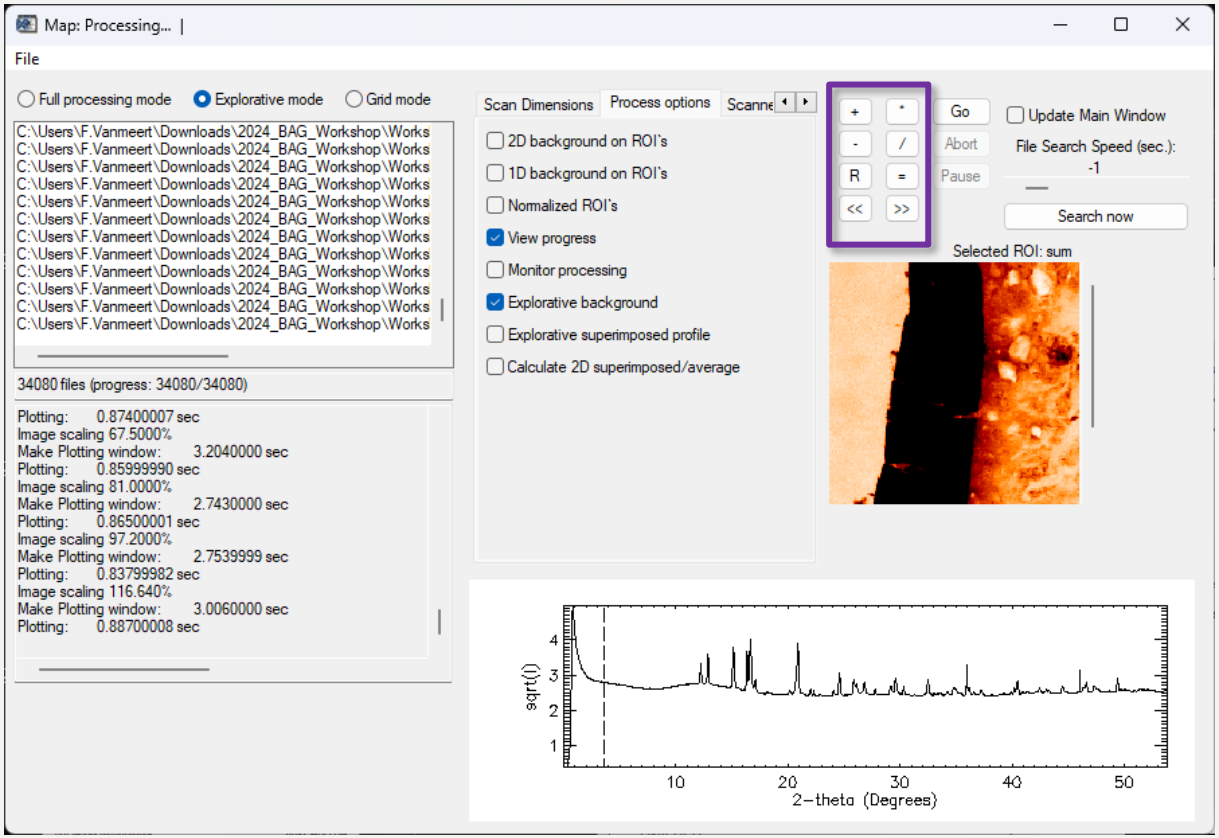
1. Loading ID13 EDF data



XRDUA BP

ROI image visualization

- **+** : increase ROI image size
- **-** : decrease ROI image size
- **R** : reset ROI image size
- ***** : decrease contrast
- **/** : increase contrast
- **=** : set value for contrast based on max. intensity



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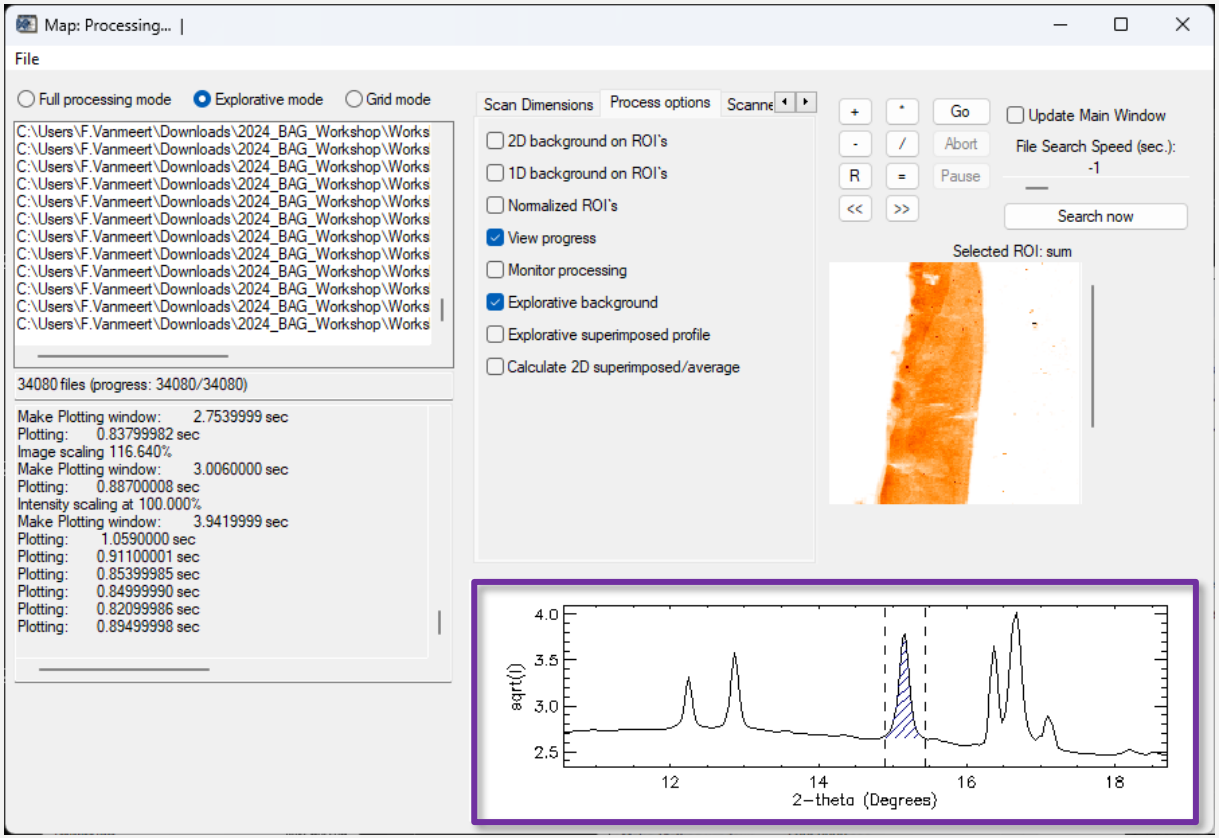
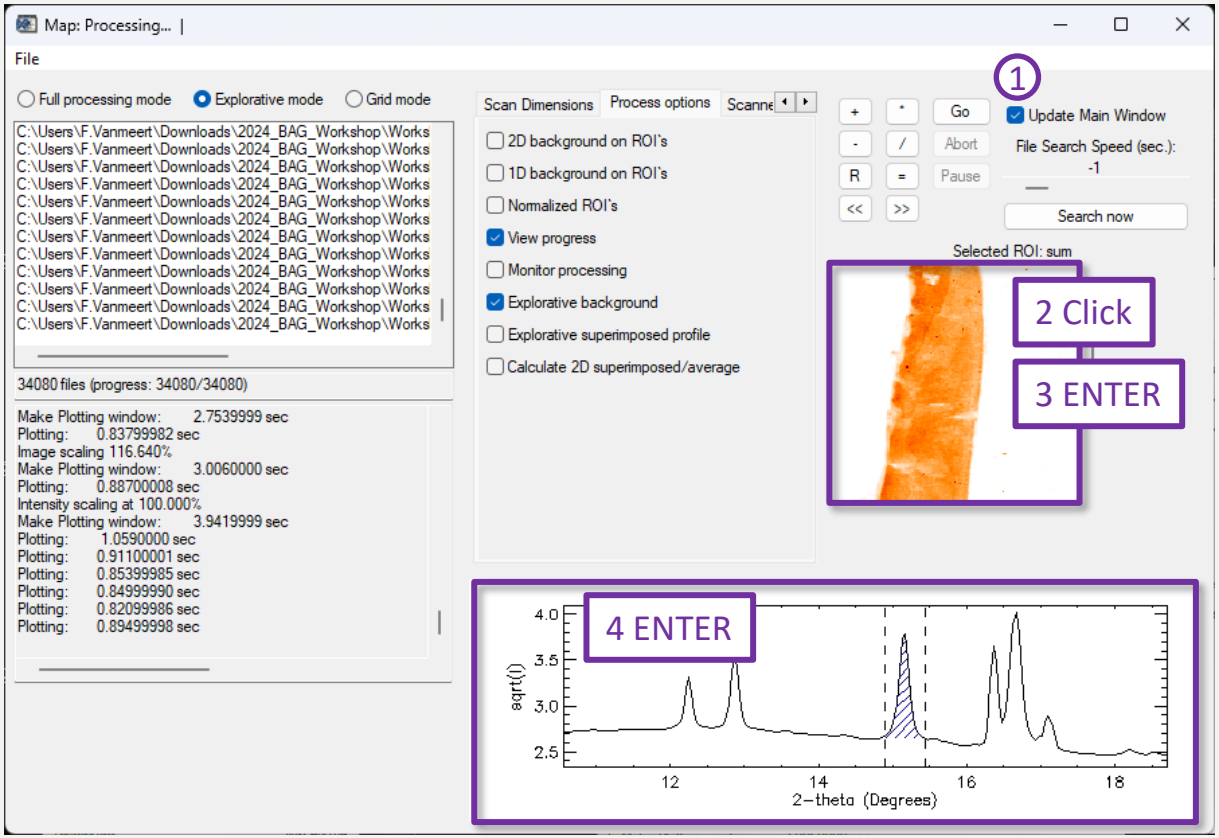


XRDUA BP

1D profile options

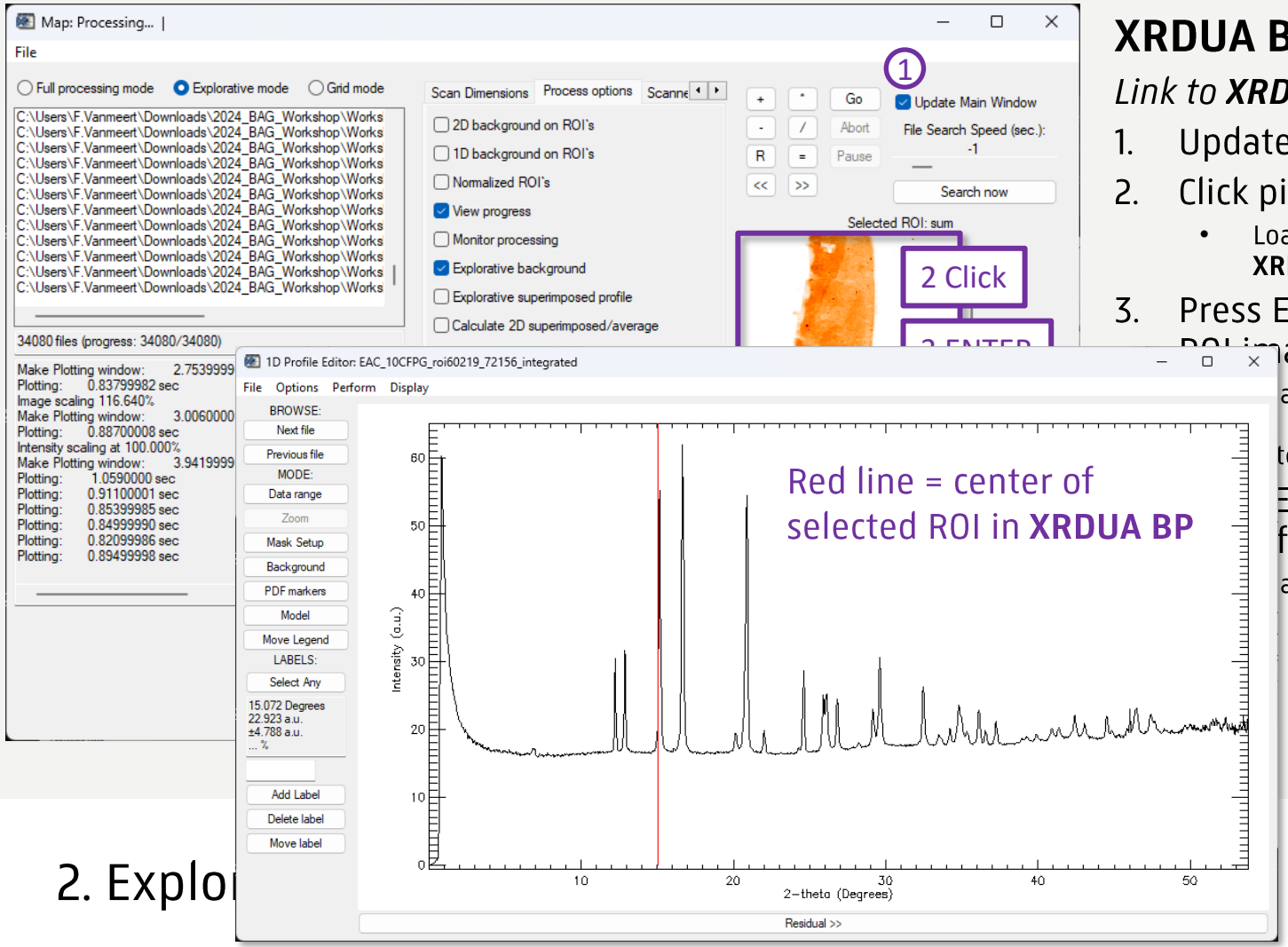
- **Right-click and hold** : zoom in
- **Single right-click** : zoom out to original
- **Left-click and hold** : select ROI
  - When a ROI is selected, you can use the left/right arrows (for small jumps) and the up/down arrows (for large jumps) to move the ROI over the 1D profile



XRDUA BP

*Link to **XRDUA 1D***

1. Update Main Window
2. Click pixel in ROI image
   - Loads 1D profile of that pixel to **XRDUA 1D**
3. Press ENTER when cursor is in ROI image
   - Loads averaged (or superimposed) 1D profile based on current ROI intensity in **XRDUA 1D**
4. Press ENTER when cursor is in 1D profile
   - Loads averaged (or superimposed) 1D profile in **XRDUA 1D**



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ENTER when cursor is in file

ads averaged (or superimposed) profile in **XRDUA 1D**

2. Explo

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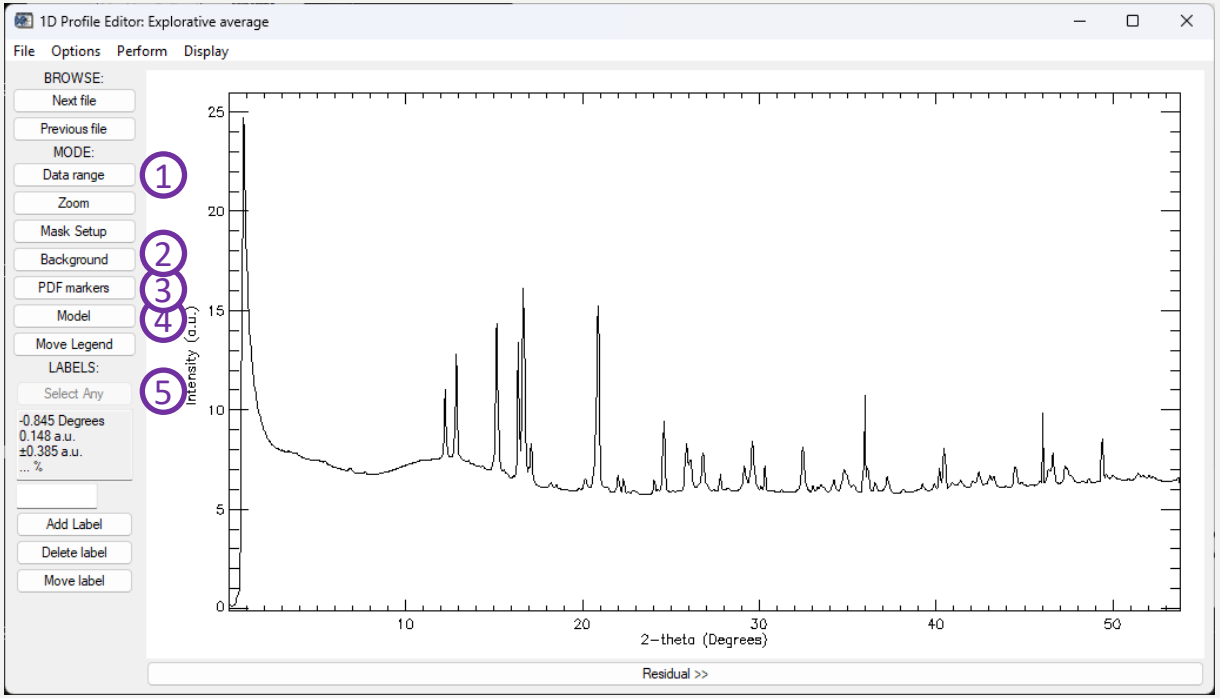
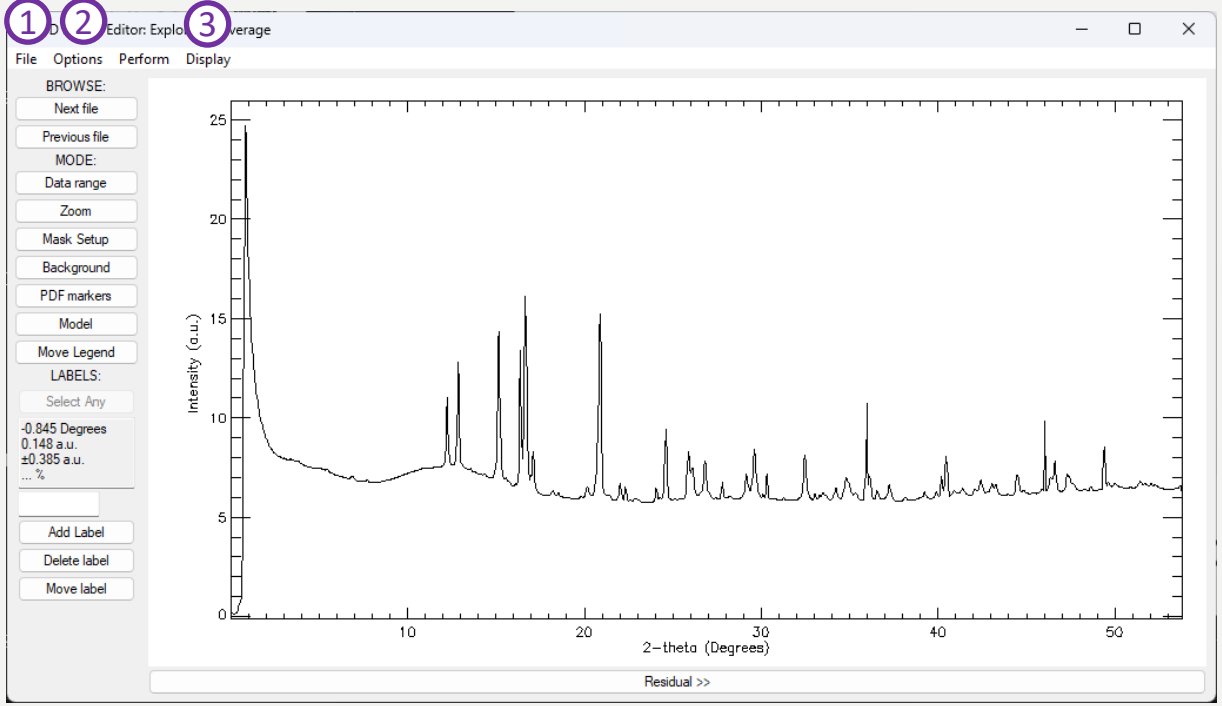


XRDUA 1D

Some main options

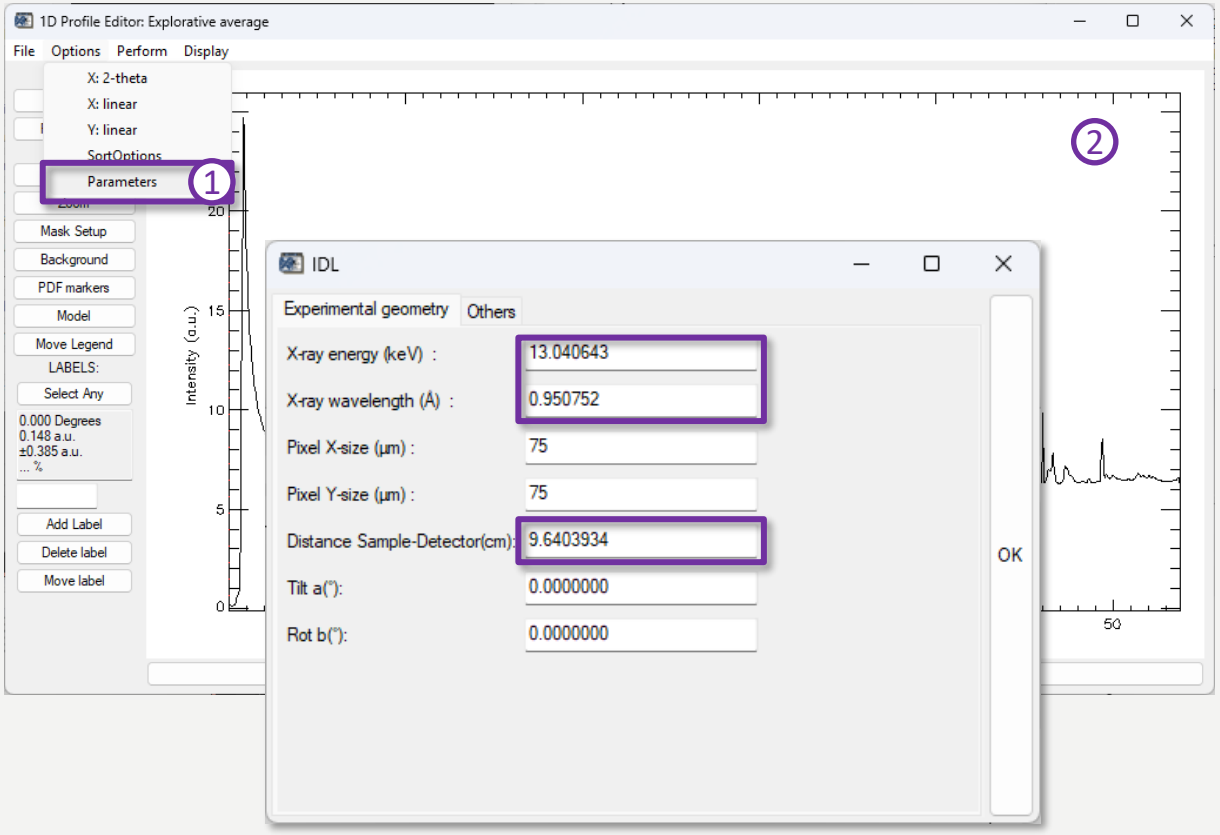
1. Set the data range to include in fit model
2. Apply background corrections
3. Go through PDF files
4. Open the Model window
5. Move the red line from **XRDUA BP**



XRDUA 1D

Some main options

1. File
   - Save/Load Mask (contains experimental parameters and fit model)
   - Load (Multiple) PDF
2. Options
   - X: Y: (Change axis options)
   - Parameters (edit geometry parameters)
3. Display
   - Fit Total (Change visualization of fit)
   - Show Peaks (Show initial estimates of the model)
   - View PDF (Enable/Disable PDF and scale PDF)



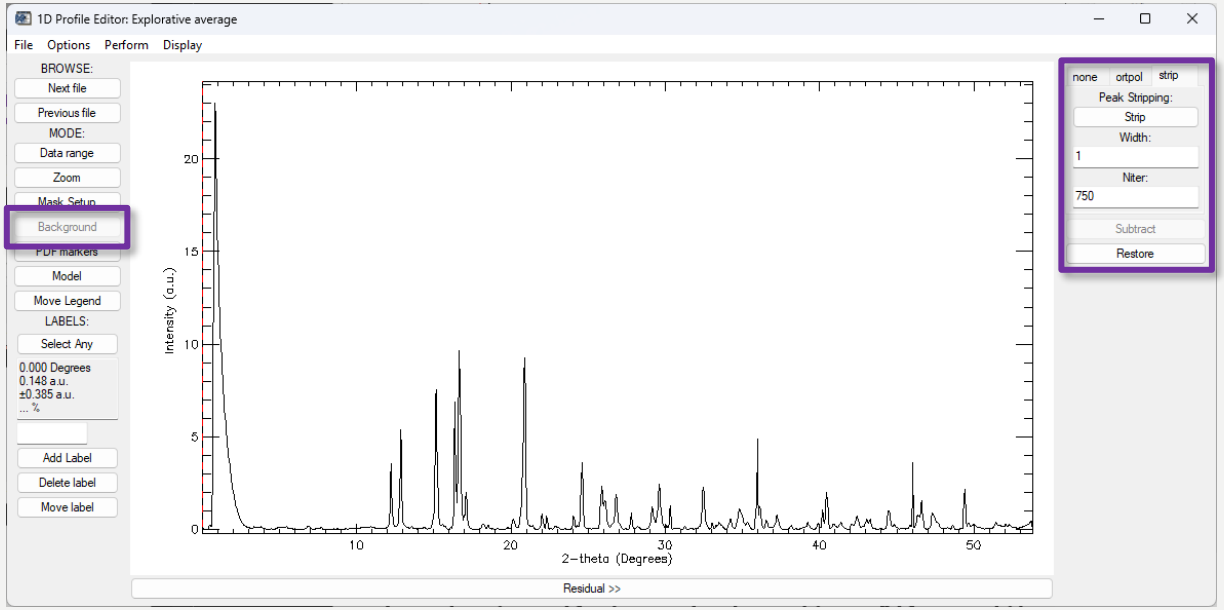
XRDUA 1D

1. Set Experimental Geometry
   - X-ray Energy/Wavelength
   - Distance Sample-Detector

These values are specific for each beamtime. Check the poni file of that beamtime for these values.

2. Reload 1D profile from **XRDUA BP**

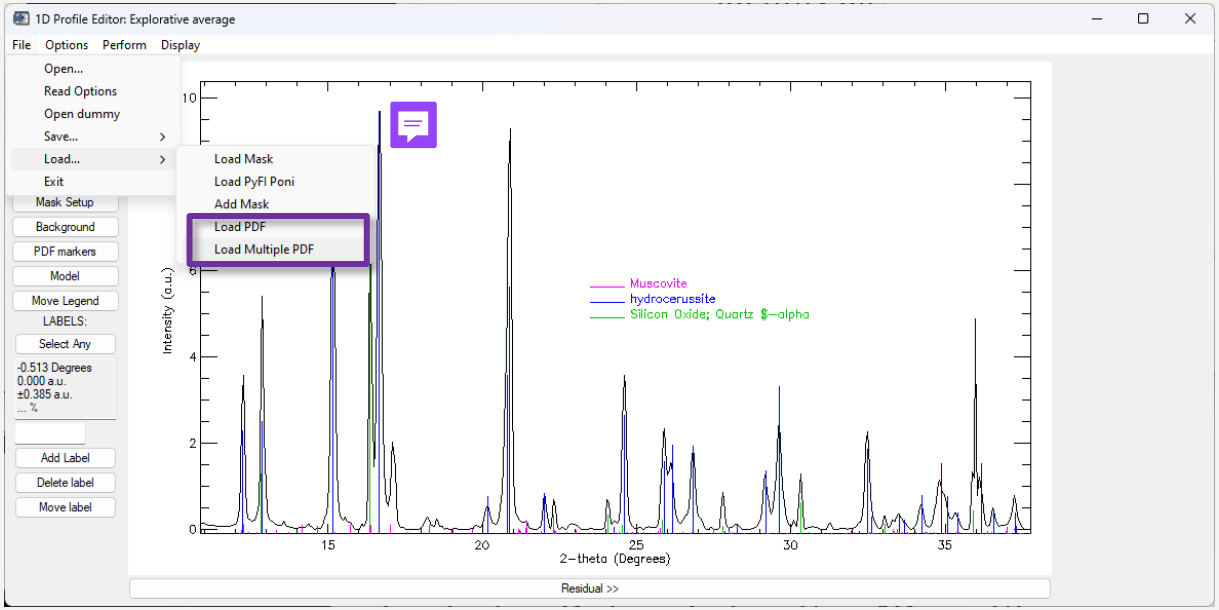
This is necessary the first time that you fill in the values of step 1 or load these values from an existing mask file.



XRDUA 1D

Background Subtraction

- The Strip background usually works well. Set the Niter value so that it follows the background without cutting away intensity from the diffraction peaks.
- The background correction is shown as a dashed green line
- Display -> Hide Background
  - To remove the dashed green line



XRDUA 1D

Identifying compounds

- Load (Multiple) PDF
- Use prior knowledge of your samples

PDF

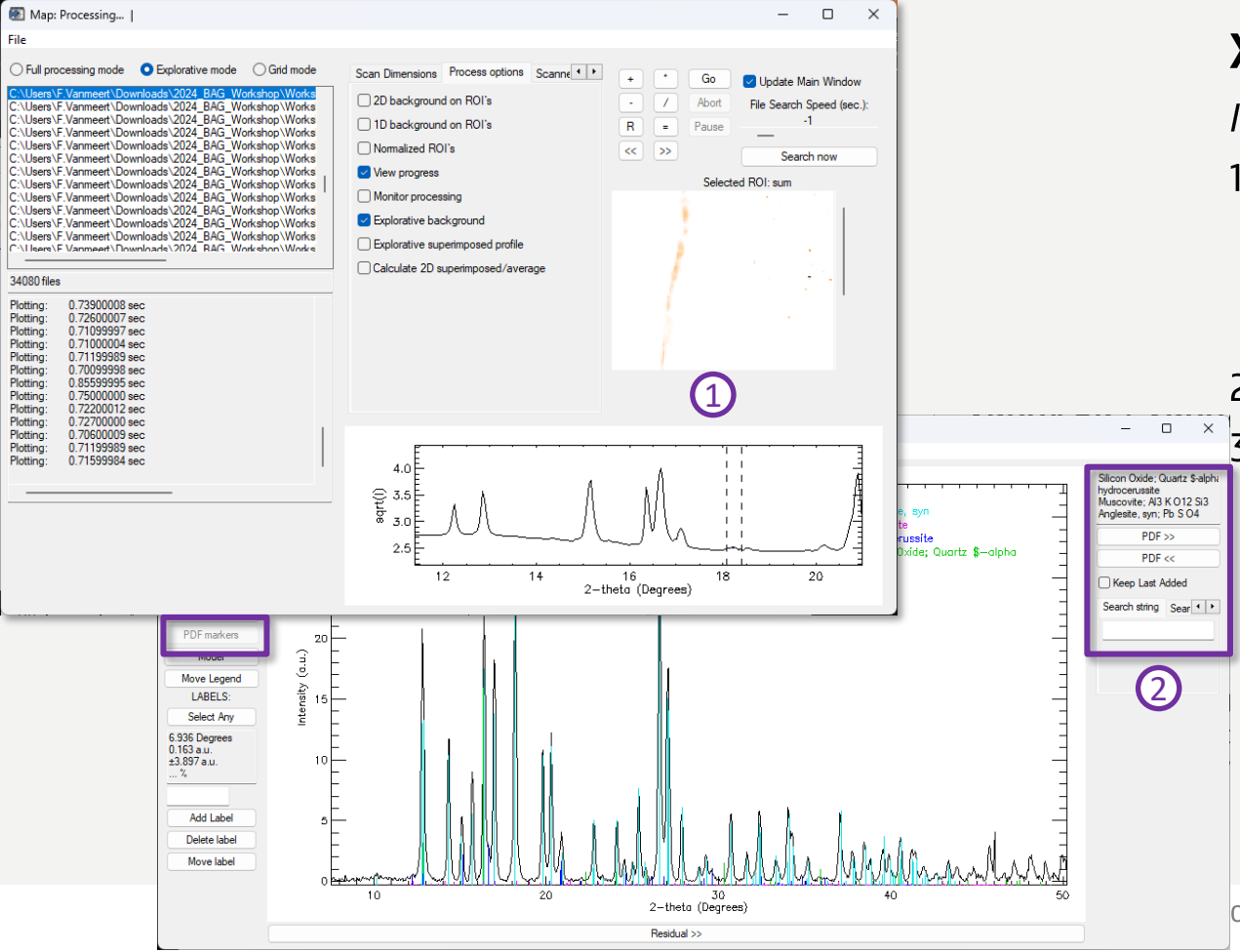
Powder diffraction files

These files are obtained through other software, such as Match! and various extensions are possible. It is also possible to create these within the XRDUA Model window (saved as .pdd).

XRDUA BP + XRDUA 1D

Identifying compounds

1. Use ROI tool in **XRDUA BP** to select unidentified peaks and obtain a good 1D profile for identification
2. Check with available PDF
3. If no PDF file can explain the signal(s), save the 1D profile in **XRDUA 1D** and use dedicated search-match software (Match!, QualX2, …).



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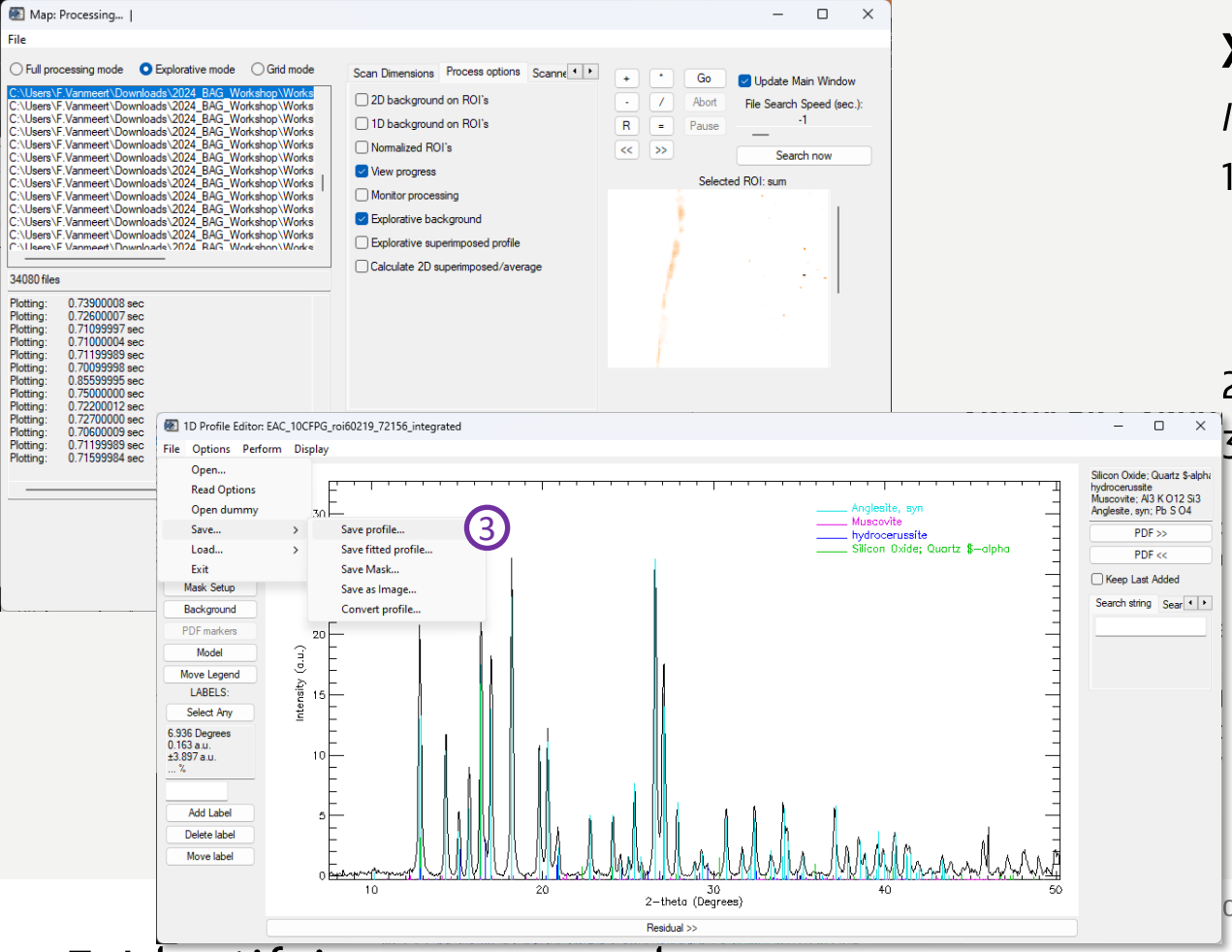

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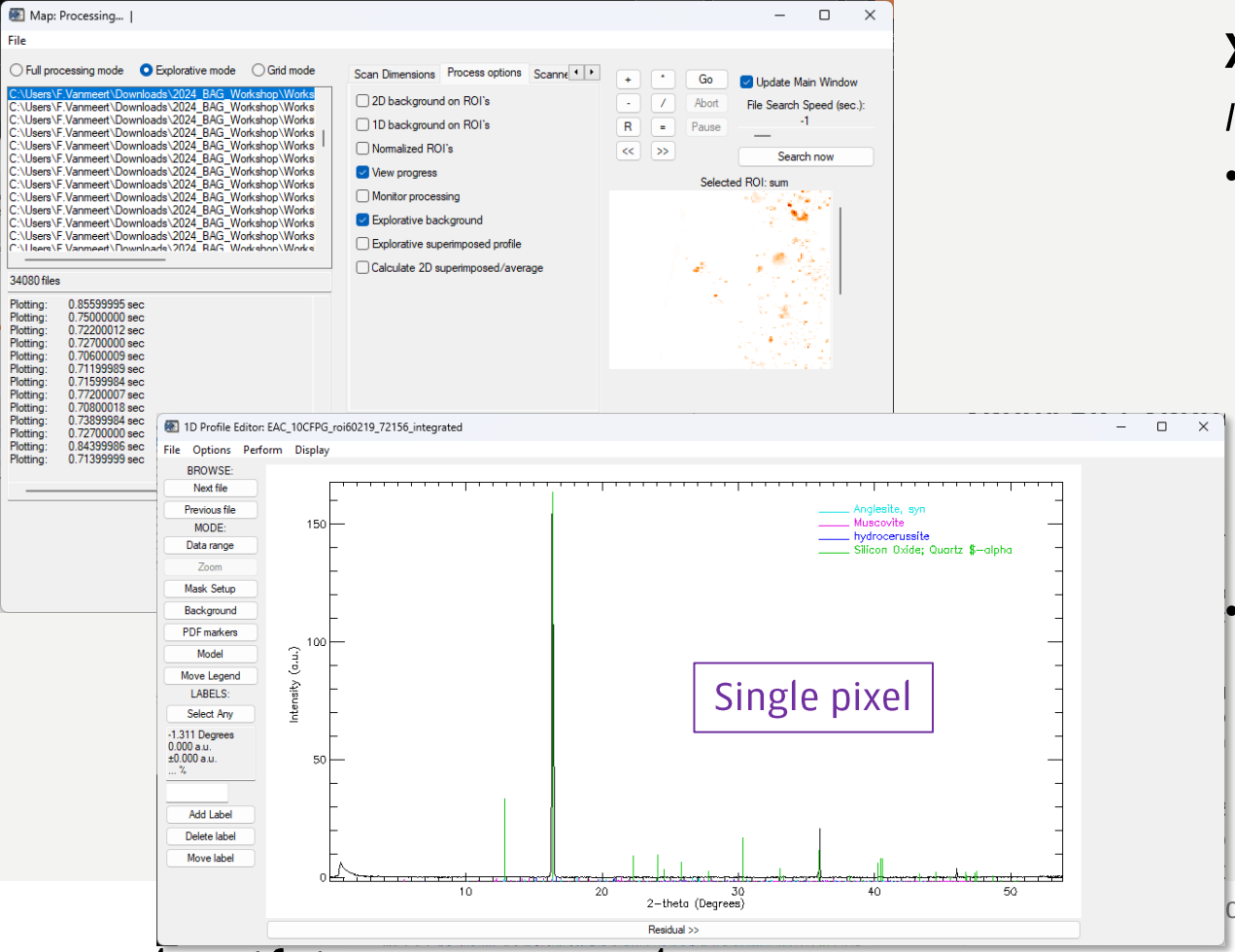

XRDUA BP + XRDUA 1D

Identifying compounds

- Several artefacts can make identification difficult and/or impossible
  - Preferred orientation: this causes a (strong) difference in relative intensity
  - "Large" crystals: this results in very strong intensities for one or a limited number of diffraction peaks for the given crystal
- No physical sample preprocessing is possible to reduce these artefacts, so be aware of the limitations of your data!

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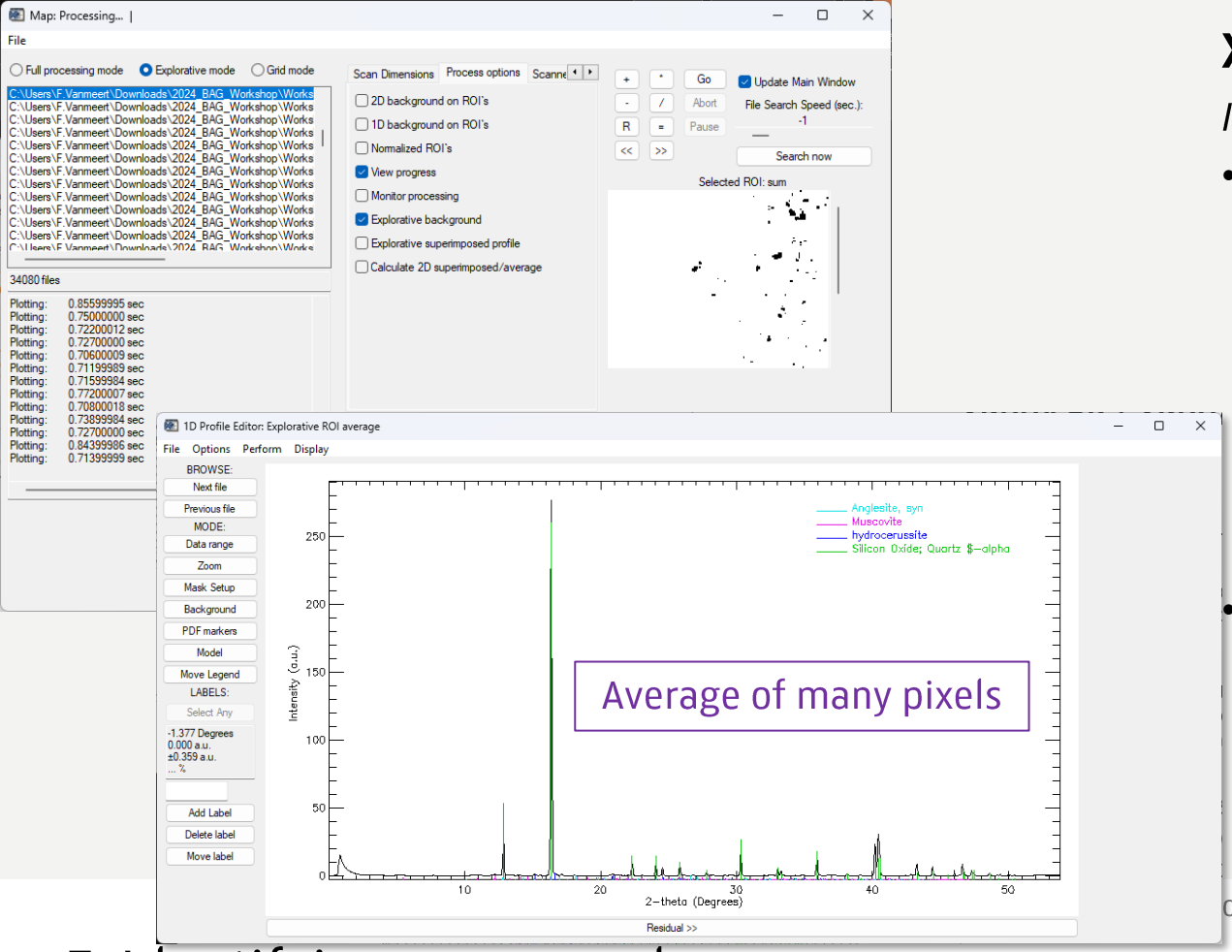

XRDUA BP + XRDUA 1D

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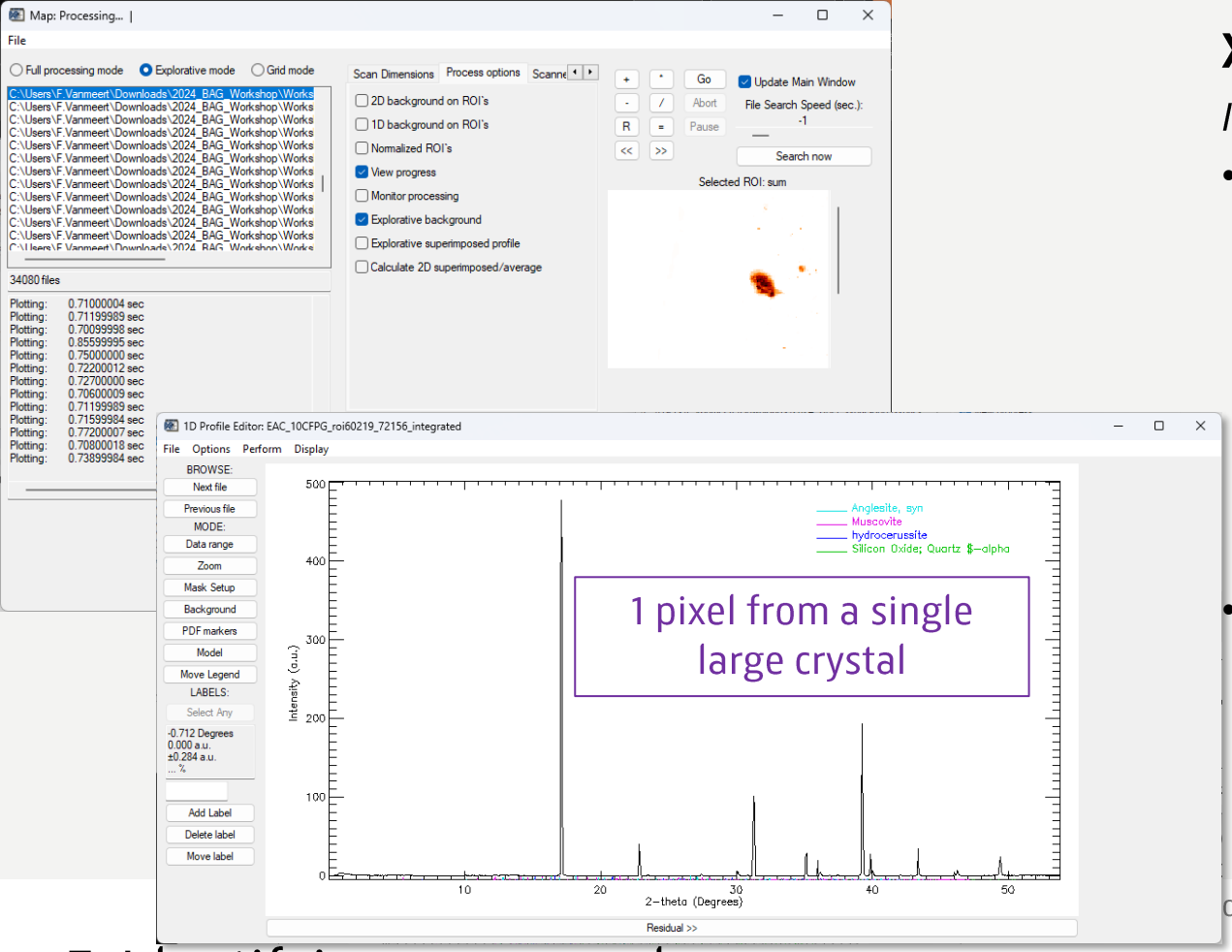

XRDUA BP + XRDUA 1D

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My strategy (depends on the goal of the analysis):

- Identify all main phases of the average profile, using as much prior information as you have.
- Use ROIs on peaks of an unknown phase to obtain a good 1D profile for that phase using **XRDUA BP** (single pixel or average). Identify it with available PDFs or with dedicated software (Match!, QualX2, …).
- Repeat this for all/most signals.
- Create a small ROI window in **XRDUA BP** and ‘scan’ this over the entire angular range and/or place ROI selectively on angles for which you expect a diffraction peak of a specific phase to check for meaningful 2D intensity distributions.
- Be aware of the limits of your data (preferred orientation, “large” crystals, …).
- Create your fitting model gradually.

XRDUA BP + XRDUA 1D

Identifying compounds

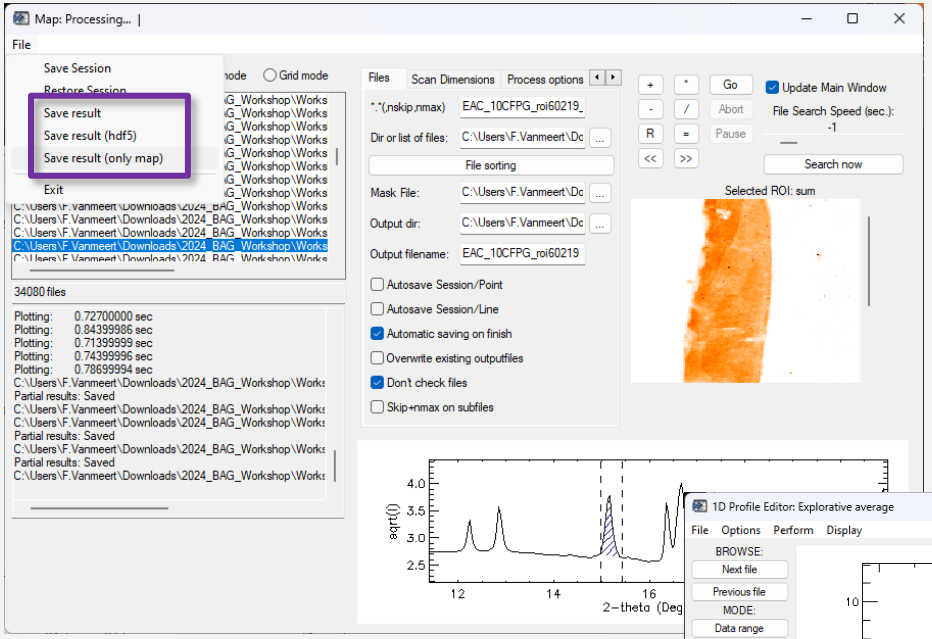
- This is the process that (usually) **requires the most time from the user.**
- A priori knowledge and complementary information are very helpful
- Sometimes it’s good to be stubborn and keep trying ;-)

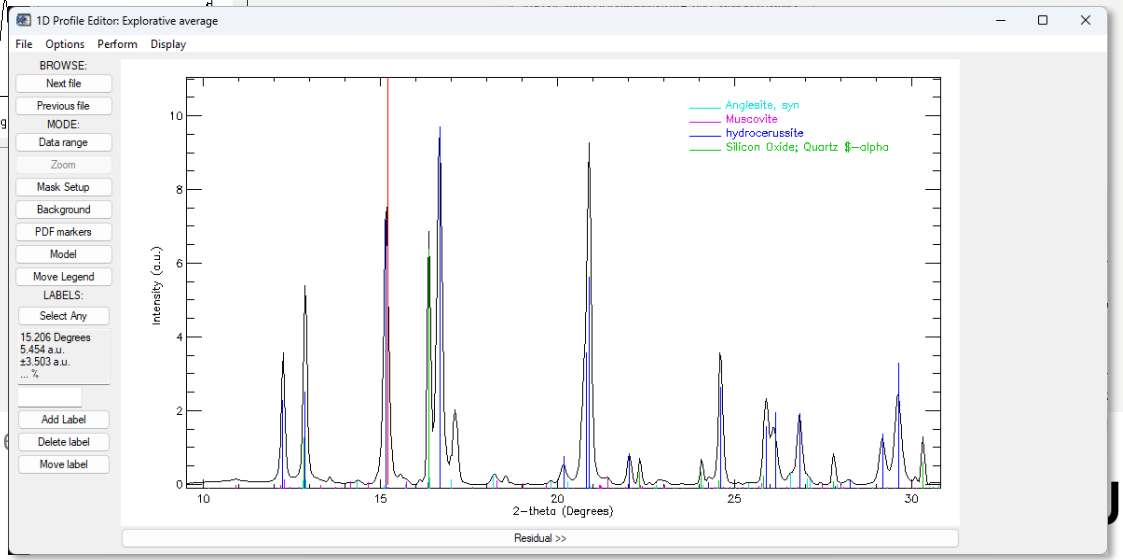
3. Identifying compounds

XRDUA BP + XRDUA 1D

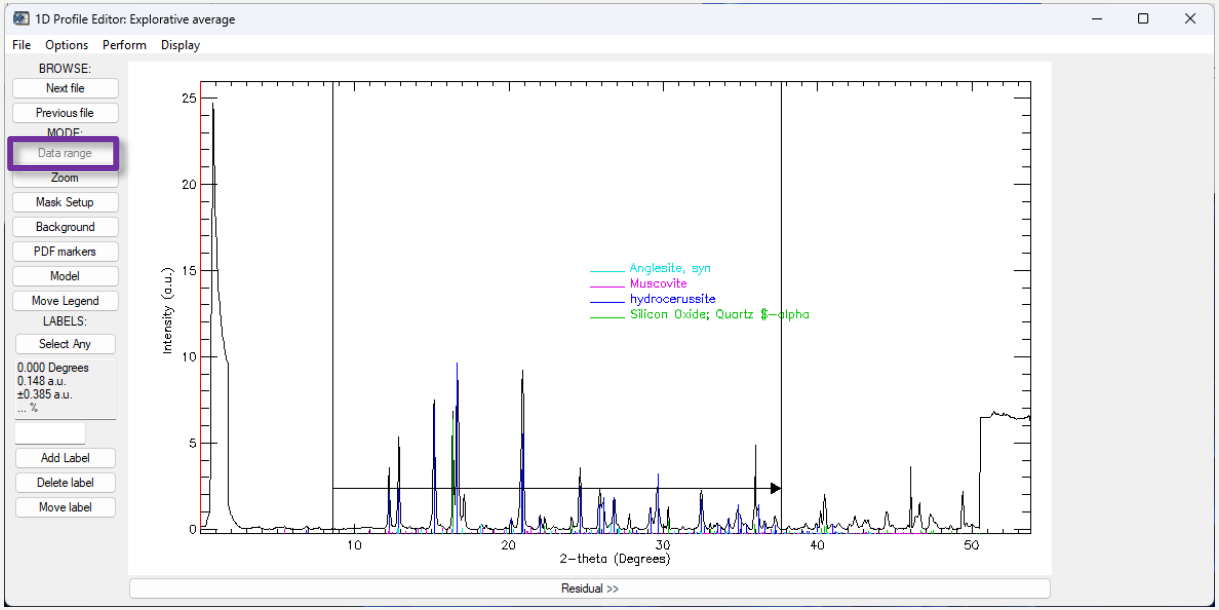
Saving ROI images

- Select ROI around peak of interest
- Check correct selection of peak in **XRDUA 1D** (when “Update Main Window” is checked)
- Save result (only map)





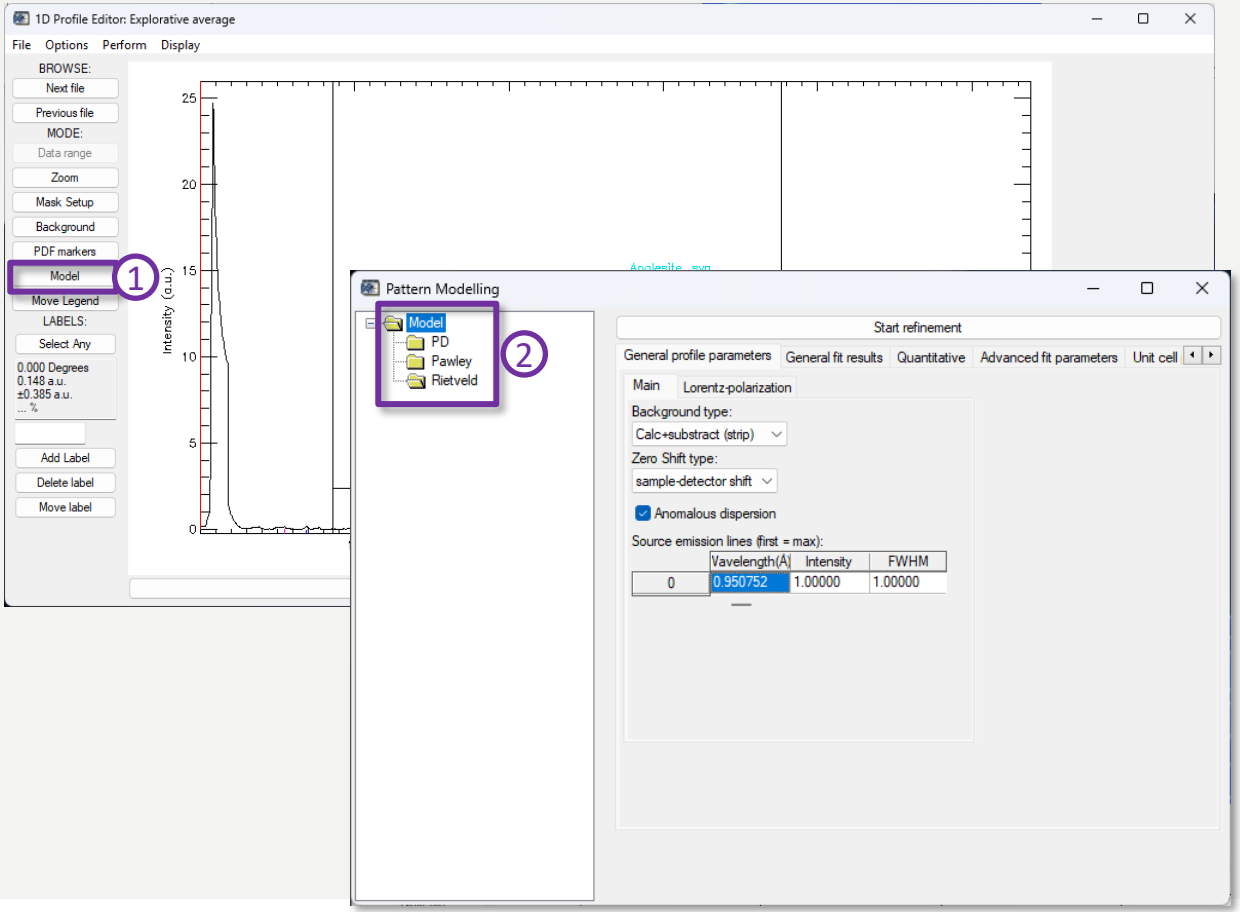
4. Saving ROI images



XRDUA 1D

Select Data Range

- Select the data range that you want to include in your fit model
- Typically:
  - Large scattering angles produce signals of low intensity of there is a lot of overlap with other phases
  - Low scattering angles show the drop in intensity of the beam stop and the decrease in intensity of the primary beam and beam scatter
  - If your phases of interest are not in those regions, avoid putting them in your model.



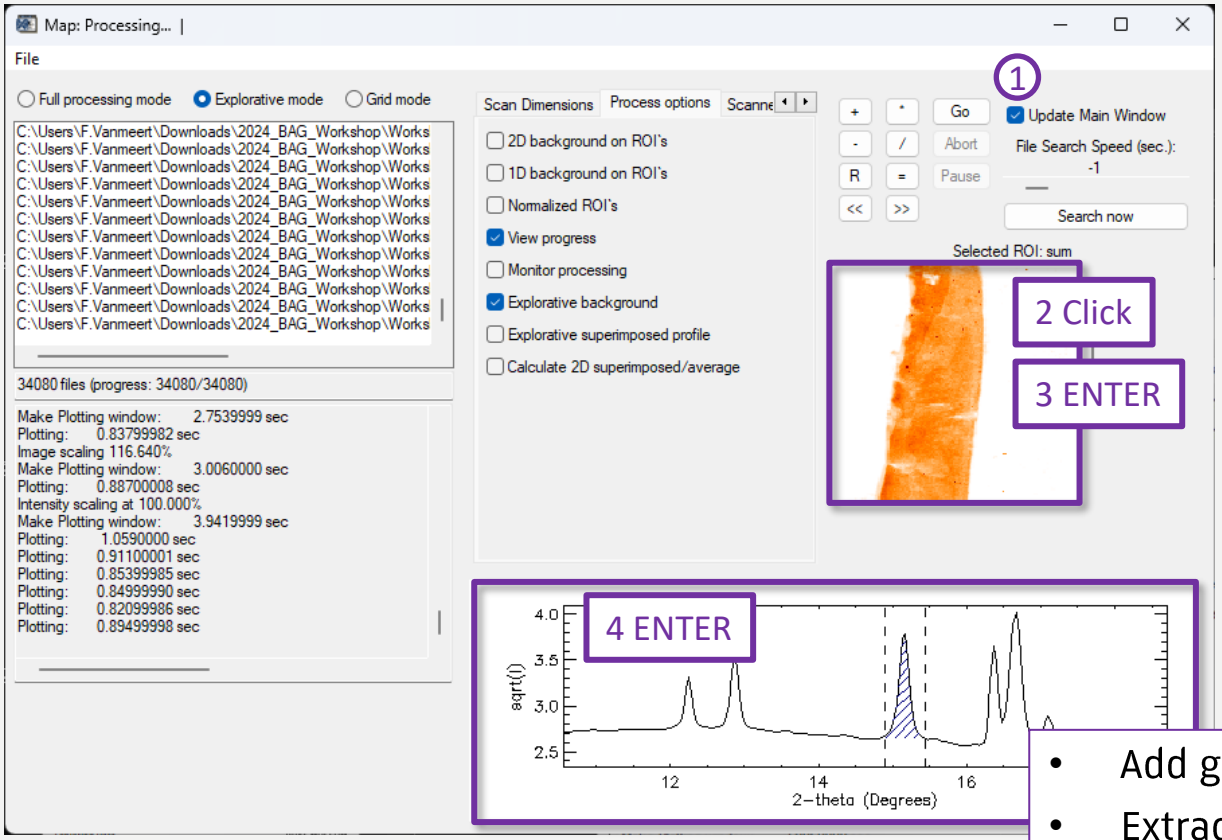
XRDUA 1D + Model

Main Model window

1. Model
   - General fit parameters
   - Default settings should be fine for most cases
   - Change Background type if you didn't use 'strip' background
2. Different 'structural' and 'structureless' groups to use in the fitting model

convert

- **Rietveld**: uses complete crystal structure (unit cell parameters, atomic coordinates, SOF, …)
- **Pawley**: uses crystal structure information without atomic information
- **PD**: contains no structural information (list of peak positions, intensities and widths)



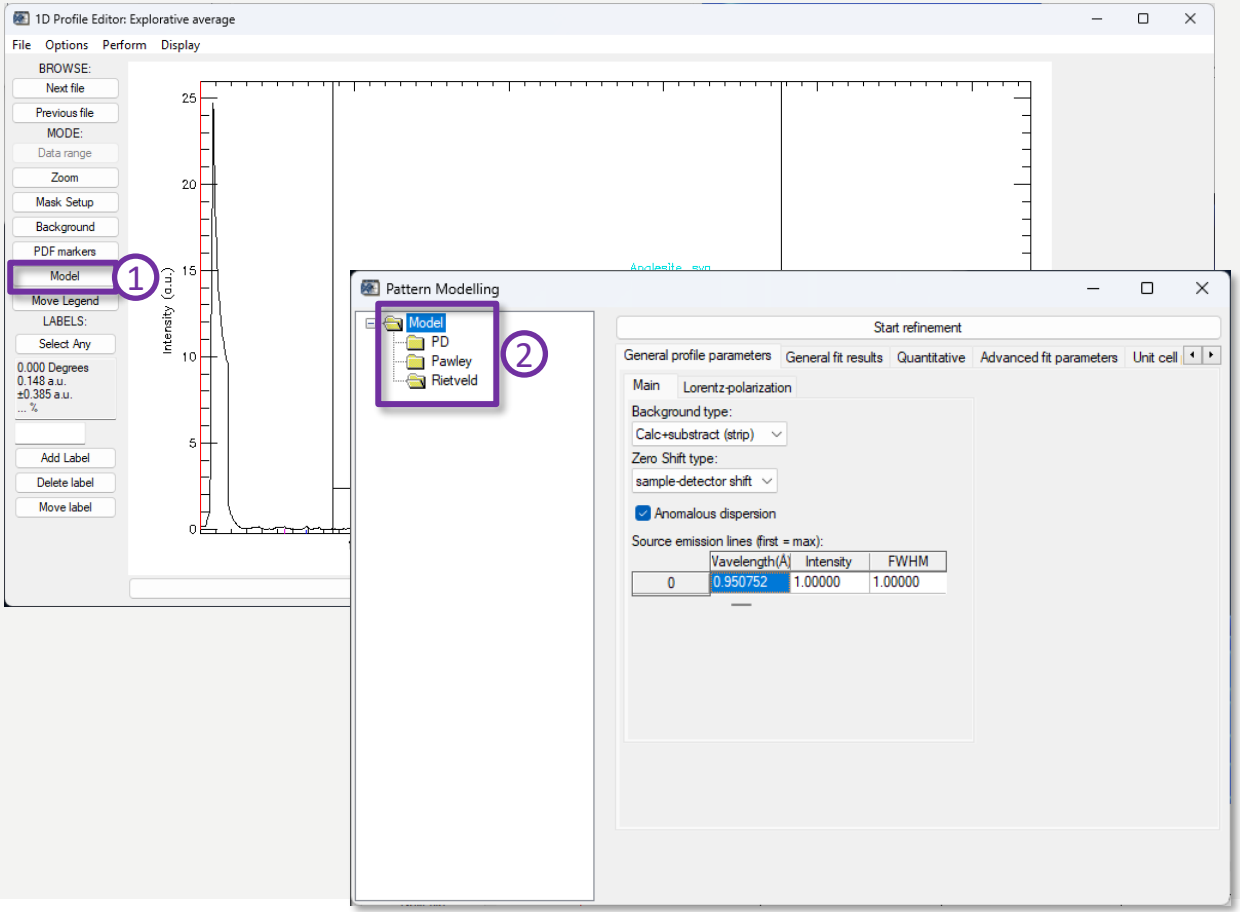
XRDUA BP

*Link to **XRDUA 1D***

1. Update Main Window
2. Click pixel in ROI image
   - Loads 1D profile of that pixel to **XRDUA 1D**
3. Press ENTER when cursor is in ROI image
   - Loads averaged (or superimposed) 1D profile based on current ROI intensity in **XRDUA 1D**
4. Press ENTER when cursor is in 1D profile
   - Loads averaged (or superimposed) 1D profile in **XRDUA 1D**

- Add groups to the model step by step
- Extract a suitable 1D profile in which the phase(s) of interest is/are clearly present.
- Use contrast/intensity scaling when needed to visualize the distribution.

5. Creating a Fit Model



XRDUA 1D + Model

Main Model window

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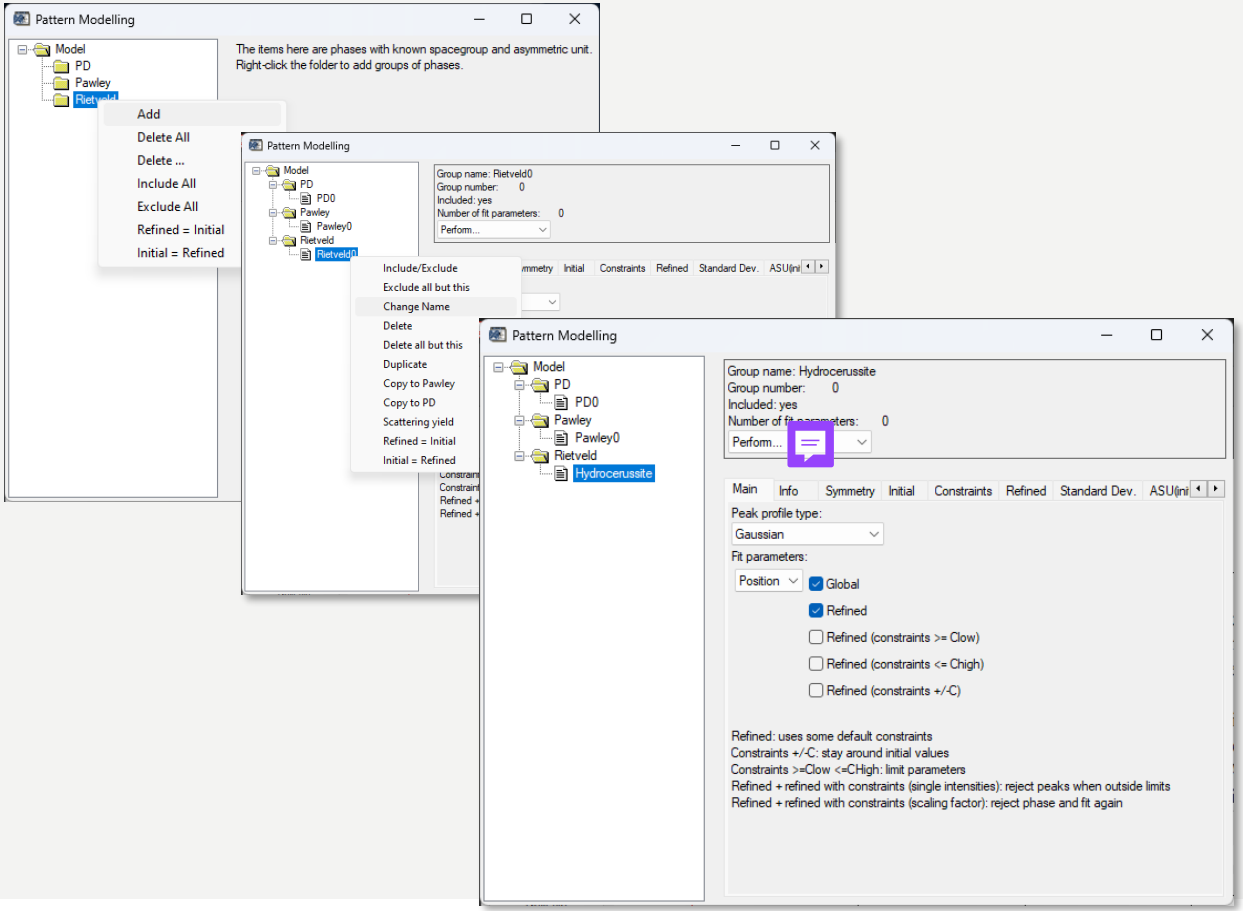
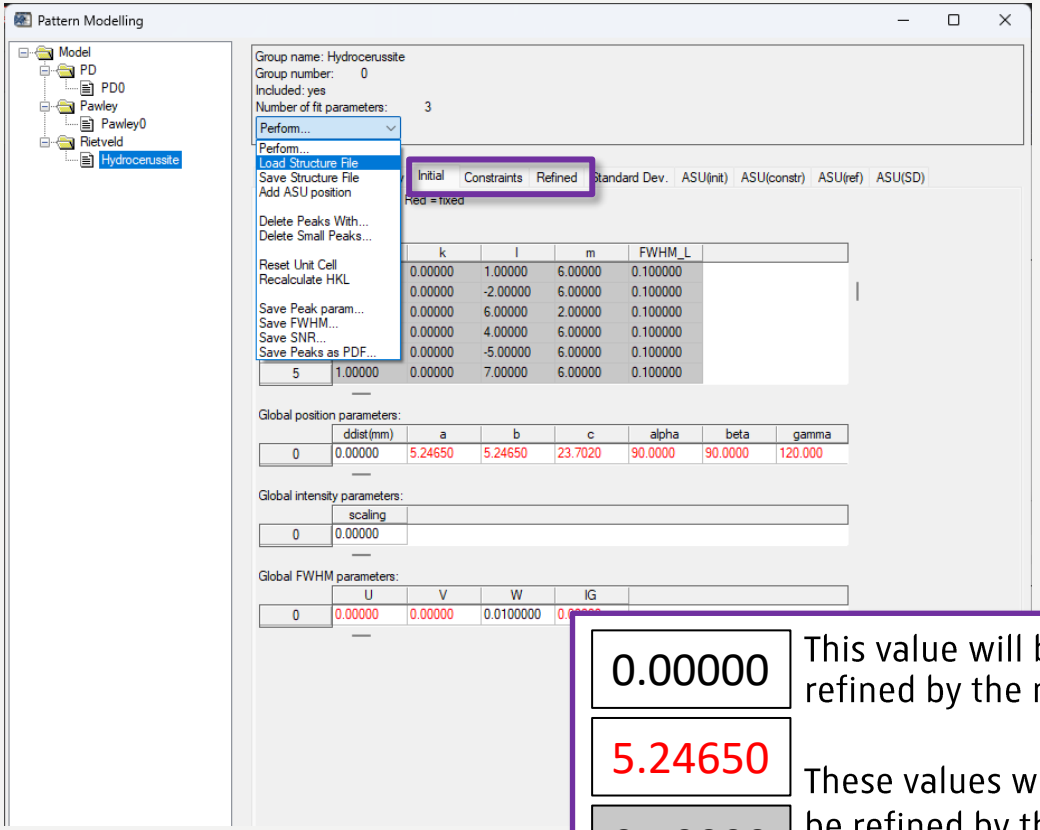


Model

Working with fitting groups

- Right-click on the group for options
- Add ‘peak group’ to different structural groups
- Change name of ‘peak group’



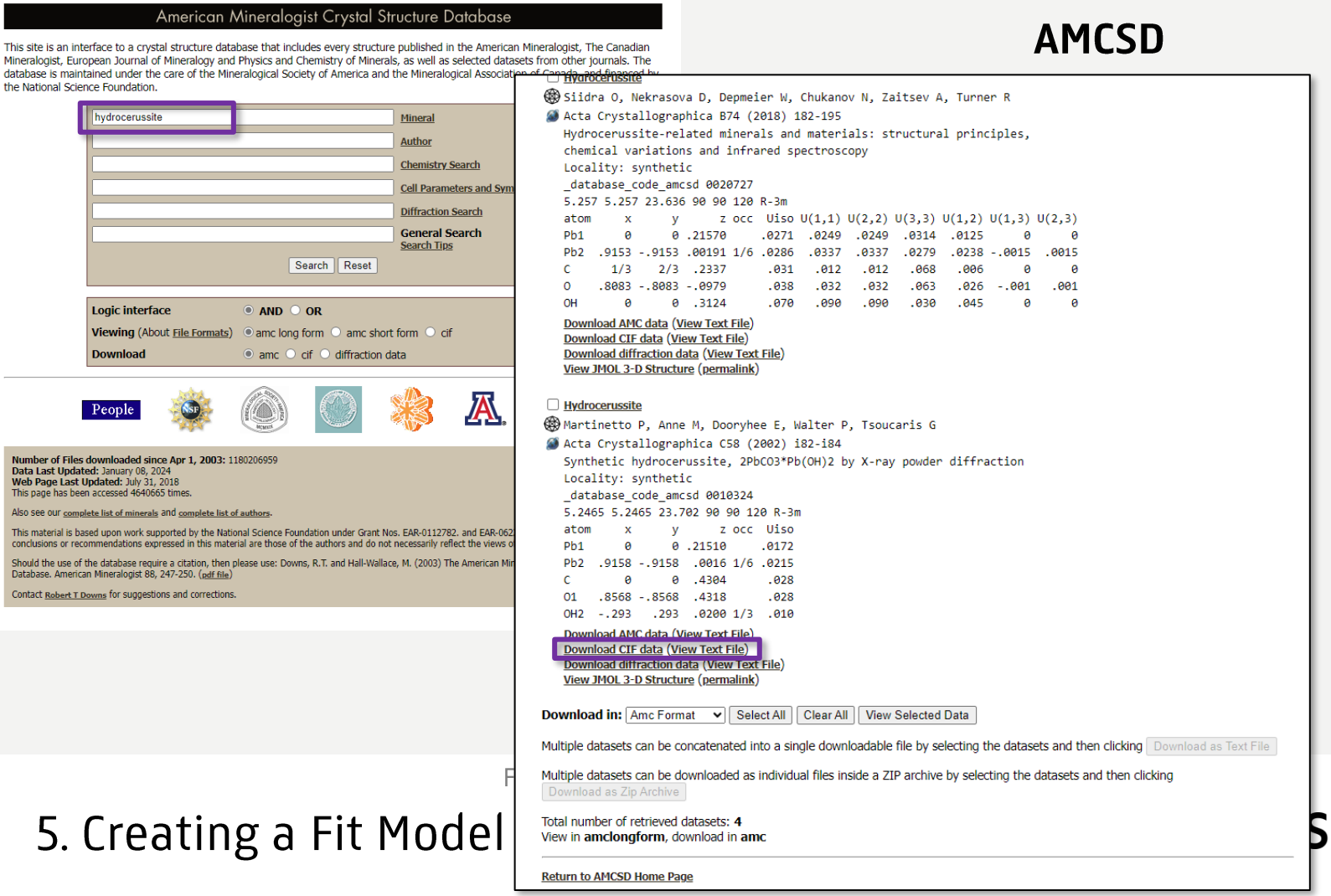
0.00000	This value will be refined by the model
5.24650	These values will NOT be refined by the model
0.10000	

Model

Rietveld Group

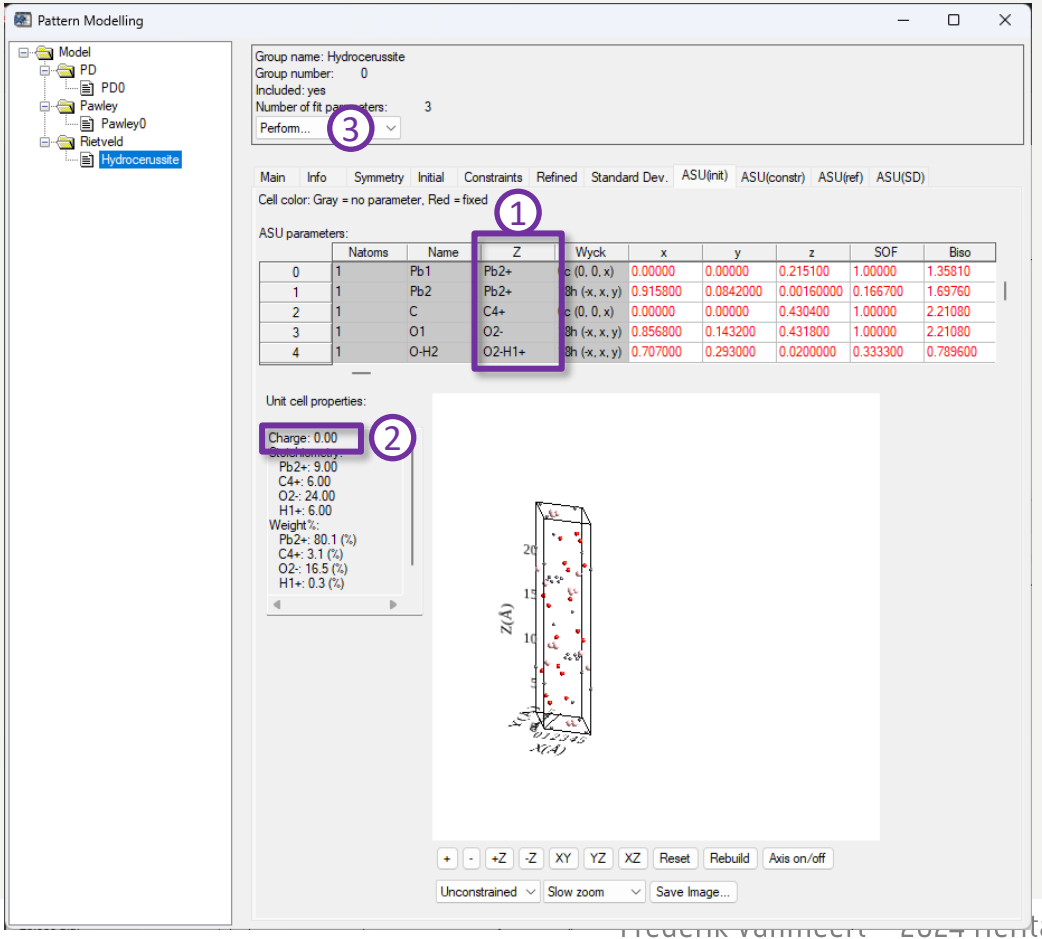
- Load Structure from structure file
  - E.g., cif files from COD or AMCSD
  - .cel from XRDUA
- Initial
  - Shows miller indices, multiplicity of the different diffraction peaks within the selected data range
  - Unit cell parameters
- Constraints
  - Set constraints on the parameters that are refined
- Refined
  - Refined values obtained by the model

AMCSD



5. Creating a Fit Model

S MUSEUM



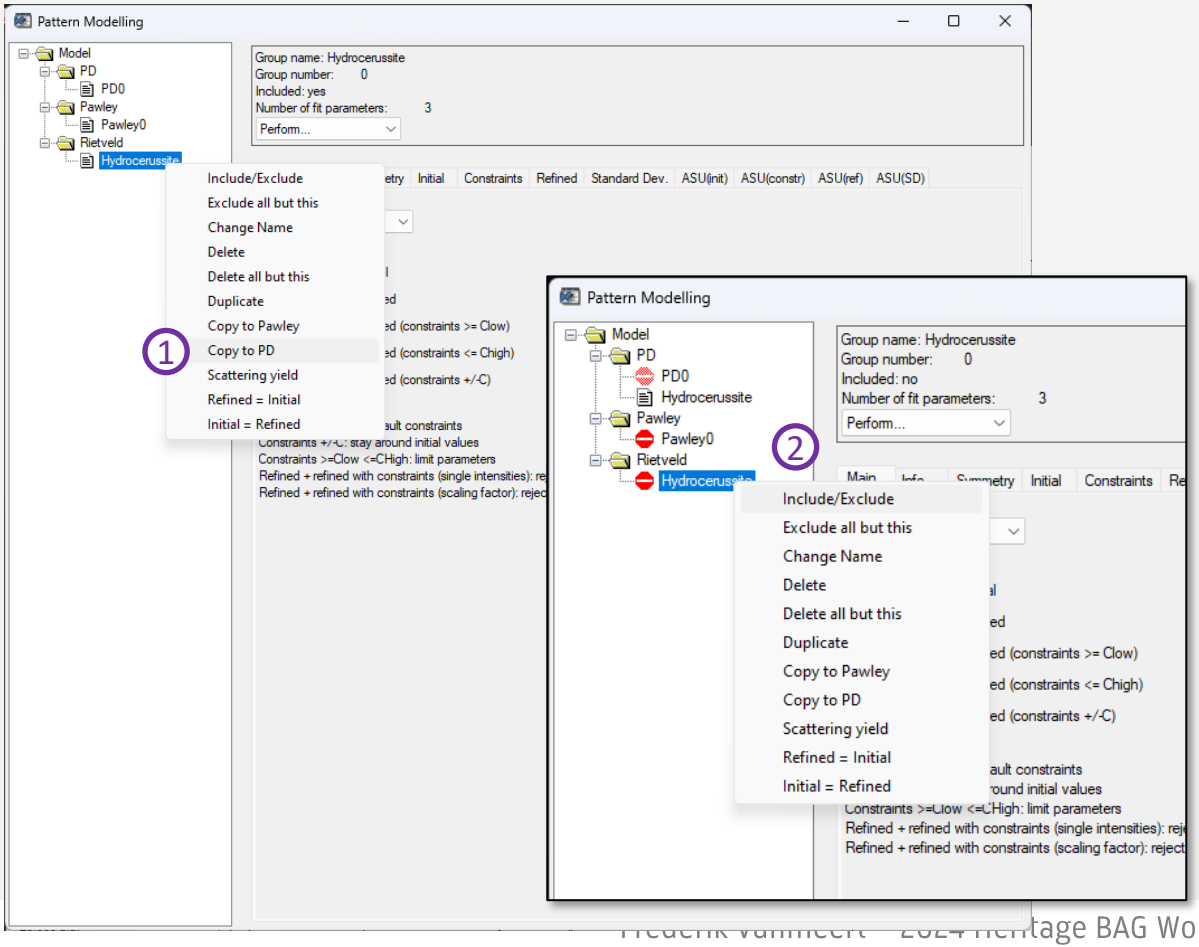
Model

Rietveld Group

- ASU(init)
  - Atomic structure information
  - Charges are default, unless the structure is loaded from a XRDUA .cel file
- Set proper charges
  1. Z, e.g.,
     - Pb: Pb2+
     - OH-: O2-H1+
     - H2O: O2-H1+2
  2. Charge: 0.00
3. You can modify and save the structure as a .cel file (Perform ...)

5. Creating a Fit Model

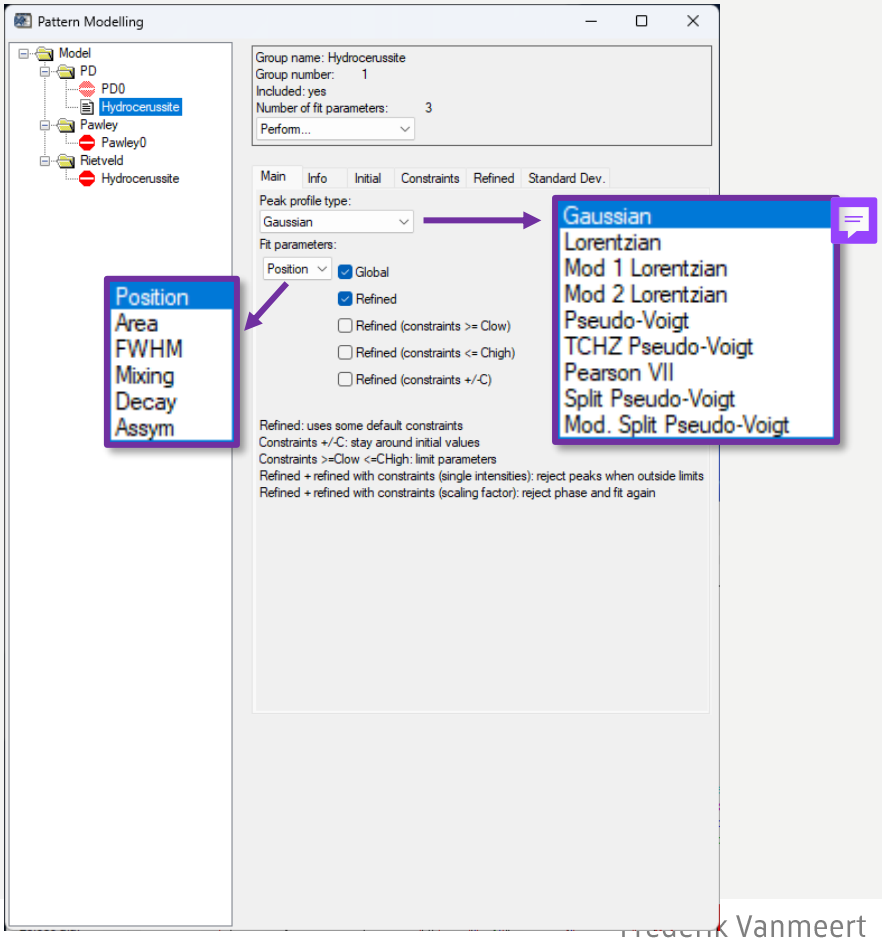
RIJKS MUSEUM



Model

Convert Rietveld to PD

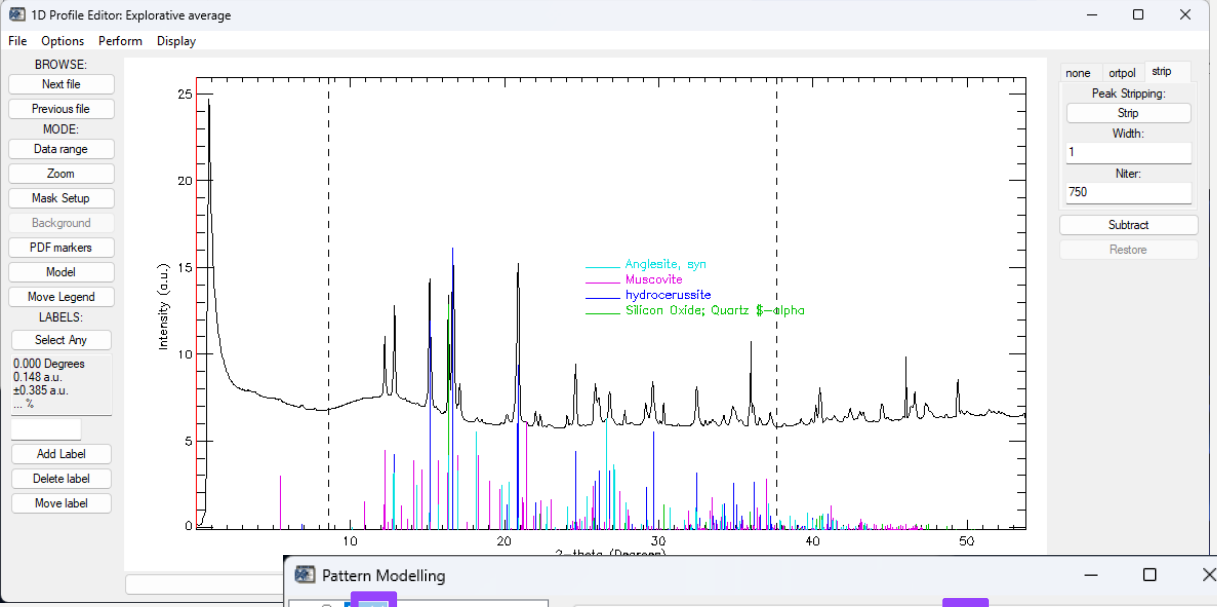
1. Peak positions and relative intensities are calculated based on the structural information in the Rietveld model. To speed up the fit, convert the Rietveld group to a PD group (i.e., a list of positions, relative intensities and widths).
2. Exclude irrelevant groups and the Rietveld group from the fit

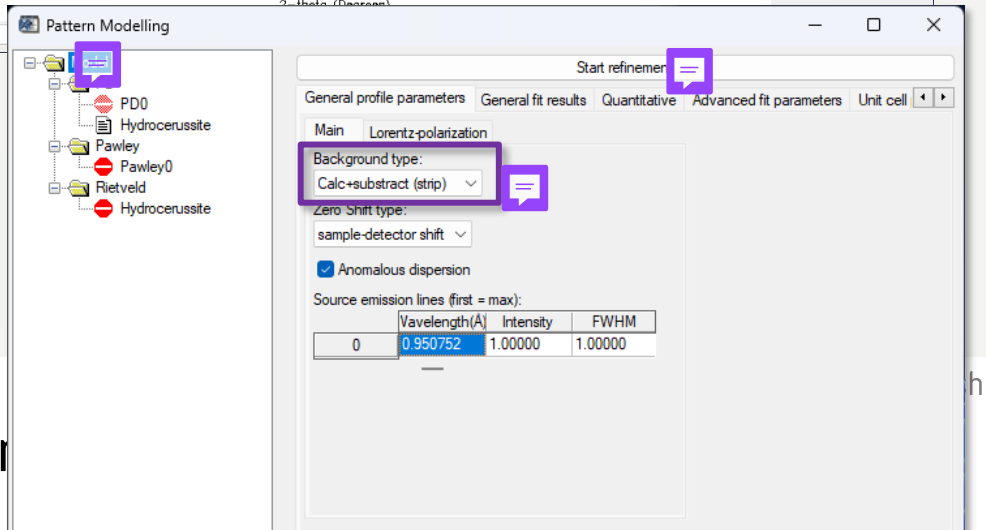


Model

Main group window

- Chose Peak profile type
- Select which parameters to refine
- 'Global' to link various parameters during refinement
  - Positions: are refined by a single parameter 'sample-detector distance')
  - Area: intensity is scaled using a single scaling factor
  - FWHM: widths are refined using the Cagliotti peak width function





XRDUA 1D + Model

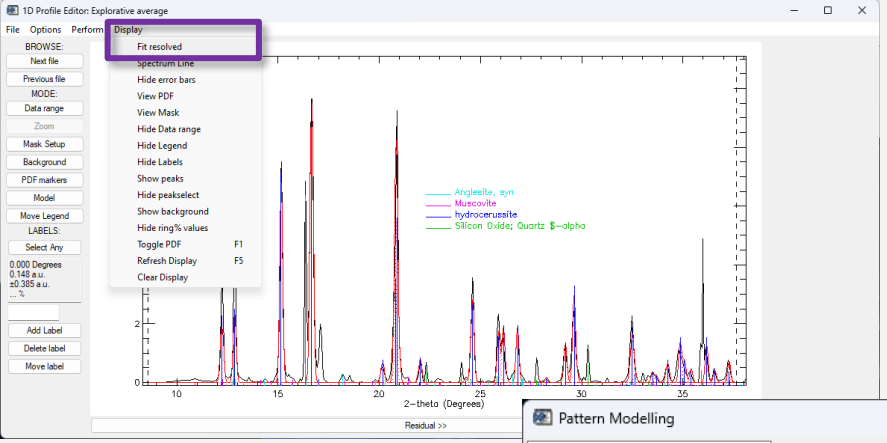
First Fitting

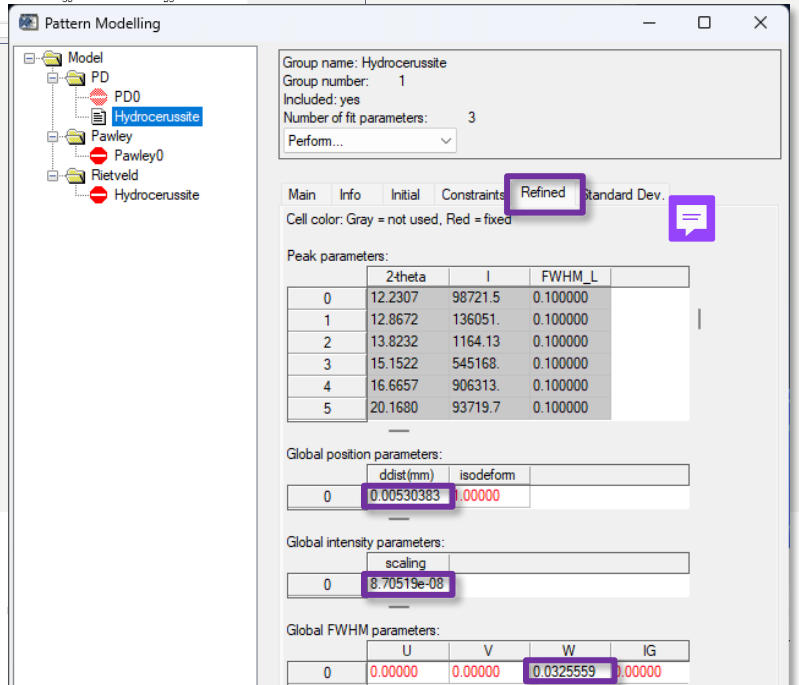
- Make sure to restore the background in **XRDUA 1D**, since the main model window has ‘Background type: ‘Calc+subtract (strip)
- Start refinement

5. Creati

hop

RIJKS MUSEUM





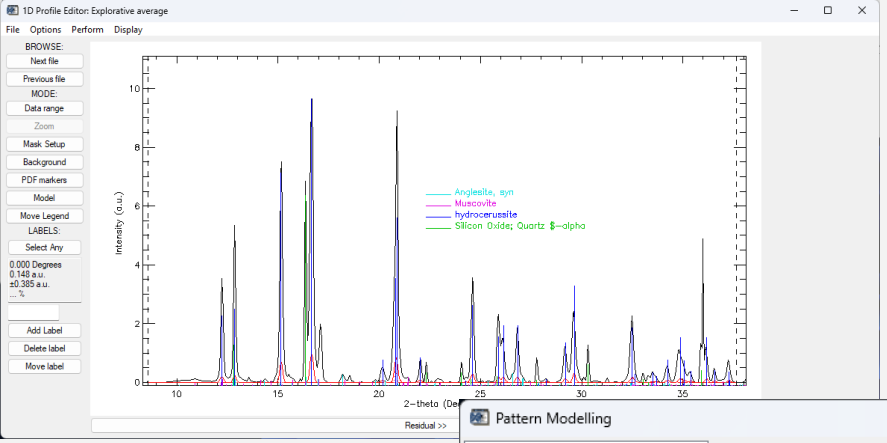
XRDUA 1D + Model

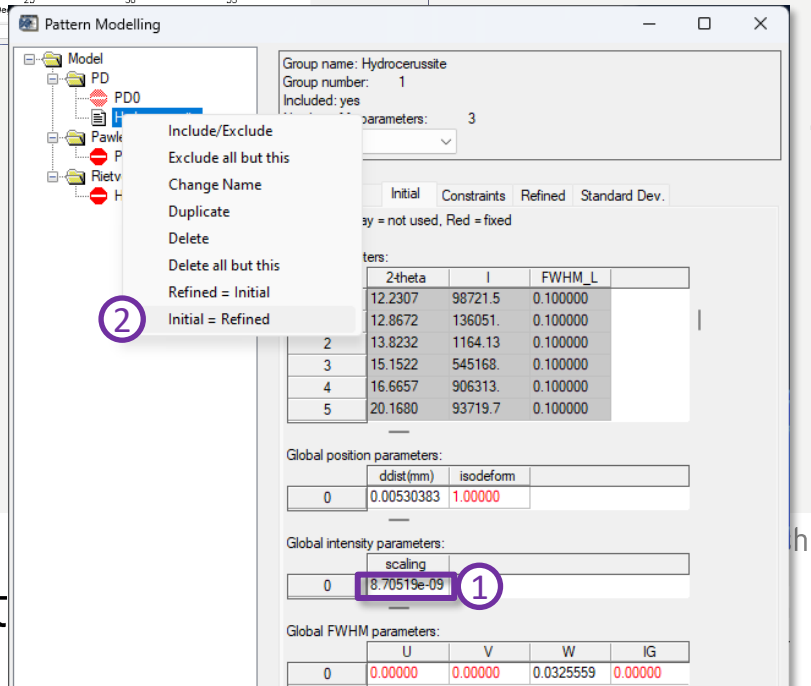
Find good starting values

1. Subtract background agai
2. Click ‘Fit Total’ (changes to ‘Fit resolved’)
3. The refined tab shows the refined values for the different parameters
   - ddist: offset to sample-detector distanc
   - scaling
   - W: Cagliotti theta independent peak width parameter

5. Creating a Fit M

RIJKS MUSEUM





XRDUA 1D + Model

Find good starting values

- Check the refined values (**Model**) and the fit (**XRDUA 1D**)

1. Decrease the value for scaling. This value is too high as a starting value for pixels that do not contain this phase
2. Copy the refined values to the initial values for the model

hop

5. Creating a Fit

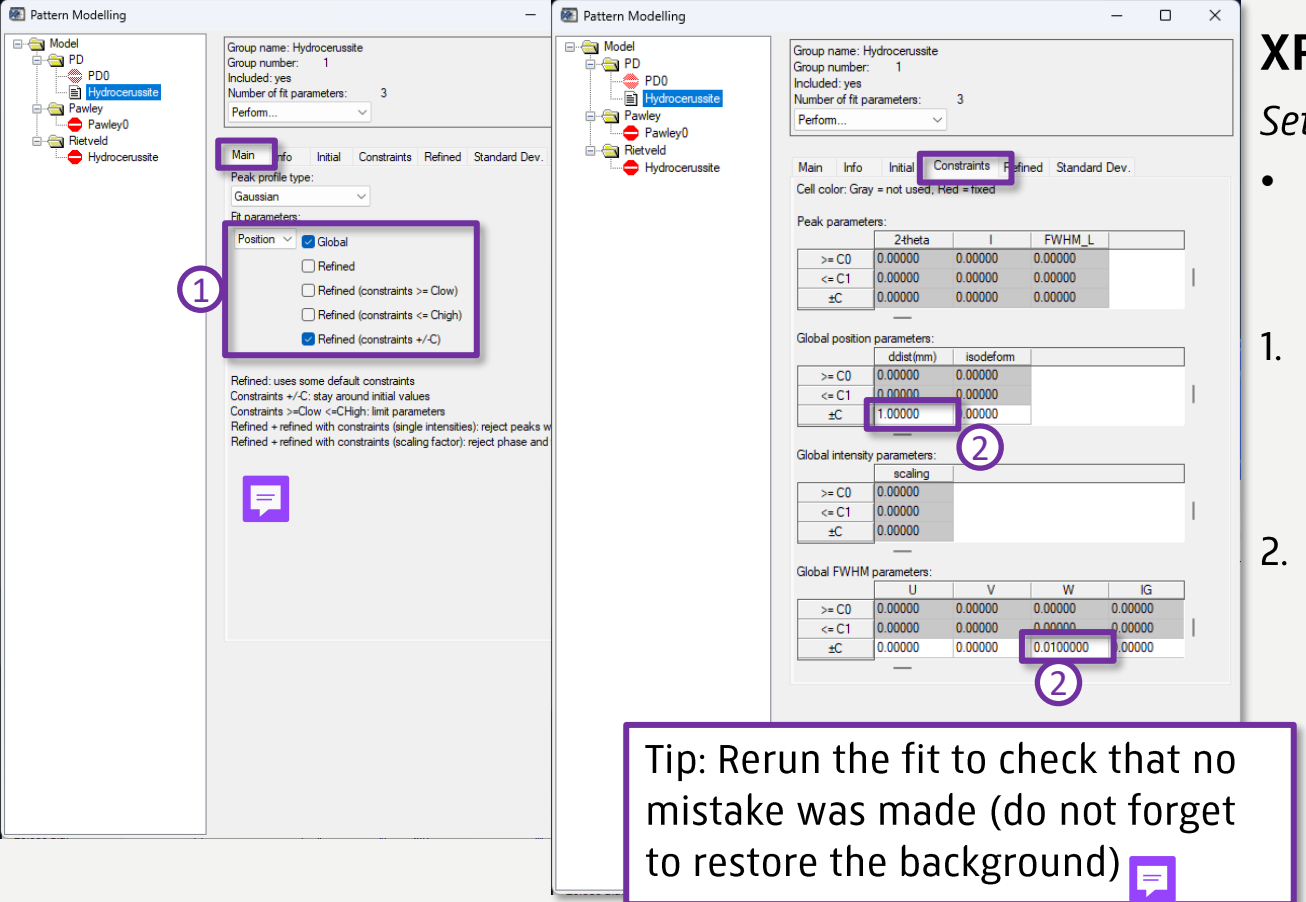
RIJKS MUSEUM

XRDUA 1D + Model

Set constraints

- It's best to constrain the position and width to avoid unrealistic values

1. Refined (constraints +/-C)
   - This constraints the value based on the initial value and a constant
2. Set the constants to be used as constraints
   - This depends on your sample, but a good start is e.g.,
     - ddist: 1 mm
     - W: ca. 1/3 of initial value



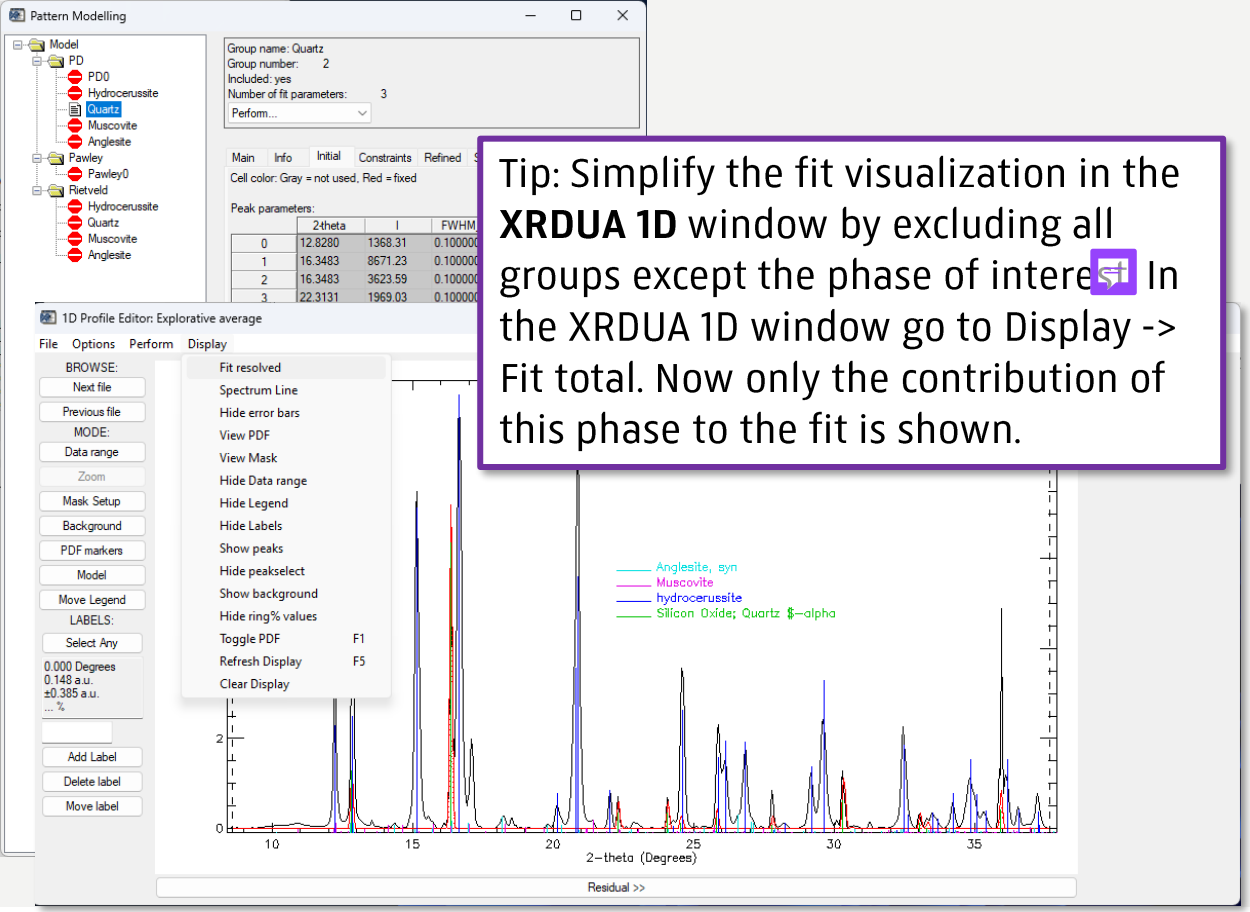
Tip: Rerun the fit to check that no mistake was made (do not forget to restore the background)

XRDUA 1D + Model

Add a new group for the next phase and repeat the procedure

1. Find a suitable 1D profile
2. Create Rietveld group
3. Load structure file
4. Check charges
5. Copy to PD
6. Start refinement
7. Check fit and refined values and lower the intensity value
8. Set Initial = Refined
9. Set constraints
10. Rerun fit

Tip: Simplify the fit visualization in the **XRDUA 1D** window by excluding all groups except the phase of interest. In the XRDUA 1D window go to Display -> Fit total. Now only the contribution of this phase to the fit is shown.

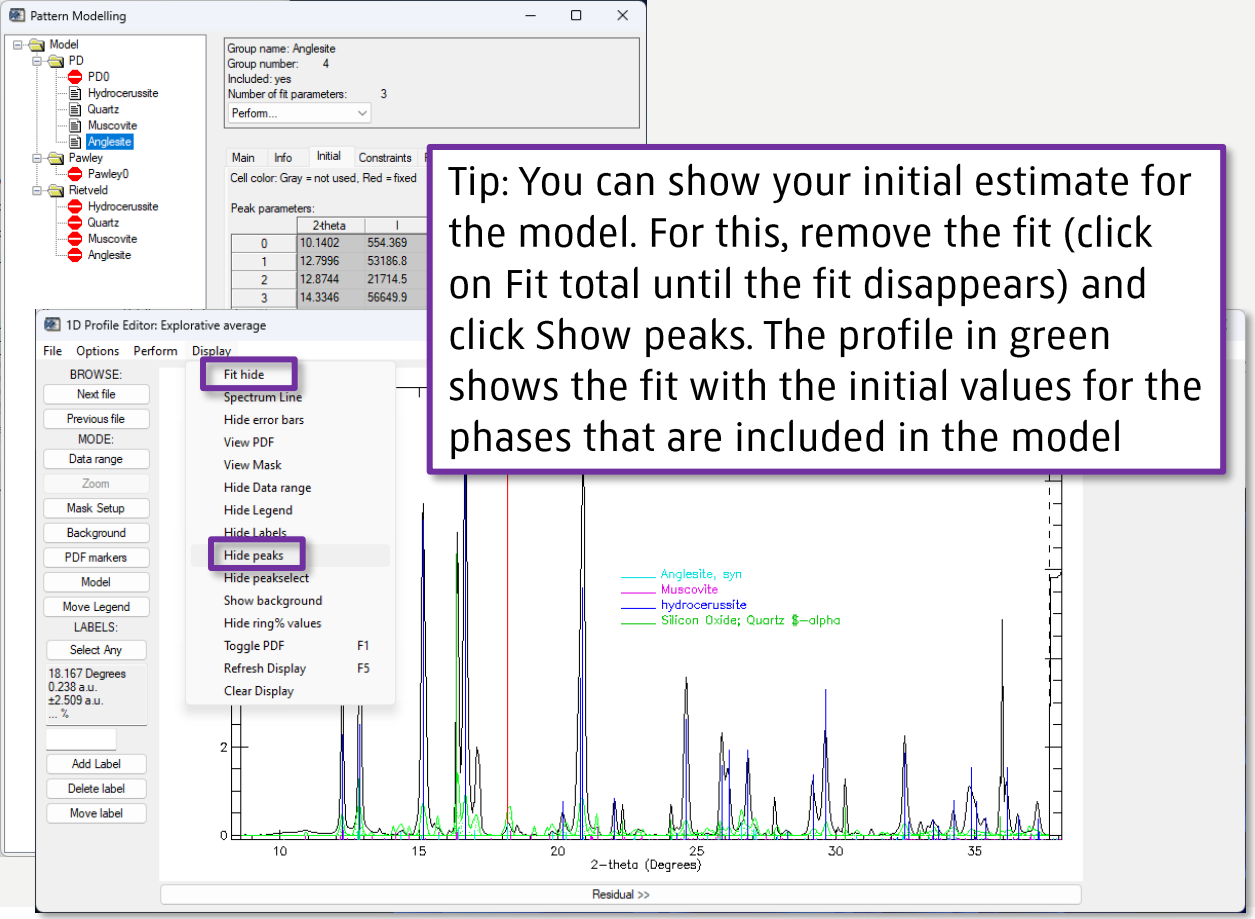


XRDUA 1D + Model

Add a new group for the next phase and repeat the procedure

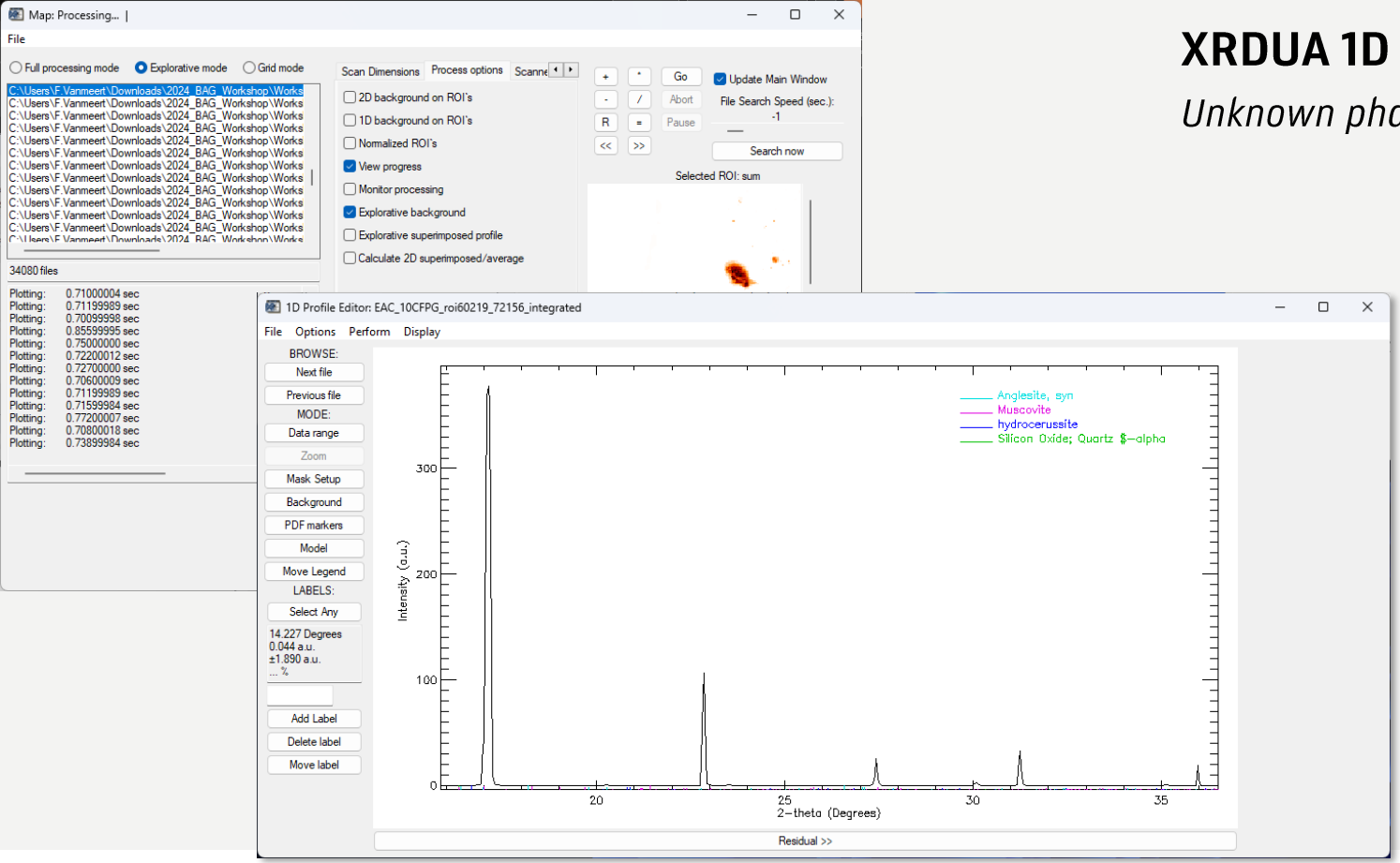
1. Find a suitable 1D profile
2. Create Rietveld group
3. Load structure file
4. Check charges
5. Copy to PD
6. Start refinement
7. Check fit and refined values and lower the intensity value
8. Set Initial = Refined
9. Set constraints
10. Rerun fit

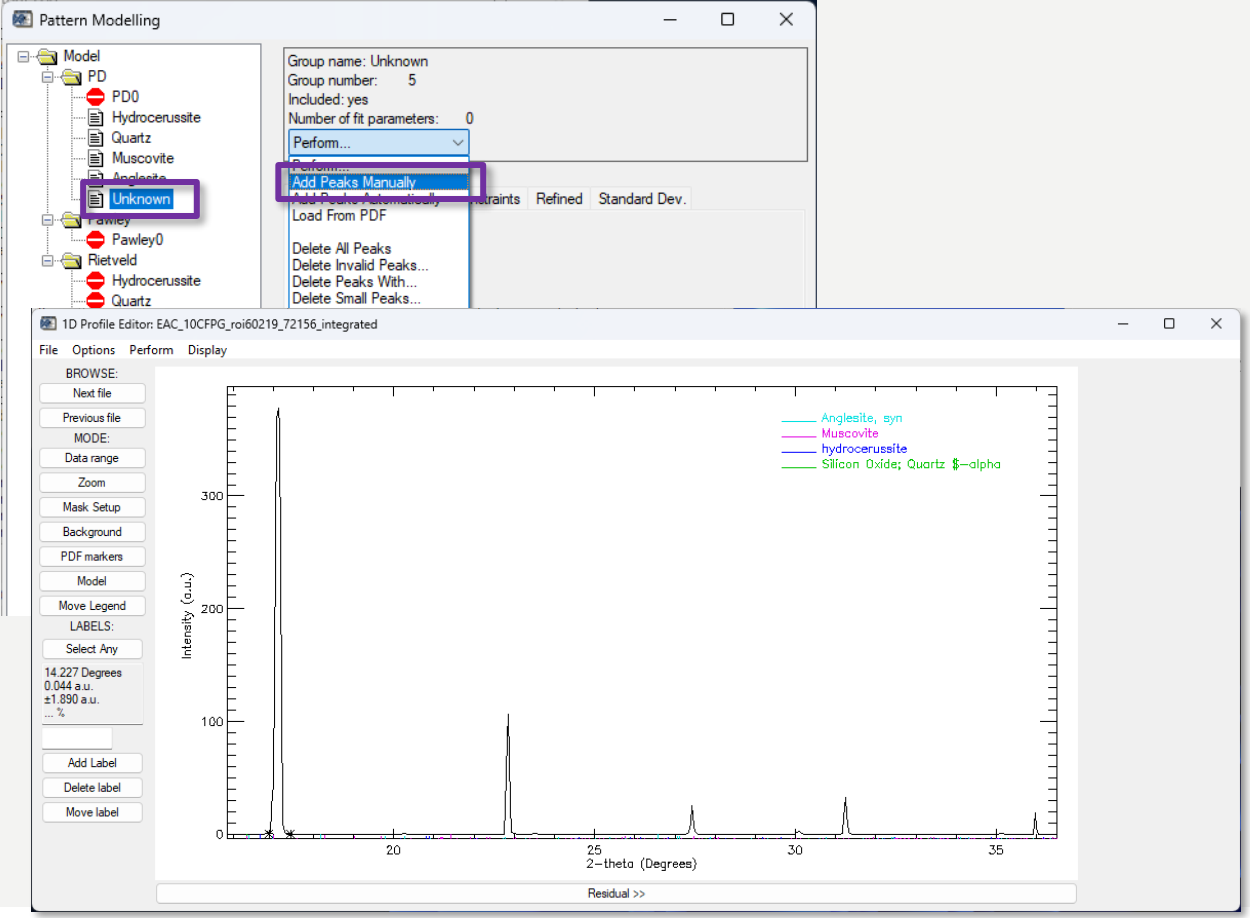
Tip: You can show your initial estimate for the model. For this, remove the fit (click on Fit total until the fit disappears) and click Show peaks. The profile in green shows the fit with the initial values for the phases that are included in the model



XRDUA 1D + Model

Unknown phase

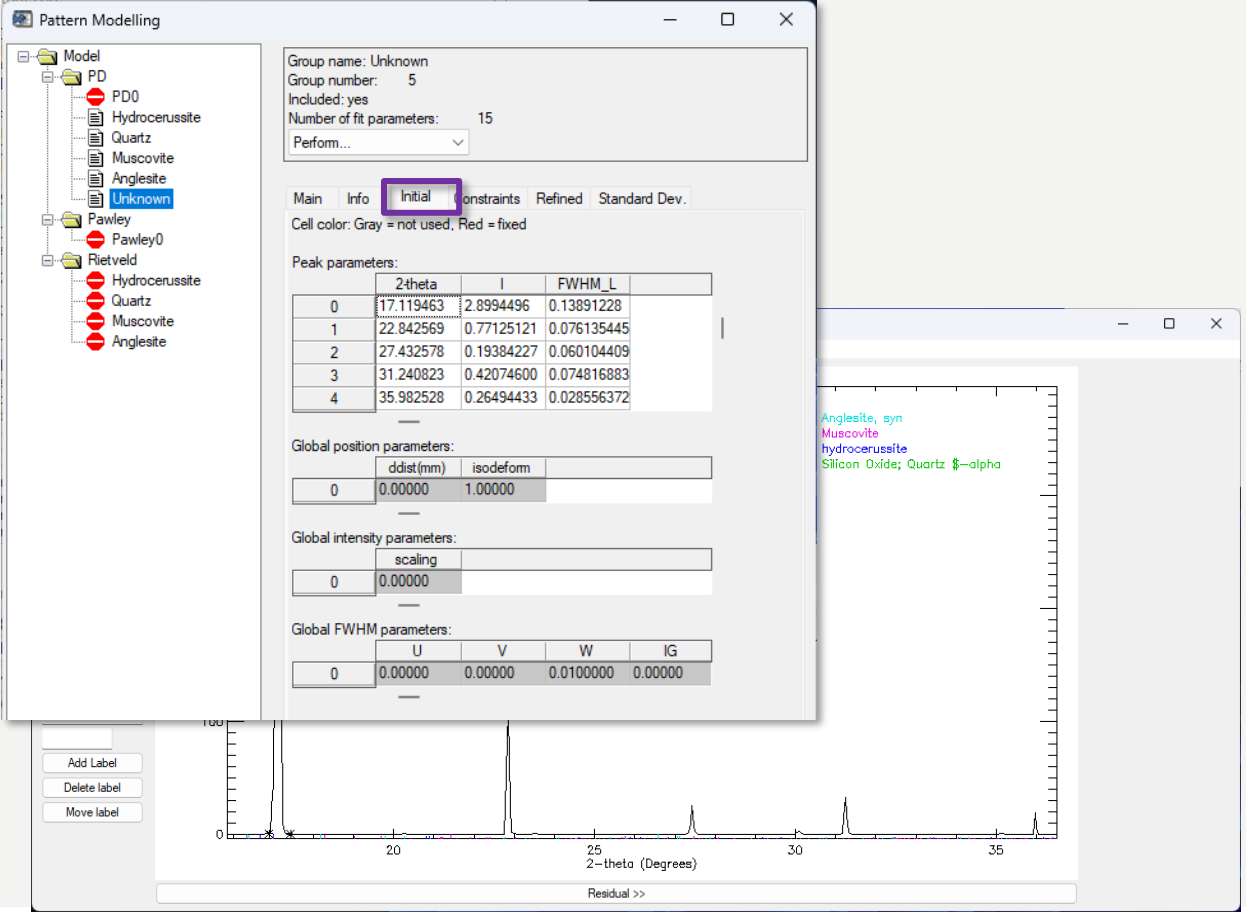




XRDUA 1D + Model

Unknown phase

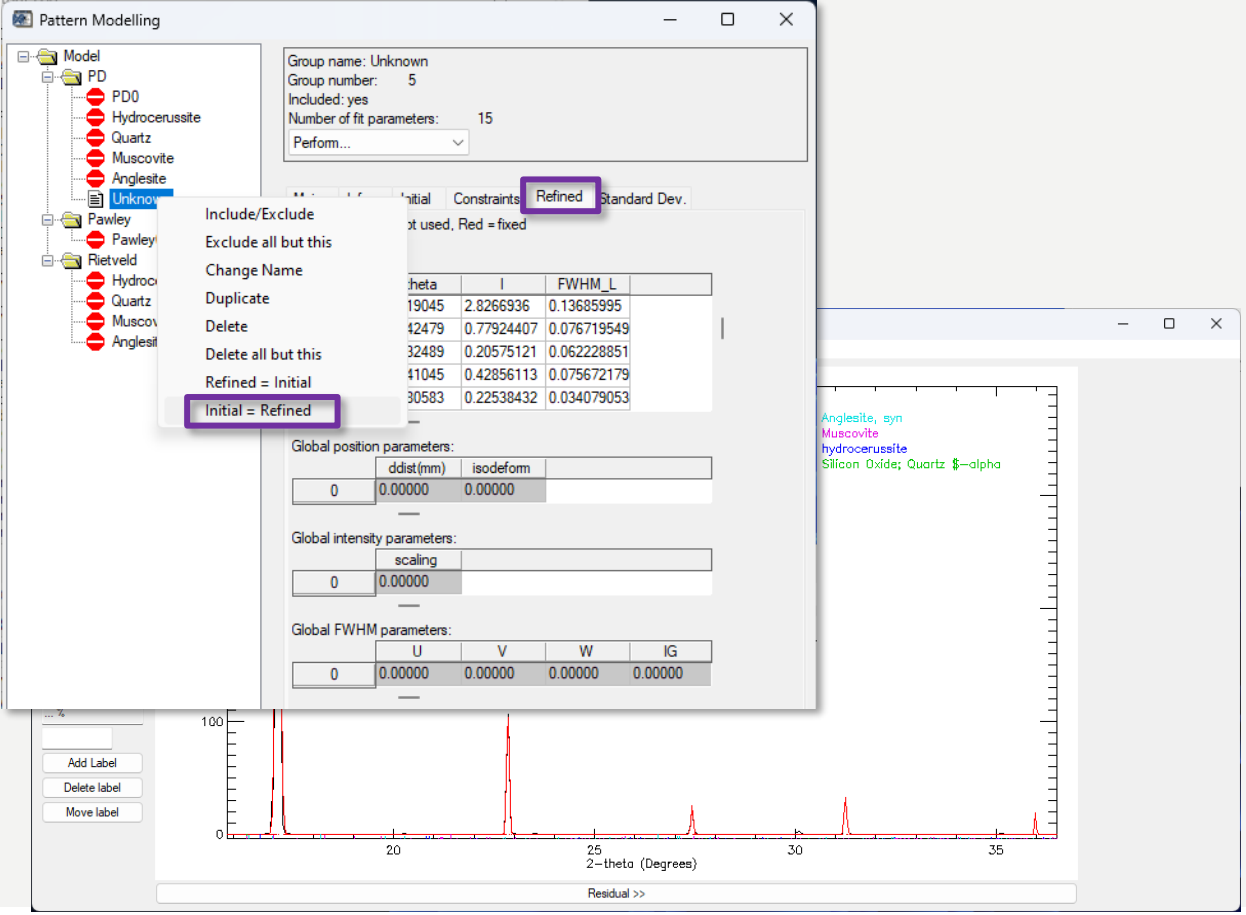
- Create a PD group (since no structural information is known)
- Add Peaks Manually and click and drag from left to right over the peak to add



XRDUA 1D + Model

Unknown phase

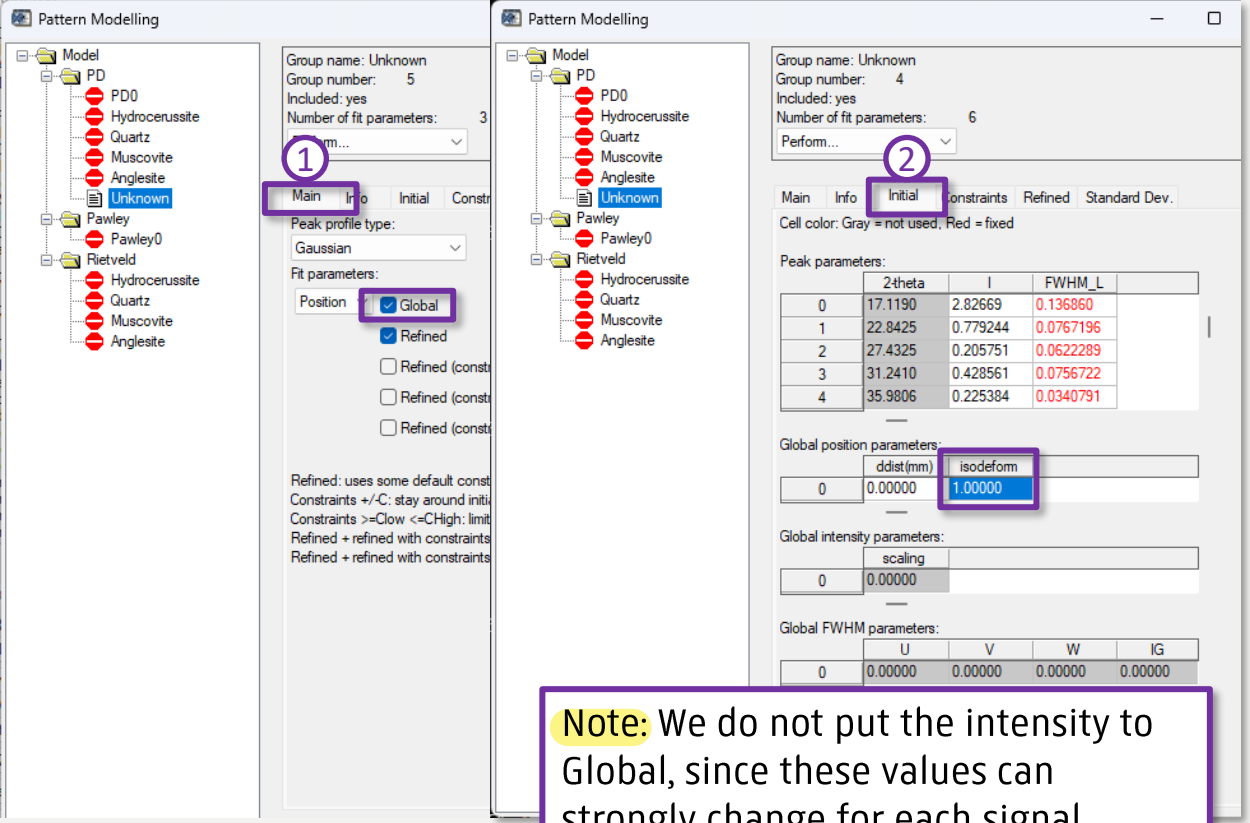
- Create a PD group (since no structural information is known)
- Add Peaks Manually and click and drag from left to right over the peak to add
- Under the initials tab, you will see the peaks being added with a position, intensity and width



XRDUA 1D + Model

Unknown phase

- Create a PD group (since no structural information is known)
- Add Peaks Manually and click and drag from left to right over the peak to add
- Under the initials tab, you will see the peaks being added with a position, intensity and width
- To get better starting values for the fit, start the refinement and check if the fit looks good.
- Perform Initial = Refined



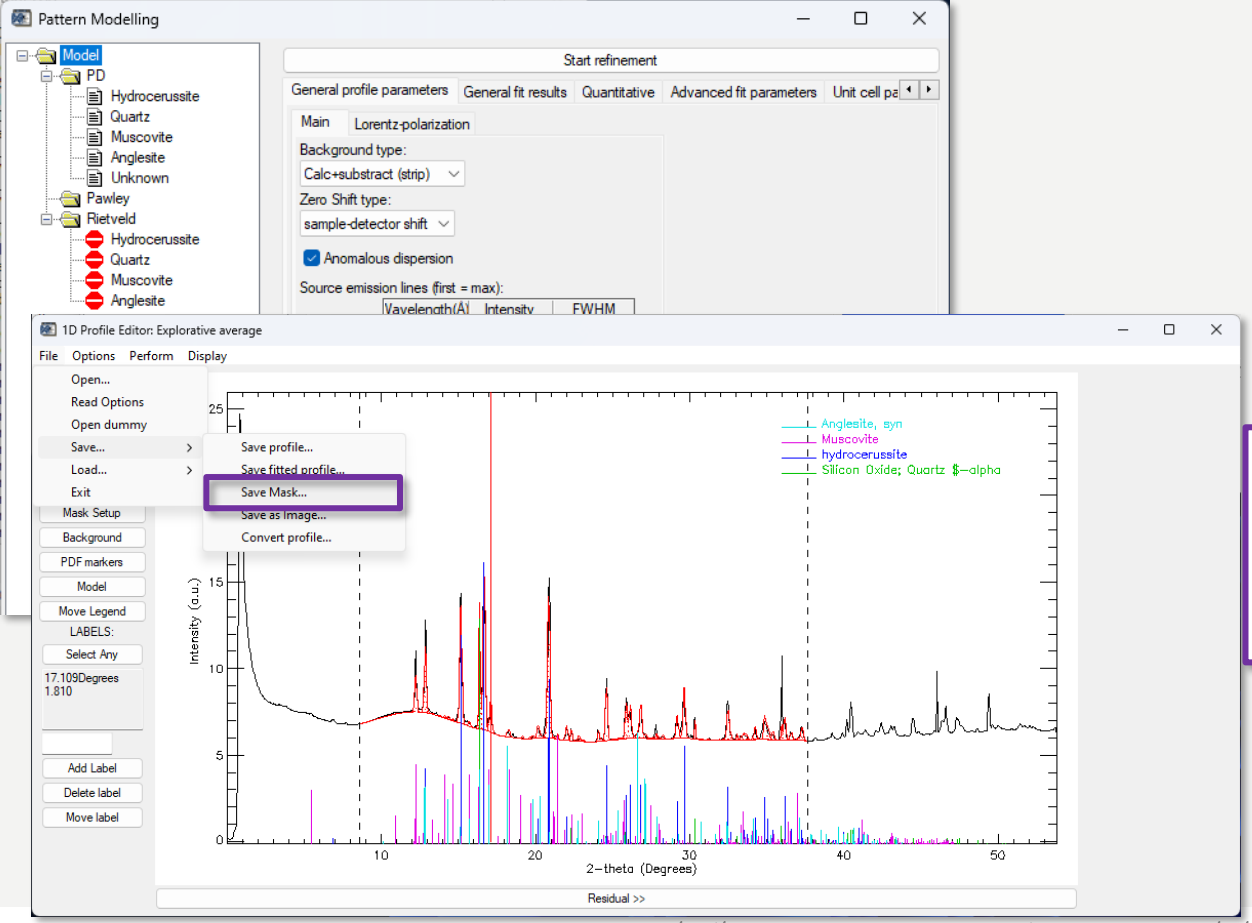
Note: We do not put the intensity to Global, since these values can strongly change for each signal depending on the hotspot intensity.

XRDUA 1D + Model

Unknown phase

1. Since the peaks selected here belong to the same phase, we want to set the Position to Global. I do not refine the individual FWHM → Deselect Refined for FWHM)
2. In the Initial tab, set isodeform to 1

- Start refinement and check fit
- Set Initial = Refined again to update the initial parameters
- Set constraints on Positio
- Rerun fit to check for mistakes ;-)

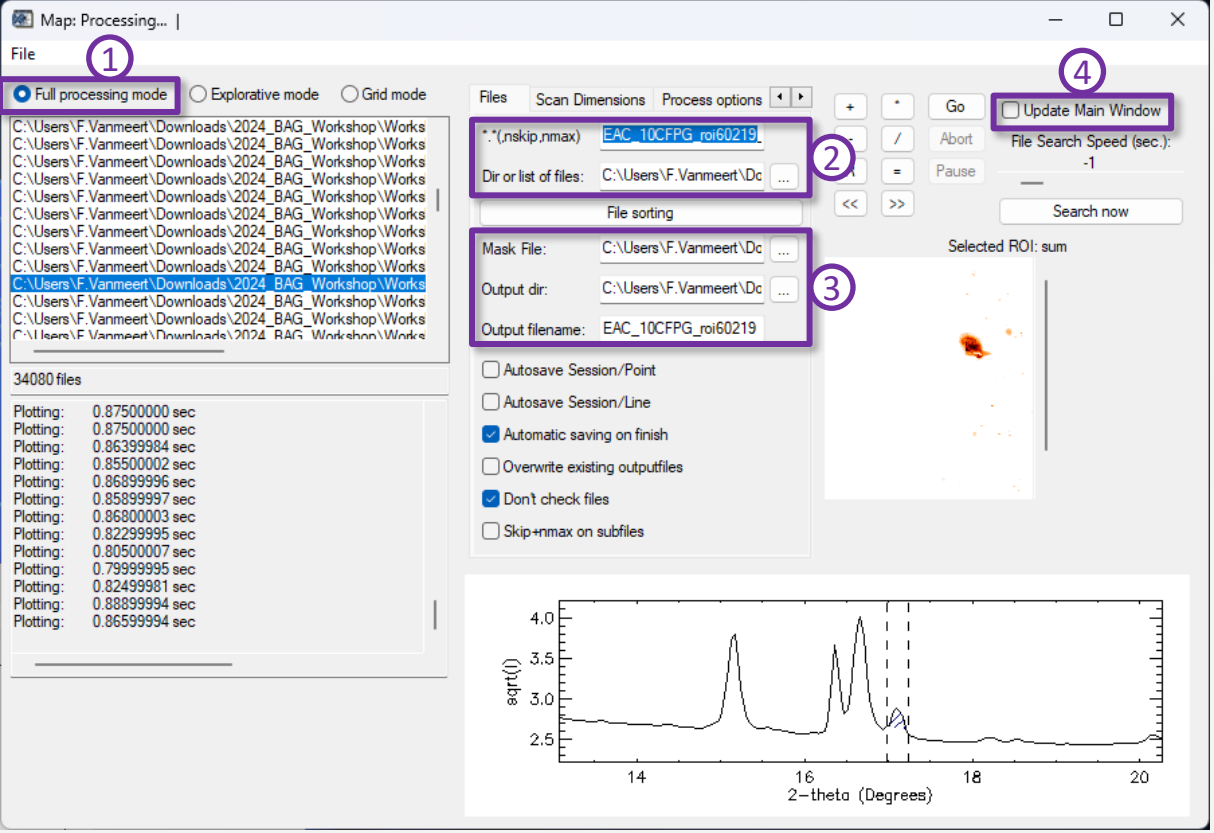


XRDUA 1D + Model

Finally ready with the model

- Restore the background
- Make sure only the relevant groups are included
- Save the model

Tip: this is also a mask file with .msk extension. Do not confuse this with the mask file used in **XRDUA BP**.



XRDUA BP

Full Processing with model

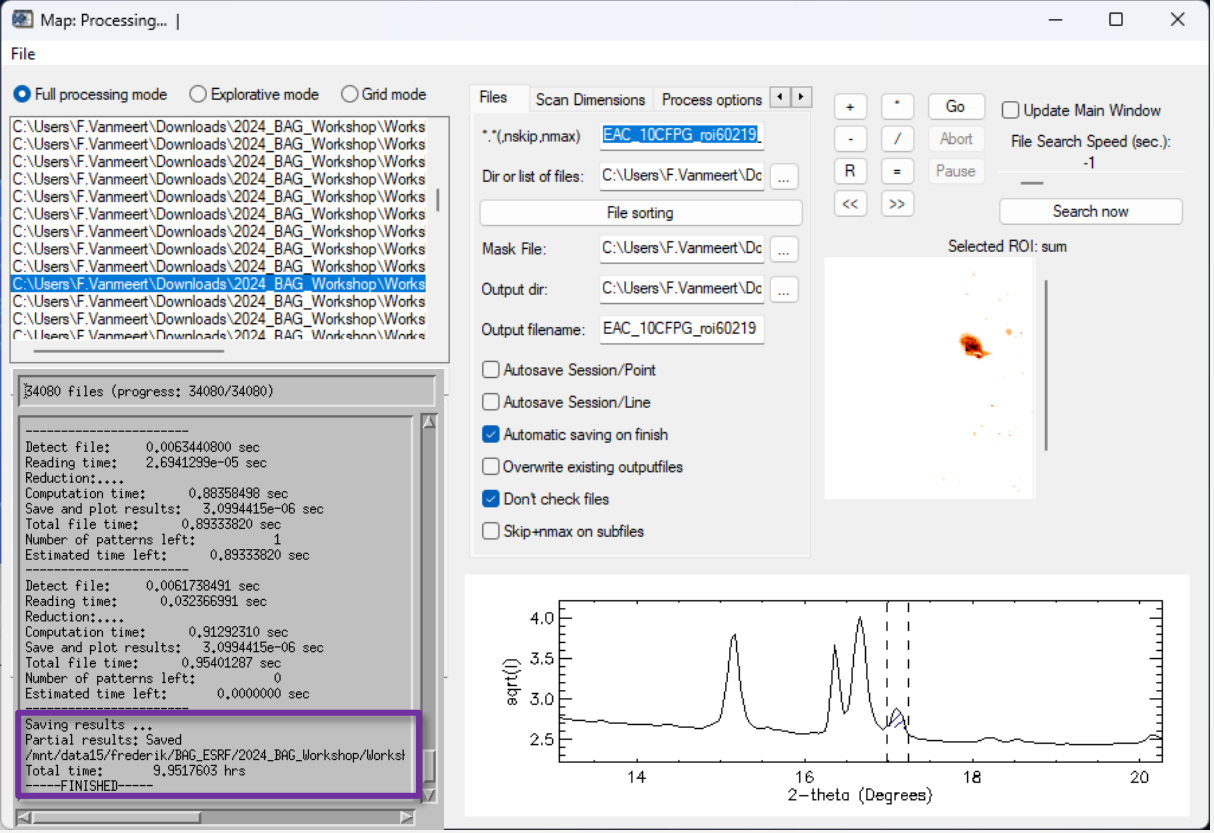
- Restore batch process (if it was closed): File -> Restore Session

1. Select Full processing mode
2. Change file to .tiff file generated by the Explorative mode batch process
3. Change Mask File to the fit model and change output directory and filename
4. Deselect Update Main Window

- Deselect View progress (under Process options)
- Press Go

6. Batch processing of fit model

RIJKS MUSEUM

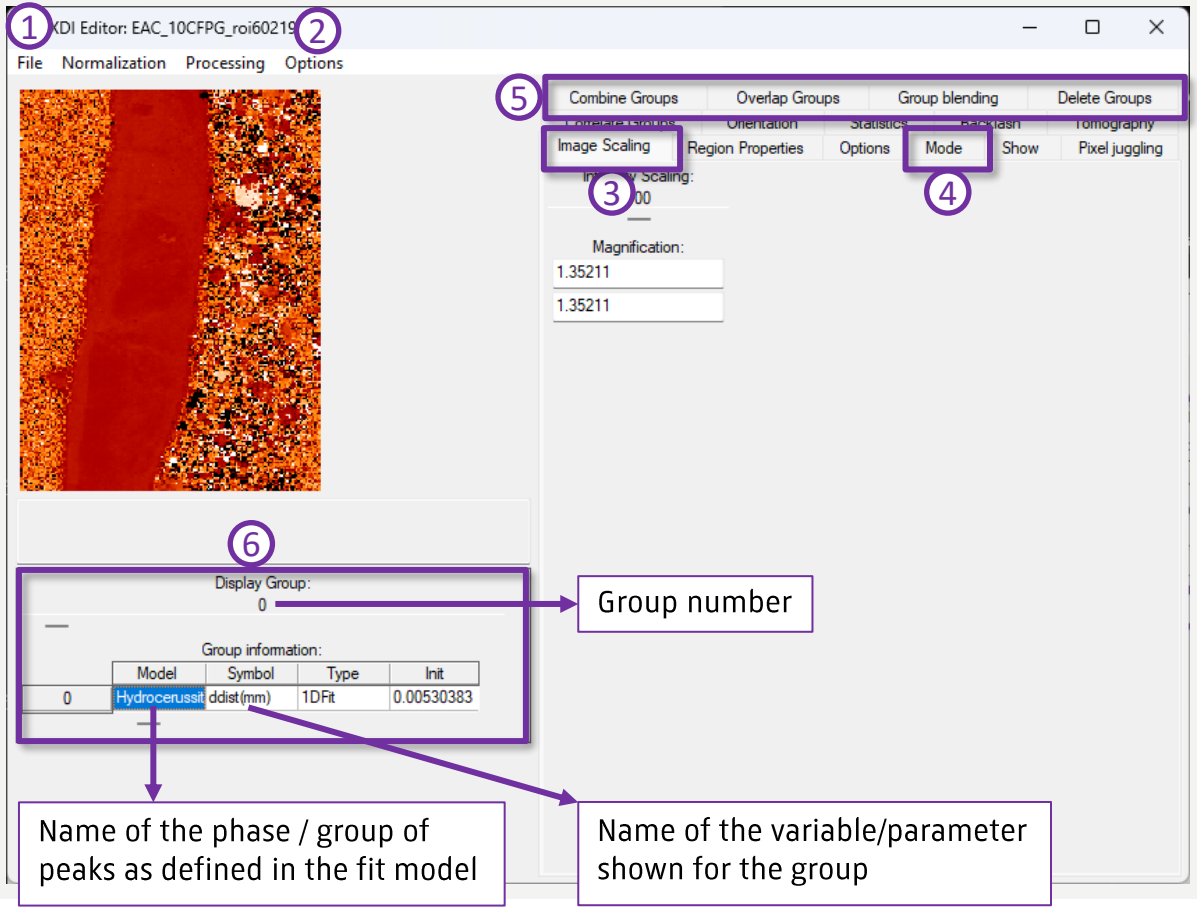


XRDUA BP

Full Processing with model

- Wait … or do something else (e.g., this fit took 10 h to finish)

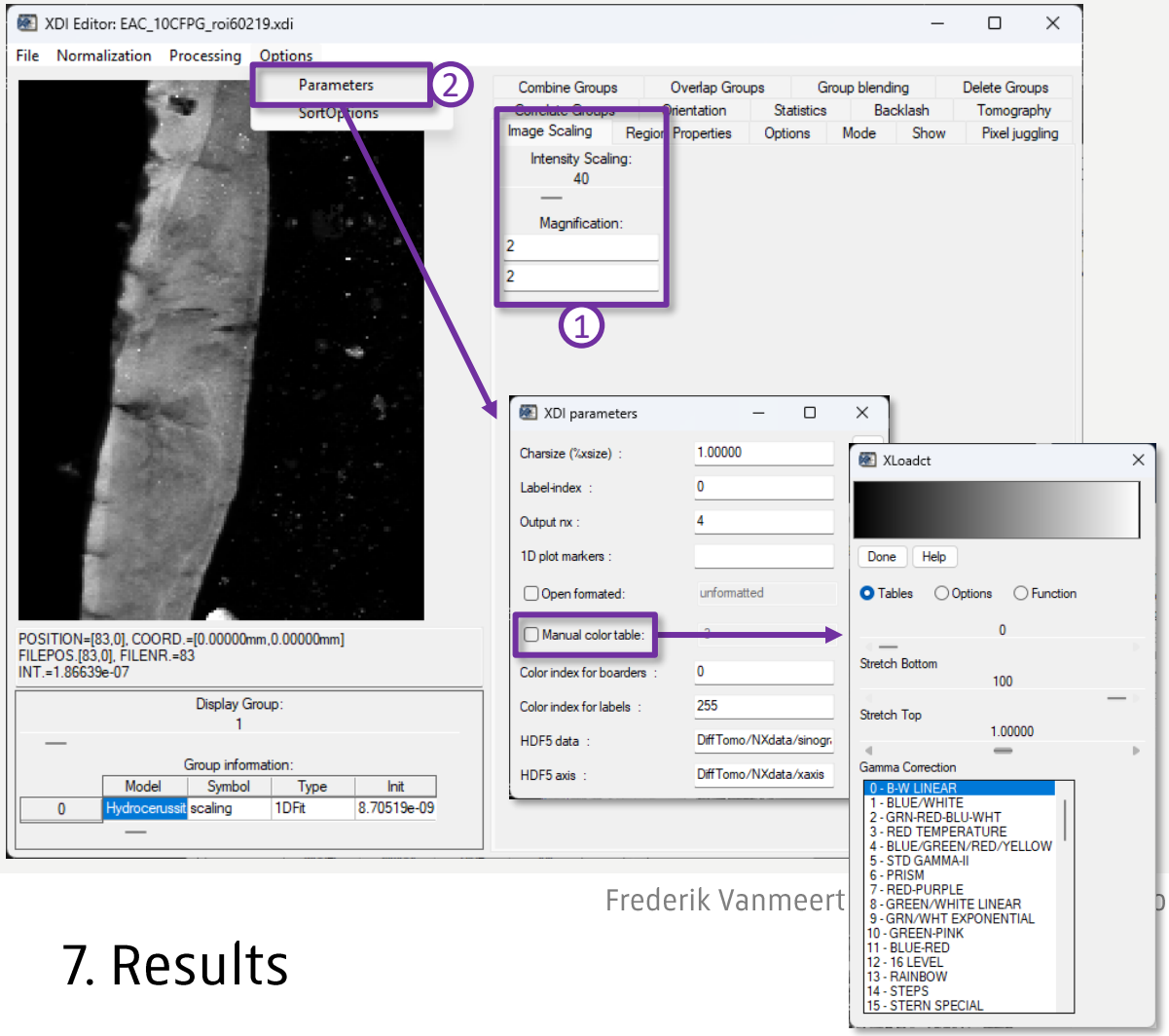
Tip: the batch session in Full processing mode does not get saved automatically. Save it so that you can easily rerun it in case you need to make a change to the fit model.



XRDUA XDI

Some main options

1. Save/Convert/Export images
2. Change color
3. Image and intensity scaling
4. Various options, including a link to **XRDUA 1D**
5. Options to manipulate groups
6. Window to scroll through the results
   - Each variable that was refined in the fit is present as a group
   - Also, standard deviations, derived parameters, quality of fit metrics are saved as a group

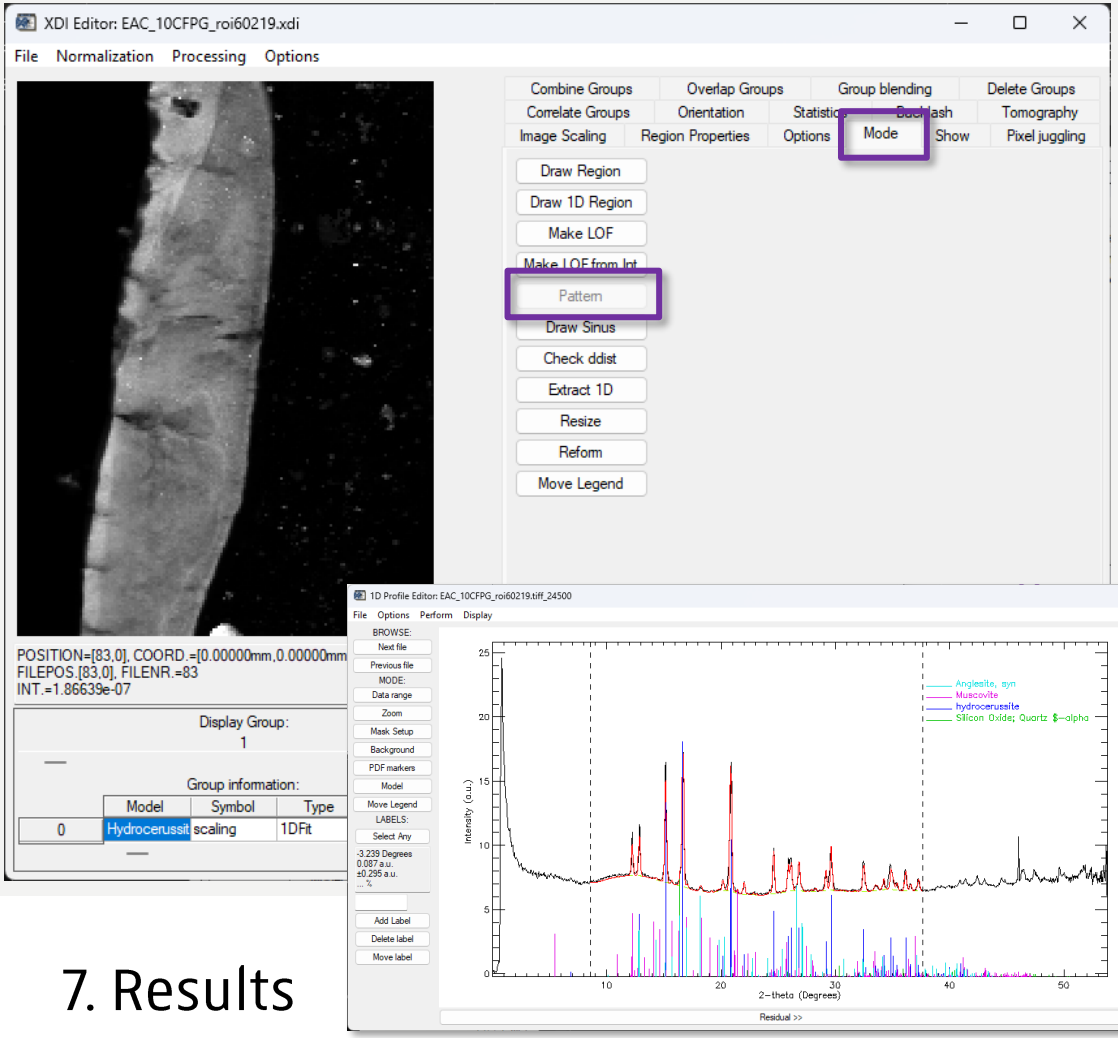


XRDUA XDI

Looking at your results

- Open .xdi file with **XRDUA XDI**

1. Change scaling (and intensity)
2. Option to change color
   - Go to Options → Parameters
   - Deselect Manual color table
   - Select color



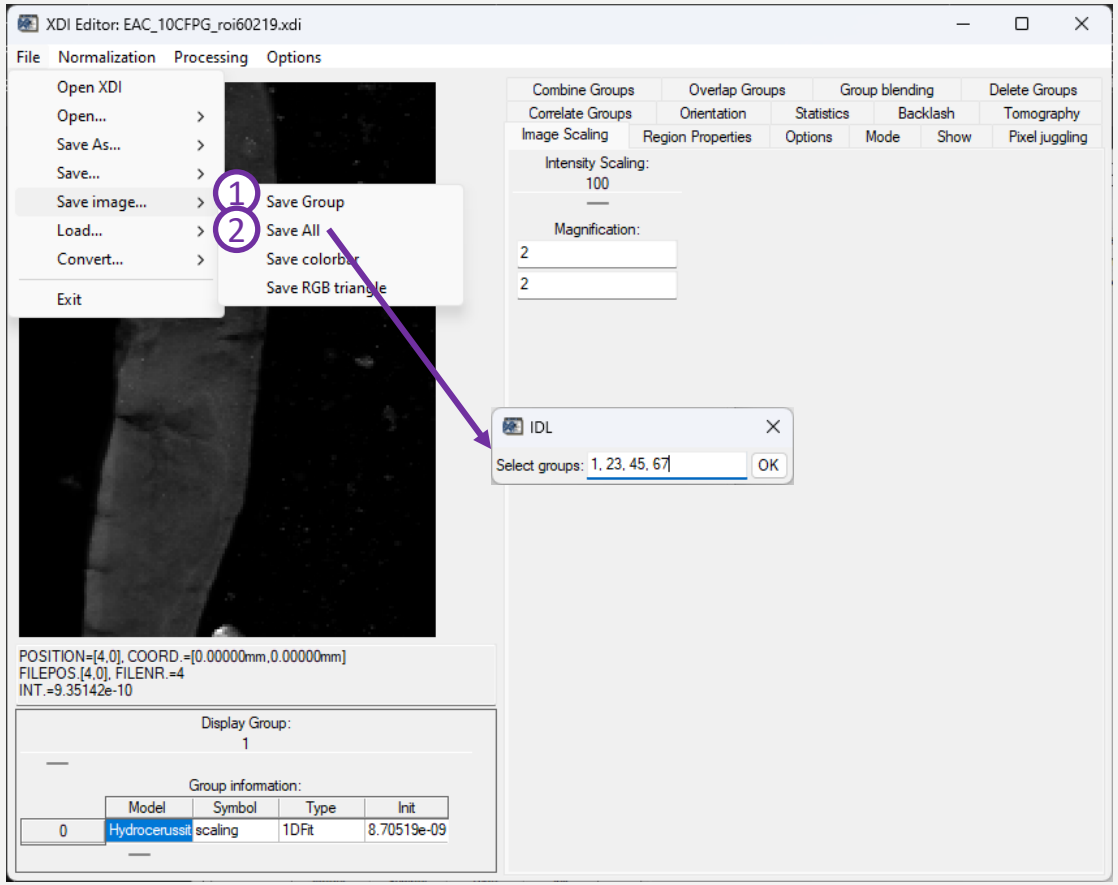
XRDUA XDI

Verify results!

- Go to Mode and click Pattern
- Click on a pixel in **XRDUA XDI** to load the 1D profile in **XRDUA 1D**
- Verify if the model is working properly in this pixel
- Make changes to the model if necessary and rerun the fit
  - E.g., change initial values, change constraints, add other phases, …

The model is constructed using only a small number of pixels. It is important to check if the model works well for the entire map!

RIJKS MUSEUM



XRDUA XDI

Exporting results

- Results are exported using the current (intensity) scaling

1. Single group
   - Go to the group that you want to save
   - Save Group
2. Multiple groups
   - Write down all the group numbers that you want to save
   - Save All
   - Separate numbers of the group with a comma ,

Useful links and reads:

- XRDUA Distribution Page
  Main XRDUA website with info, tutorials, and download link to latest version
- XRDUA download | SourceForge.net
  Sourceforge page for XRDUA
- NV5 | Customer Portal
  Portal to register and download IDL virtual machine needed to run XRDUA
- XRDUA Publication and general data processing strategy
  https://journals.iucr.org/paper?S1600576714008218
- American Mineralogist Crystal Structure Database
  Free crystal structure database with atomic information
- Crystallography Open Database
  Free crystal structure database with atomic information
- QualX – Software Ic
  Free Search-Match software