

XRDUA – How to process (ID13) XRD imaging data

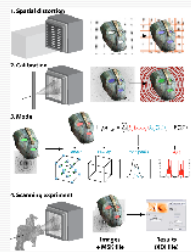
Frederik Vanmeert
03/12/2024



Latest XRDUAA version: 7.7.1.1

[Overview](#) [What's new](#) [Download](#) [Source code](#) [Documentation](#) [Older versions](#) [Support](#)

XRDUAA is a software package developed by the Antwerp X-ray Imaging/Instrumentation Laboratory (AXIL) at the University of Antwerp. Its main purpose is to automate the processing of two dimensional x-ray diffraction images from scanning (μ)XRPD or (μ)XRPD tomography. It accepts images from flat area detectors and allows correction, calibration and modeling (Rietveld, Pawley, Pattern Decomposition). The primary goal is to provide a virtual cross section (or in a virtual cross section) of the amount of material, structural properties and phase composition, which can be visualized as well.



XRDUAA mask file: contains corrected



Frederik Vanmeert - 2024 Heritage BAG Workshop

Starting with XRDUAA

1. Install IDL 8.3 or higher (no license required).
2. Download XRDUAA 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**).

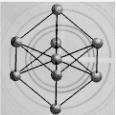
What to expect

- 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

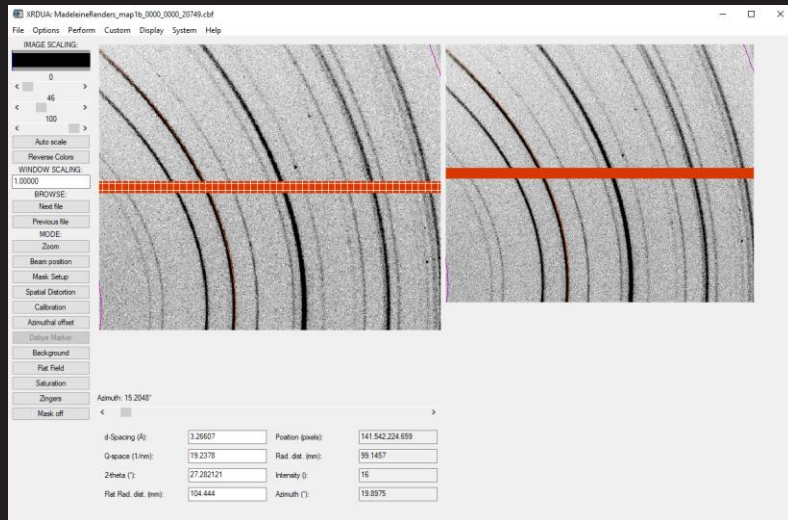


Universiteit
Antwerpen

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

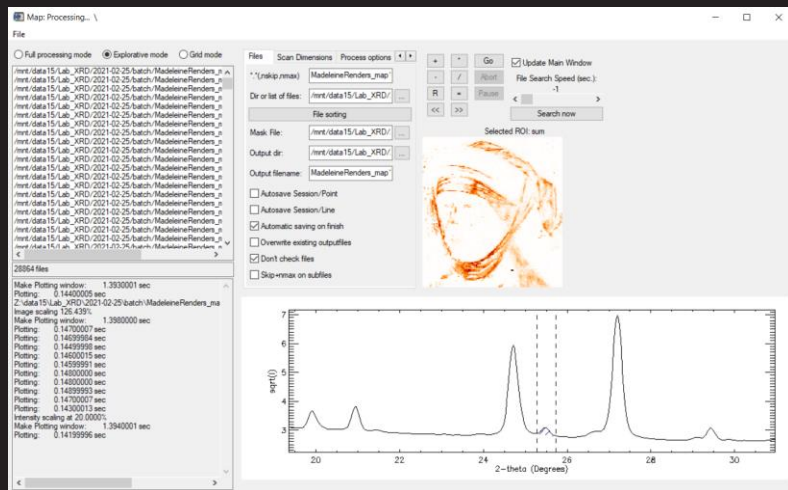


Dr. Wout de Nolf
(ESRF)

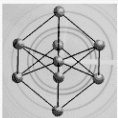


From raw diffraction data to phase distributions

- 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, ...)
- ...



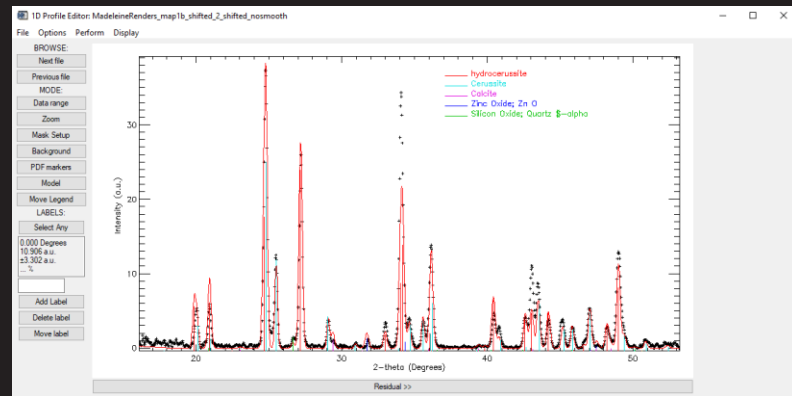
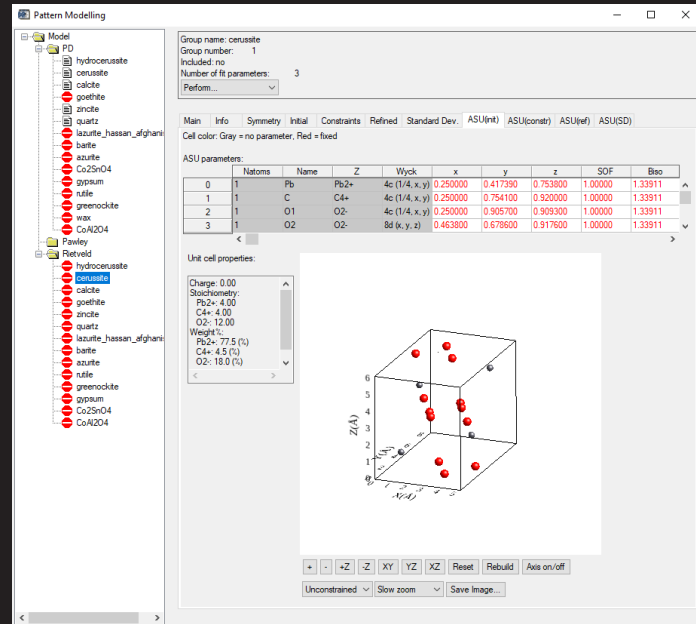
De Nolf, W., et al. (2014). "XRDUA: crystalline phase distribution maps by two-dimensional scanning and tomographic (micro) X-ray powder diffraction." *Journal of Applied Crystallography* 47(3): 1107-1117.



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, ...)
- ...






Latest XRDUA version: 7.7.1.1

[Overview](#) [What's new](#) [Download](#) [Source code](#) [Documentation](#) [Older versions](#) [Support](#)

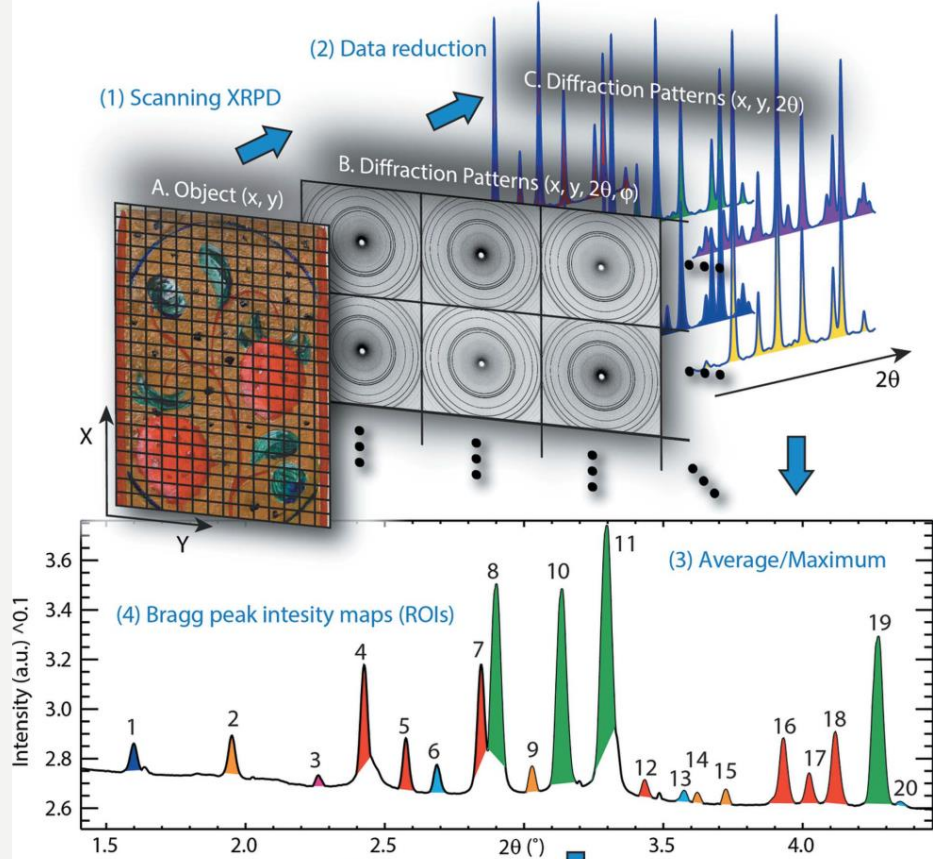
The XRDUA Documentation Series contains:

- Part1 (**obsolete: needs updating!**): Reference Manual 
- Part2: Tutorial manuscript  with example data 

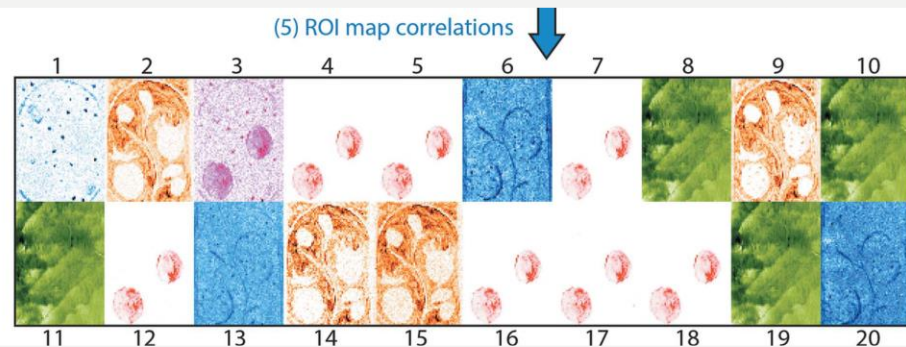
A more elaborate tutorial with manual and example data is available on the XRDUA website:
[XRDUA Distribution Page](#)

Contents

1	XRDUA introduction	1
1.1	Install and run	1
1.2	Getting started	1
1.3	Overview	2
1.4	Some common operations	4
2	From raw data to reciprocal space	7
2.1	Raw image corrections	7
2.1.1	Zinger removal	8
2.1.2	Spatial distortion	8
2.1.3	Saturation removal	12
2.1.4	Background removal	12
2.1.5	Flat field correction	13
2.1.6	Masking off	13
2.2	Calibration	13
2.2.1	Calibration standard	14
2.2.2	Initial estimation of the beam position	14
2.2.3	Initial estimation of energy and sample-detector distance	15
2.2.4	Debye ring selection: manually	16
2.2.5	Debye ring selection: automatically	16
2.2.6	Calibrate	17
2.3	Azimuthal integration: from 2D to 1D	18
3	Live at the synchrotron	23
3.1	Two-dimensional regions	23
3.2	One-dimensional regions	27
3.3	Explorative batch processing	28
4	From reciprocal space to real life	31
4.1	Phase identification and pattern decomposition	31
4.1.1	Pattern decomposition	33
4.1.2	Identification and global parameters	37
4.1.3	Identification strategy	39
4.1.4	Phase distributions	41
4.1.5	Constraints	41
4.2	Pawley fitting	42
4.3	Rietveld refinement	44



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.

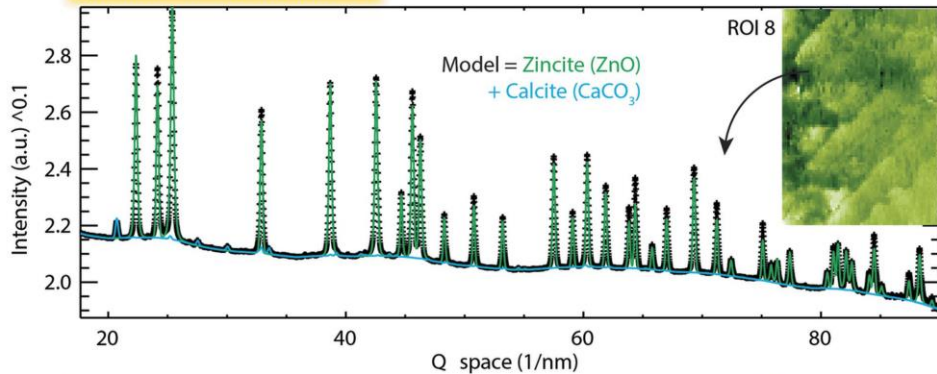


Frederik Vanmeert - 2024 Heritage BAG Workshop

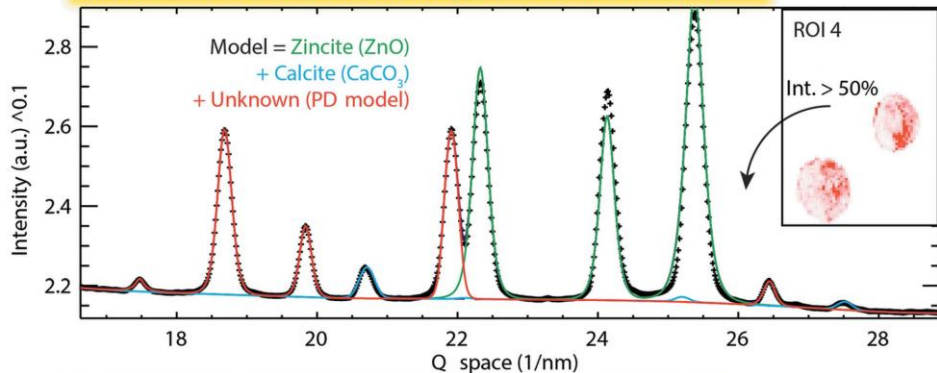
XRDUA Workflow

RIJKS MUSEUM

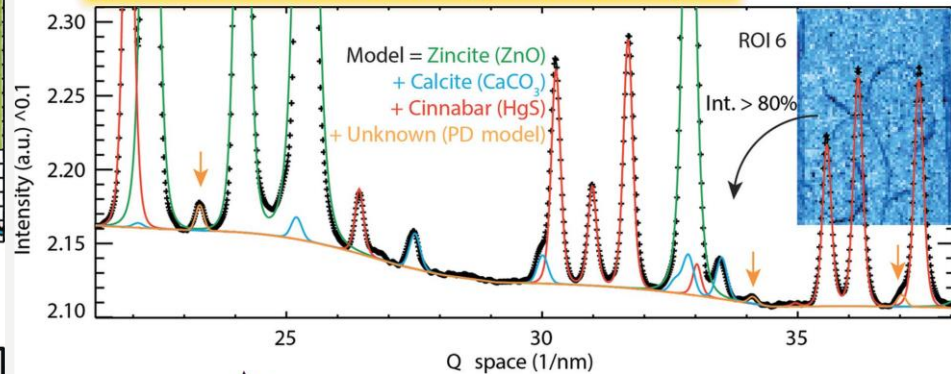
(a) Identification of major phases



(b) Identification based on unindexed Bragg peaks from the strawberry area



(c) Identification based on unindexed Bragg from the red markup lines



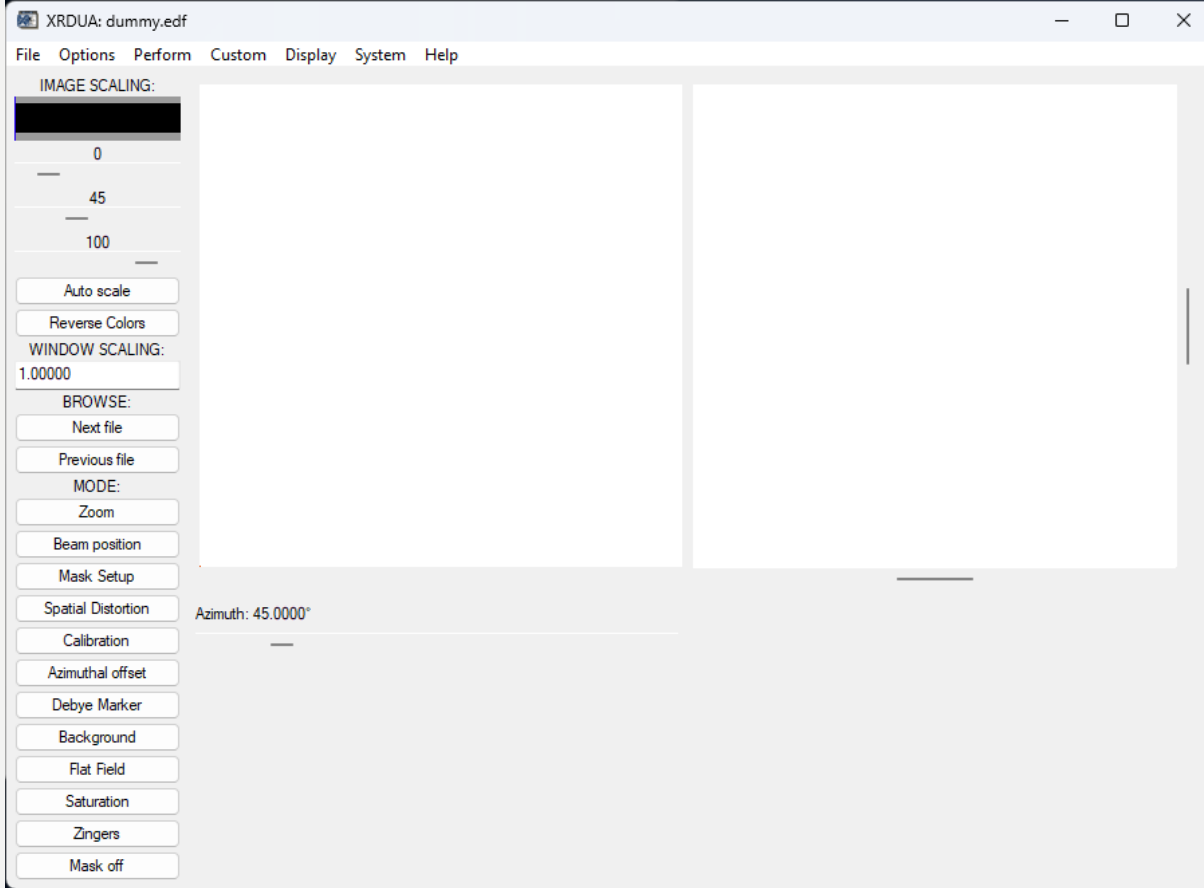
(d) Batch fitting with complete model



- Hematite (Fe_2O_3)
- Goethite ($\text{FeO}(\text{OH})$)
- Prussian blue ($\text{Fe}_4[\text{Fe}(\text{CN})_6]_3 \cdot 14\text{H}_2\text{O}$)
- Cinnabar (HgS)
- Unidentified

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

Frederik Vanmeert - 2024 Heritage BAG Workshop



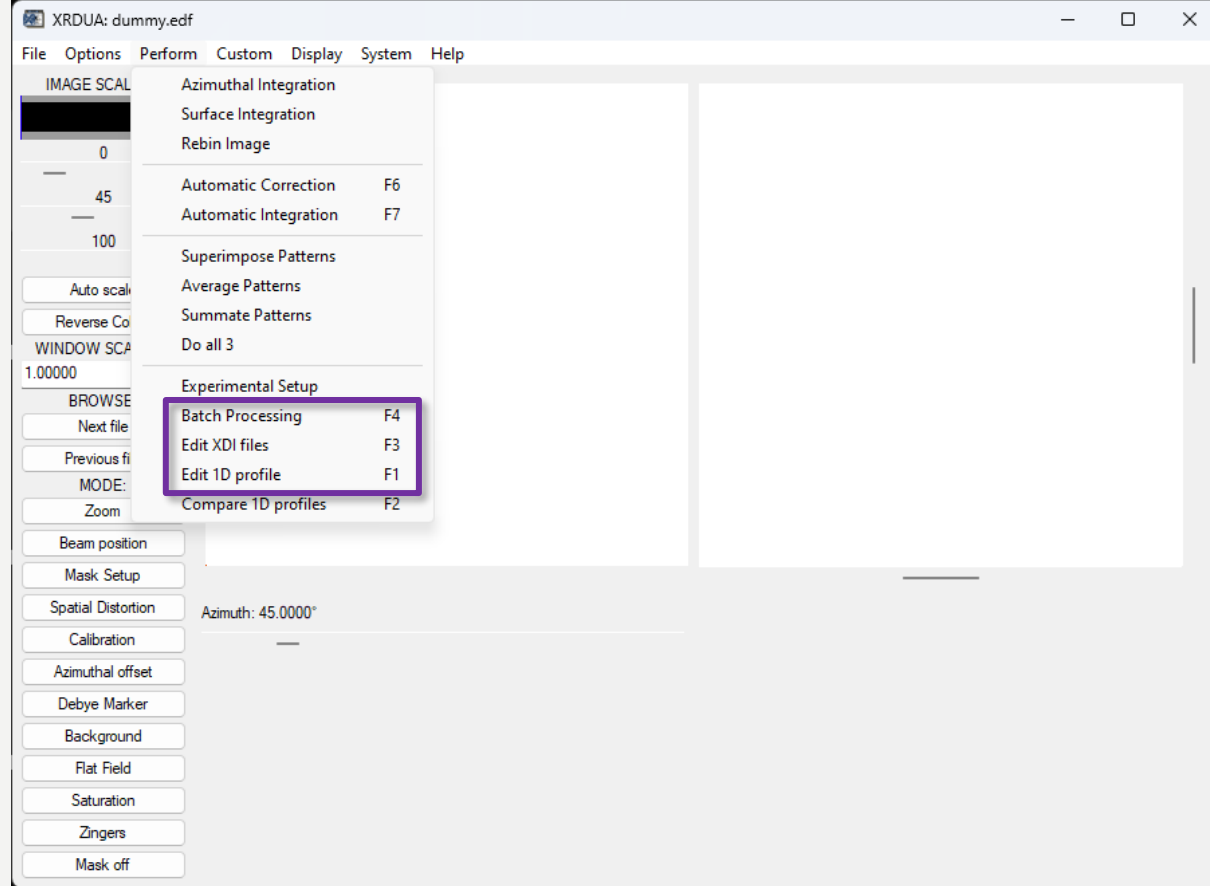
XRDU 2D

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

Frederik Vanmeert - 2024 Heritage BAG Workshop

Main windows: **XRDU 2D**

RIJKS MUSEUM



XRDU 2D

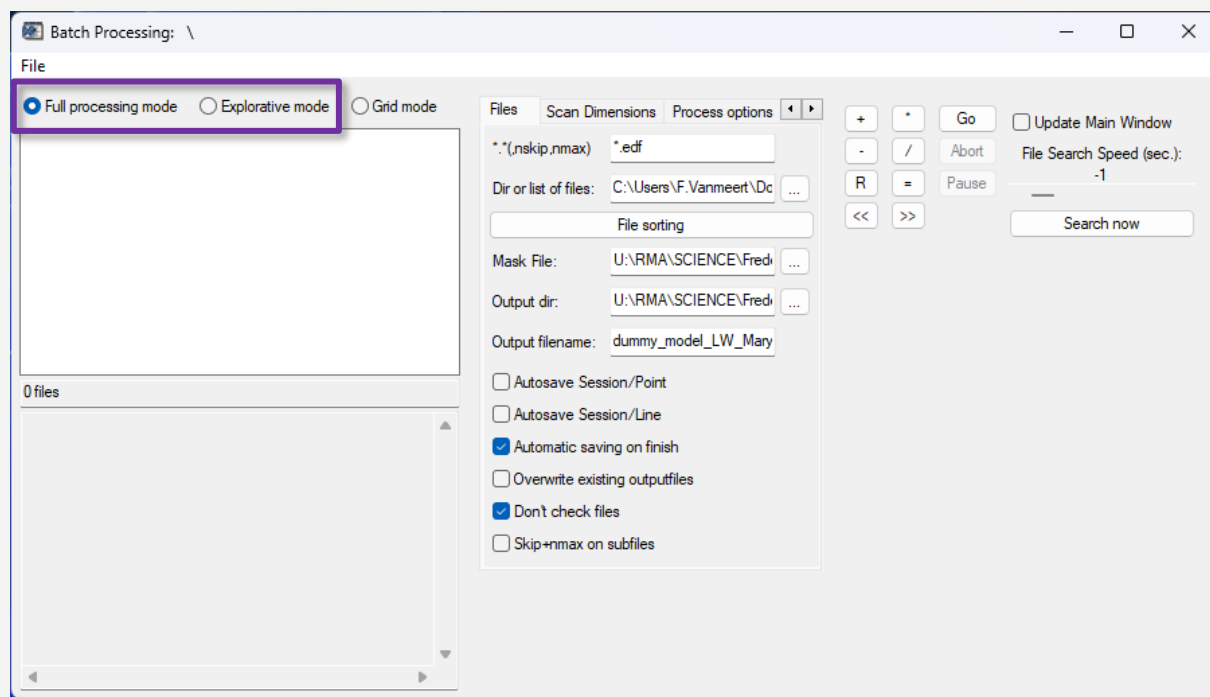
Perform ->

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

Frederik Vanmeert - 2024 Heritage BAG Workshop

Main windows: **XRDU 2D**

RIJKS MUSEUM



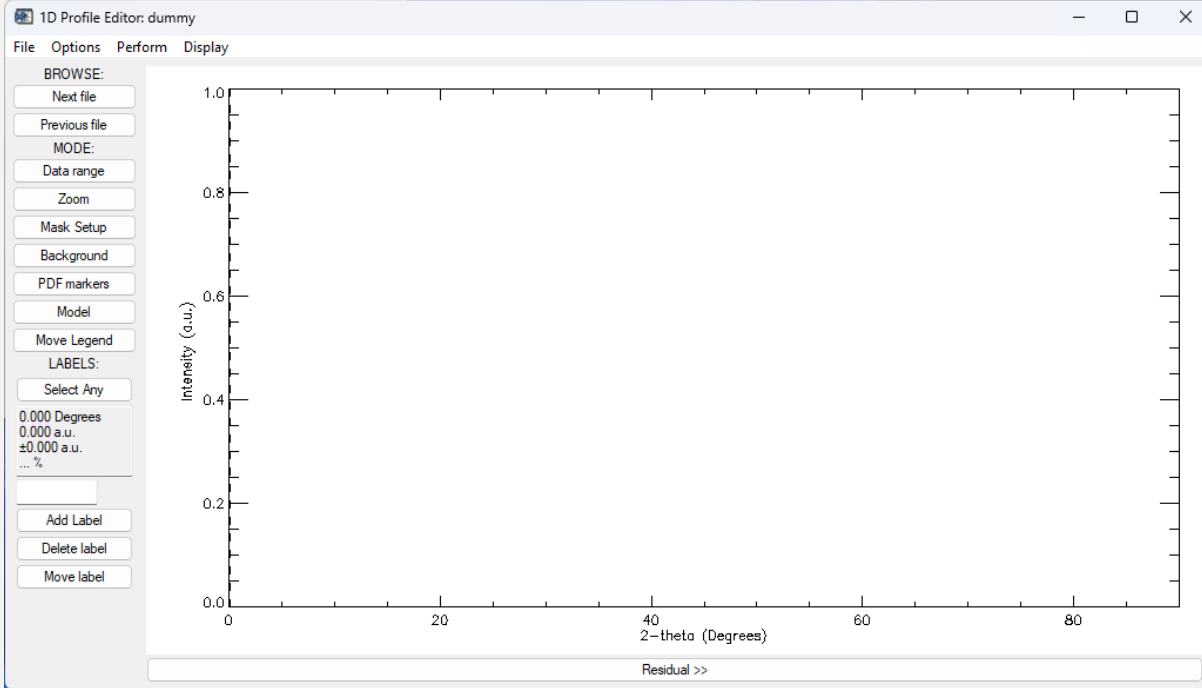
XRDUA BP

- *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)

Frederik Vanmeert - 2024 Heritage BAG Workshop

Main windows: **XRDUA BP**

RIJKS MUSEUM



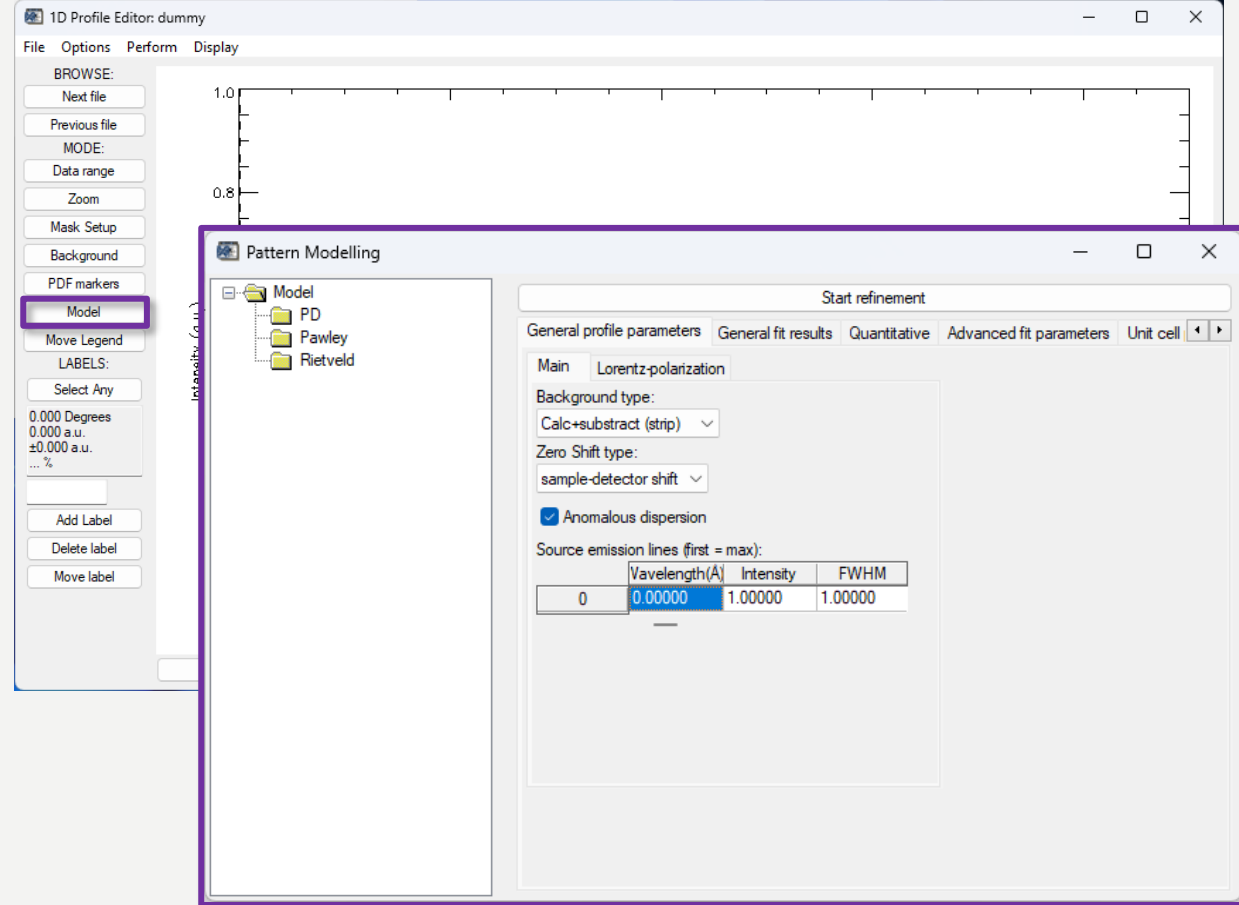
XRDUA 1D

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

Frederik Vanmeert - 2024 Heritage BAG Workshop

Main windows: **XRDUA 1D**

RIJKS MUSEUM



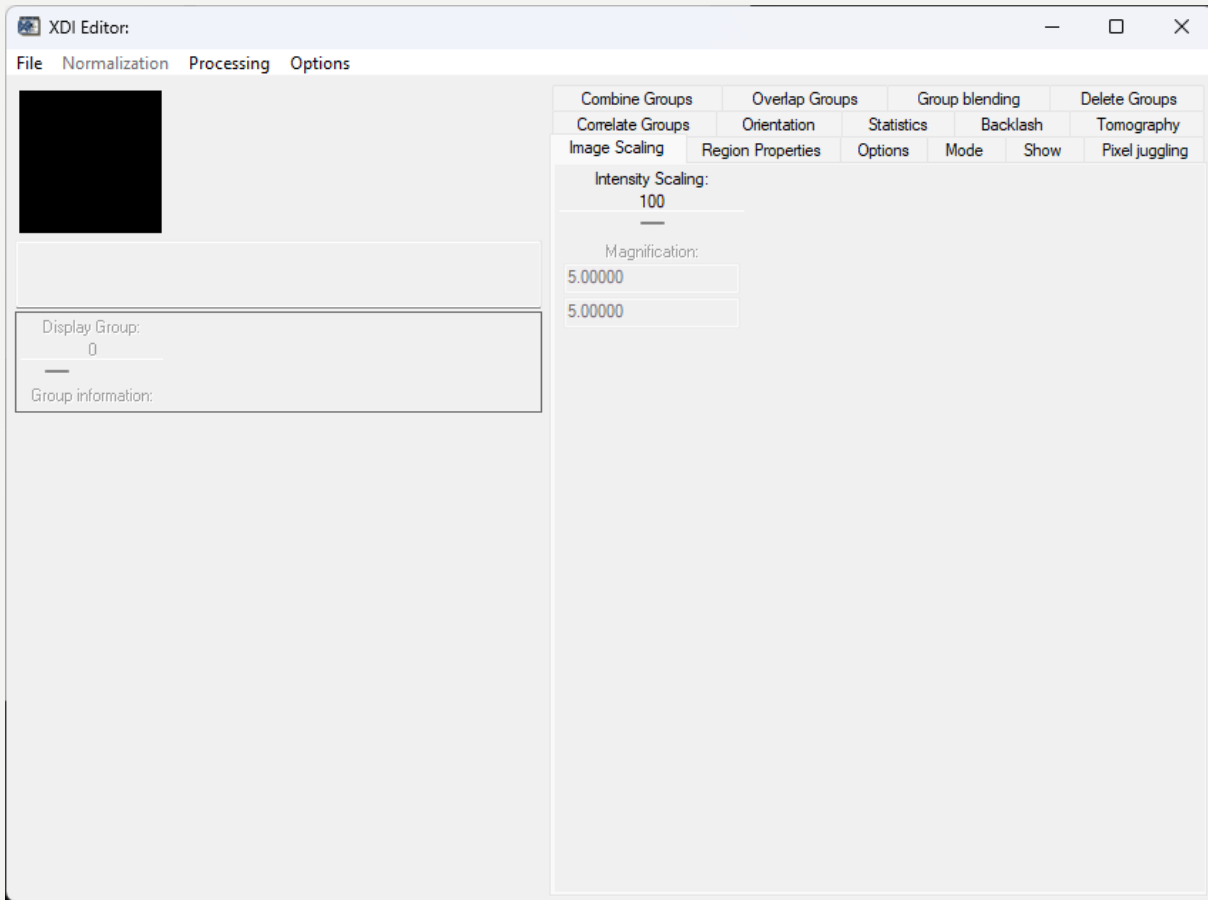
XRDUA 1D

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

Frederik Vanmeert - 2024 Heritage BAG Workshop

Main windows: **Modelling**

RIJKS MUSEUM



XRDUA XDI

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

Frederik Vanmeert - 2024 Heritage BAG Workshop

Main windows: **XRDUA XDI**

RIJKS MUSEUM

Batch Processing: |

File

Full processing mode Explorative mode Grid mode

C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works

34080 files (progress: 34080/34080)

Plotting: 0.93400002 sec
Save and plot results: 0.93999982 sec
Total file time: 14.439000 sec
Number of patterns left: 0
Estimated time left: 0.0000000 sec

Saving results ...
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
Partial results: Saved
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
Total time: 22.383000 sec
---FINISHED---

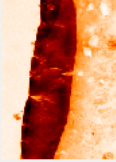
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works

Scan Dimensions Process options Scanne

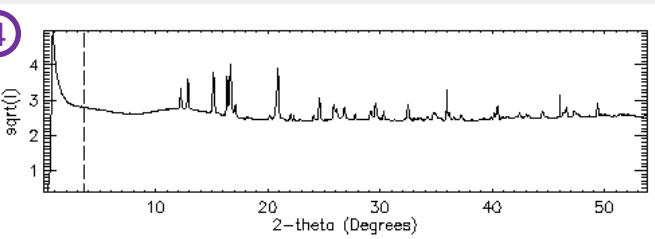
2D background on ROI's
 1D background on ROI's
 Normalized ROI's
 View progress
 Monitor processing
 Explorative background
 Explorative superimposed profile
 Calculate 2D superimposed/average

+ * Go Update Main Window
- / Abort File Search Speed (sec.):
R = Pause -1
<< >> Search now

Selected ROI: sum



sqrt(I)



2-theta (Degrees)

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

Frederik Vanmeert - 2024 Heritage BAG Workshop

1. Loading ID13 EDF data

RIJKS MUSEUM

Batch Processing: |

File

Full processing mode Explorative mode Grid mode

C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works

34080 files (progress: 34080/34080)

Plotting: 0.93400002 sec
Save and plot results: 0.93999982 sec
Total file time: 14.439000 sec
Number of patterns left: 0
Estimated time left: 0.0000000 sec

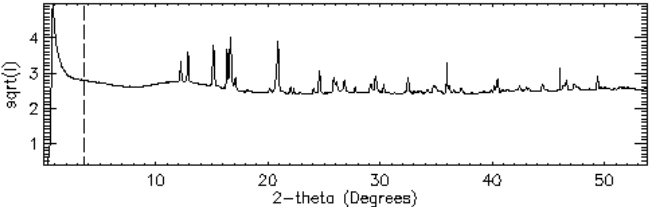
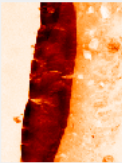
Saving results ...
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
Partial results: Saved
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
Total time: 22.383000 sec
----FINISHED----
C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works

Scan Dimensions Process options Scanne

2D background on ROI's
 1D background on ROI's
 Normalized ROI's
 View progress
 Monitor processing
 Explorative background
 Explorative superimposed profile
 Calculate 2D superimposed/average

Go Update Main Window
Abort File Search Speed (sec.): -1
Pause
Search now

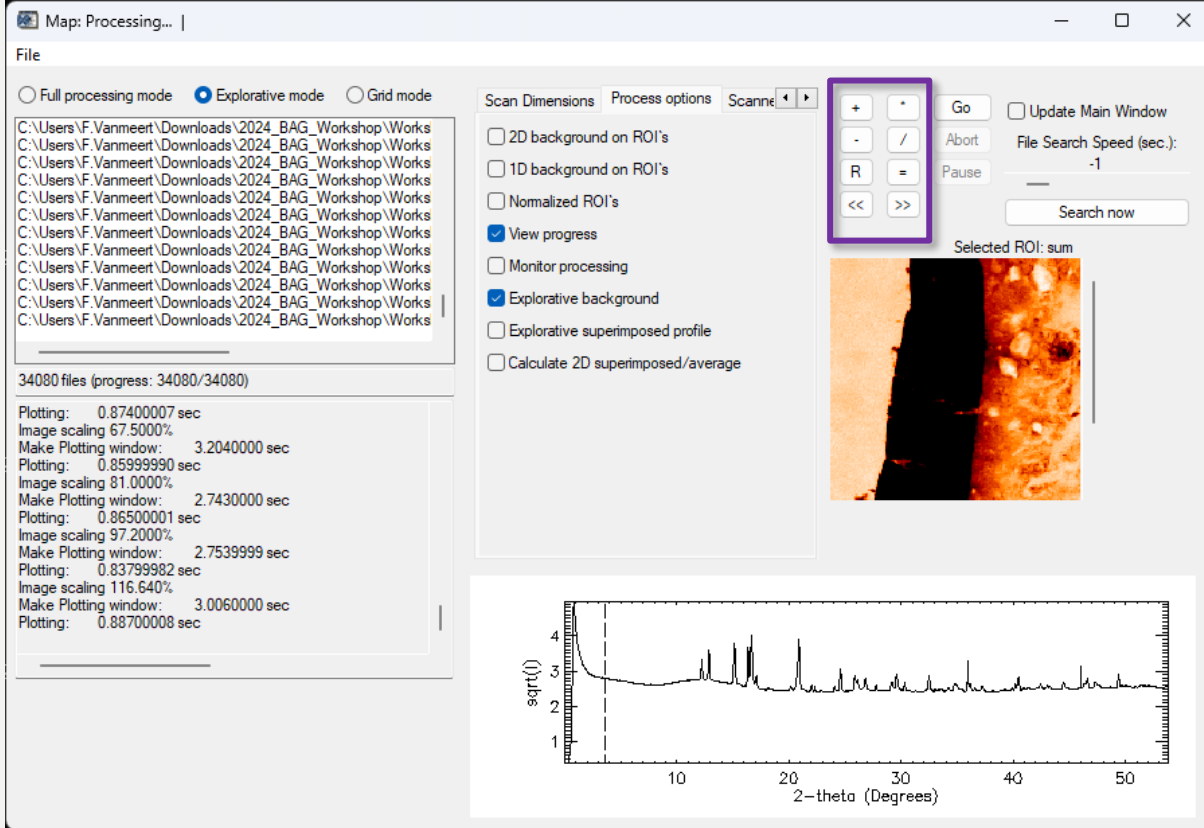
Selected ROI: sum



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



XRDUA BP

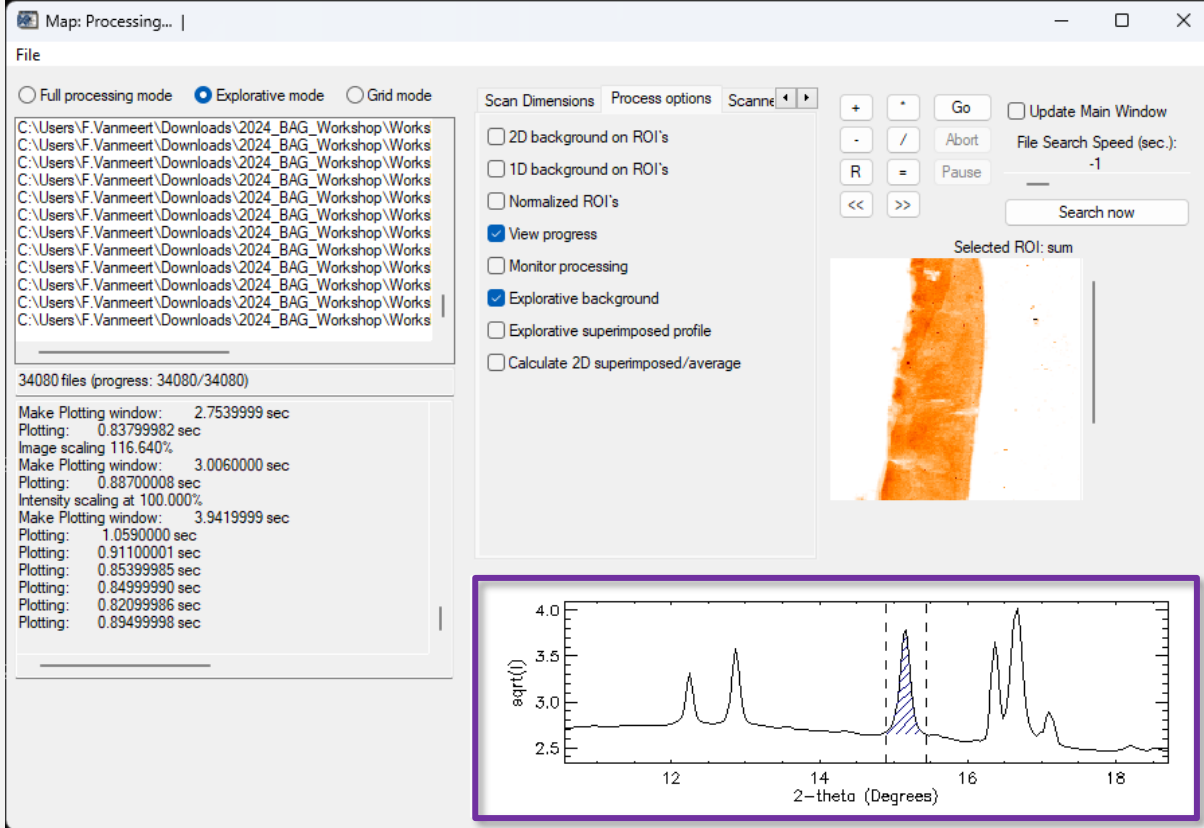
ROI image visualization

- **+** : 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

Frederik Vanmeert - 2024 Heritage BAG Workshop

2. Exploring Data

RIJKS MUSEUM



XRDUA BP

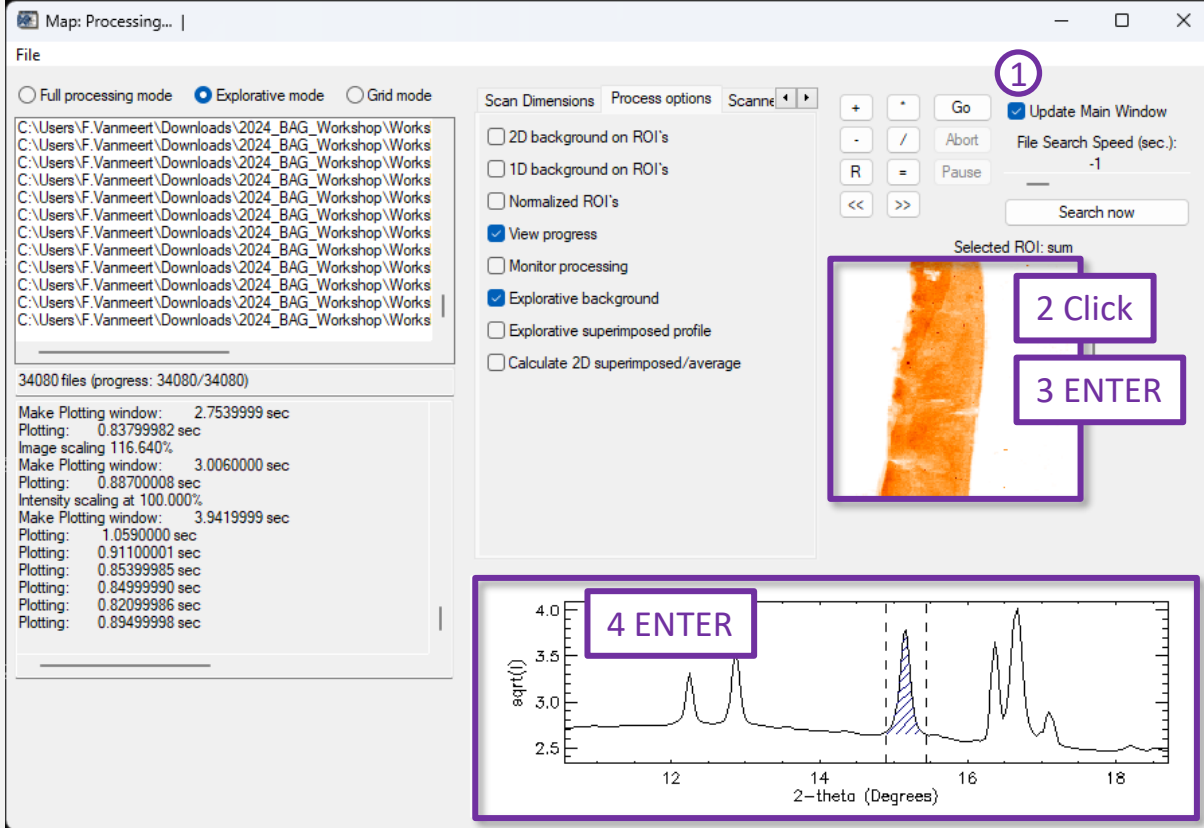
1D profile options

- **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

Frederik Vanmeert - 2024 Heritage BAG Workshop

2. Exploring Data

RIJKS MUSEUM



XRDUA BP

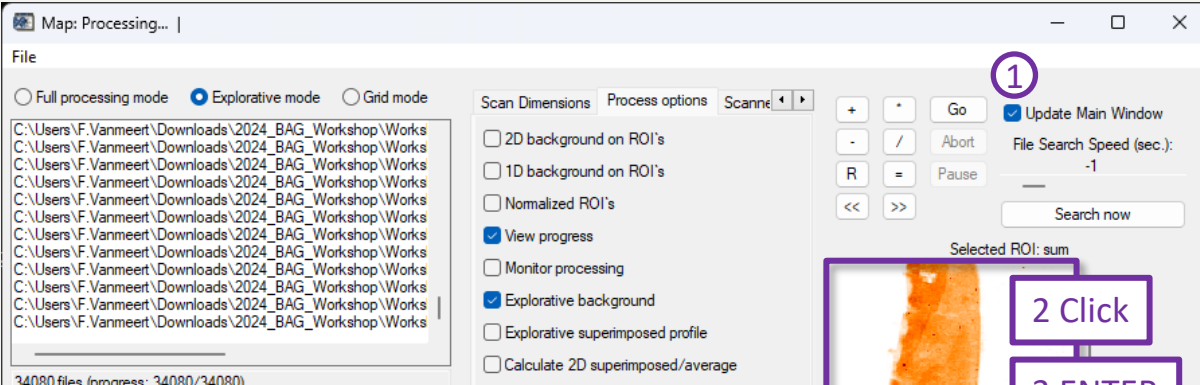
Link to **XRDUA 1D**

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**

Frederik Vanmeert - 2024 Heritage BAG Workshop

2. Exploring Data – Extracting 1D profiles

RJKS MUSEUM



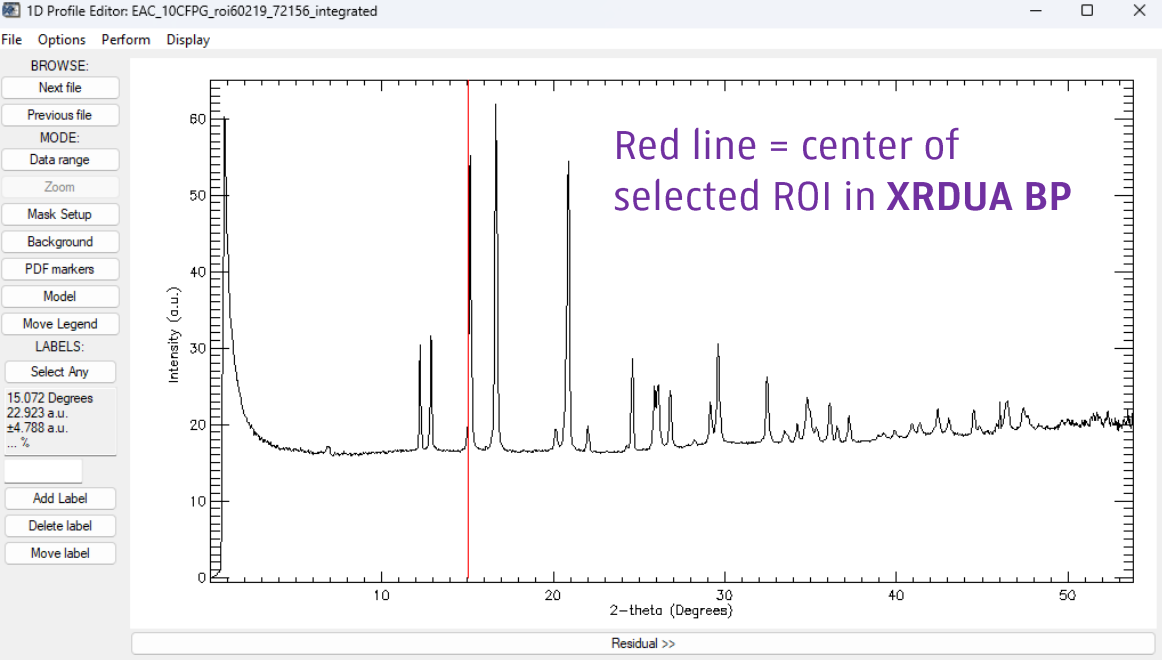
XRDUA BP

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

34080 files (progress: 34080/34080)

Make Plotting window: 2.7539999
 Plotting: 0.83799982 sec
 Image scaling 116.640%
 Make Plotting window: 3.0060000
 Plotting: 0.88700008 sec
 Intensity scaling at 100.000%
 Make Plotting window: 3.9419999
 Plotting: 1.0590000 sec
 Plotting: 0.91100001 sec
 Plotting: 0.85399985 sec
 Plotting: 0.84999990 sec
 Plotting: 0.82099986 sec
 Plotting: 0.89499998 sec



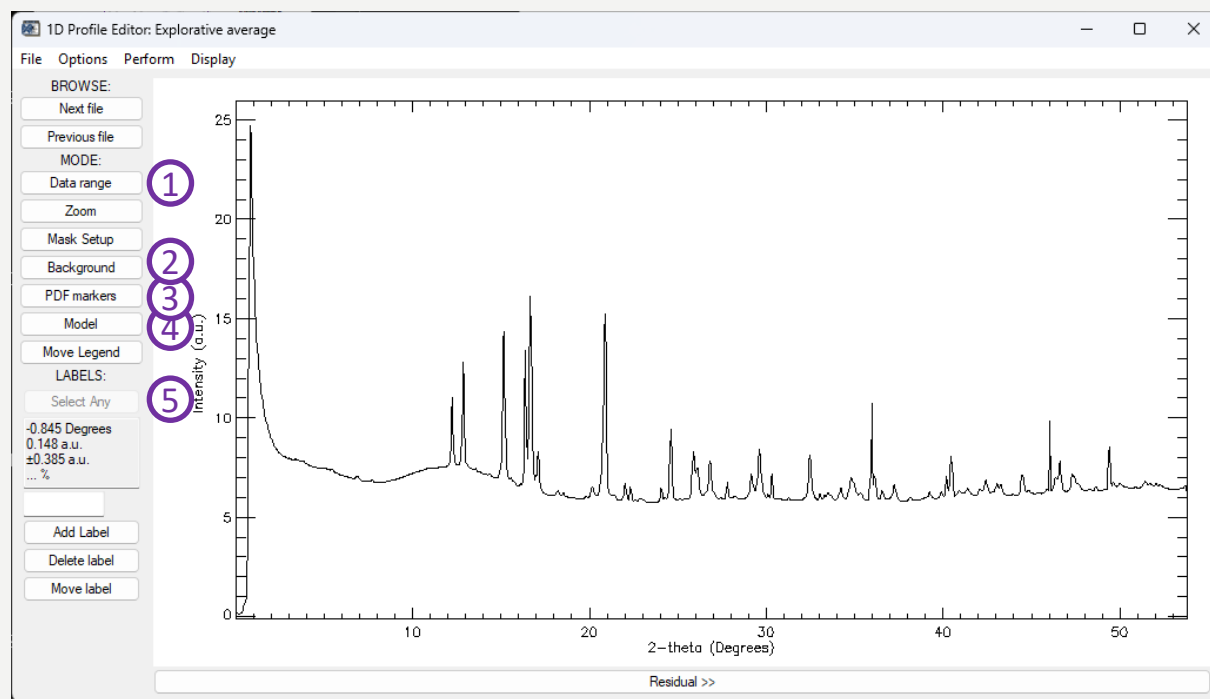
loads averaged (or superimposed) profile based on current ROI intensity in **XRDUA 1D**

ENTER when cursor is in file

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

2. Explor

RIJKS MUSEUM



XRDUA 1D

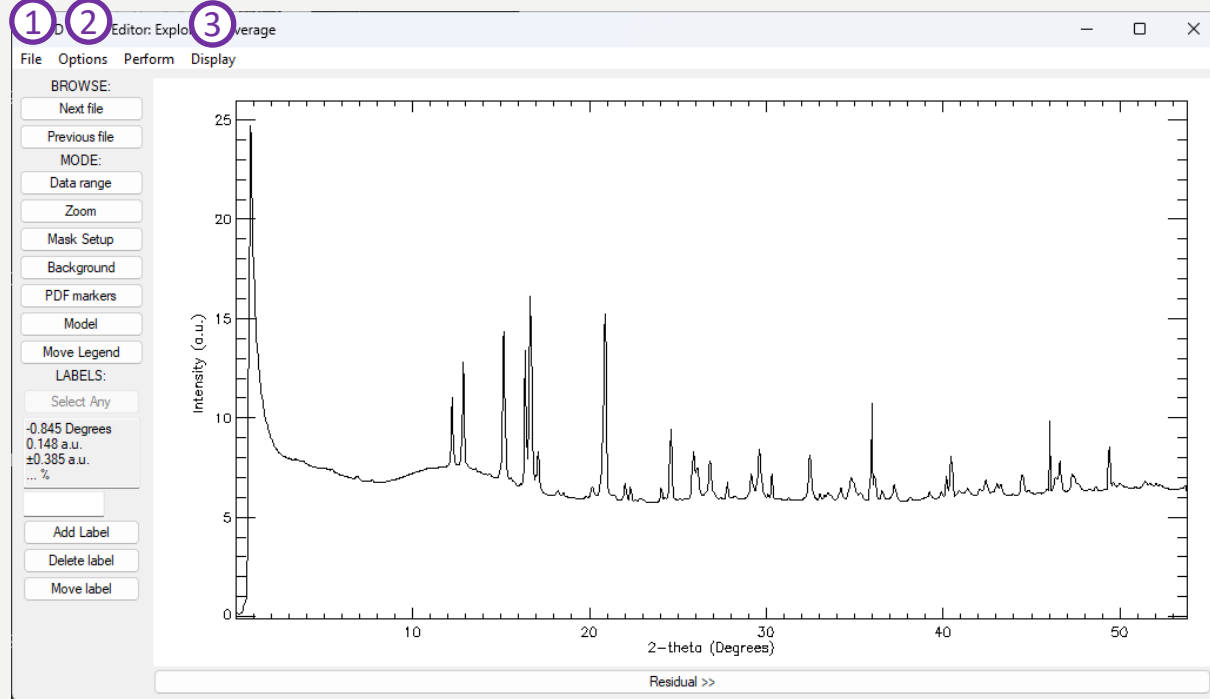
Some main options

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**

Frederik Vanmeert - 2024 Heritage BAG Workshop

XRDUA 1D

RIJKS MUSEUM



XRDUA 1D

Some main options

1. File

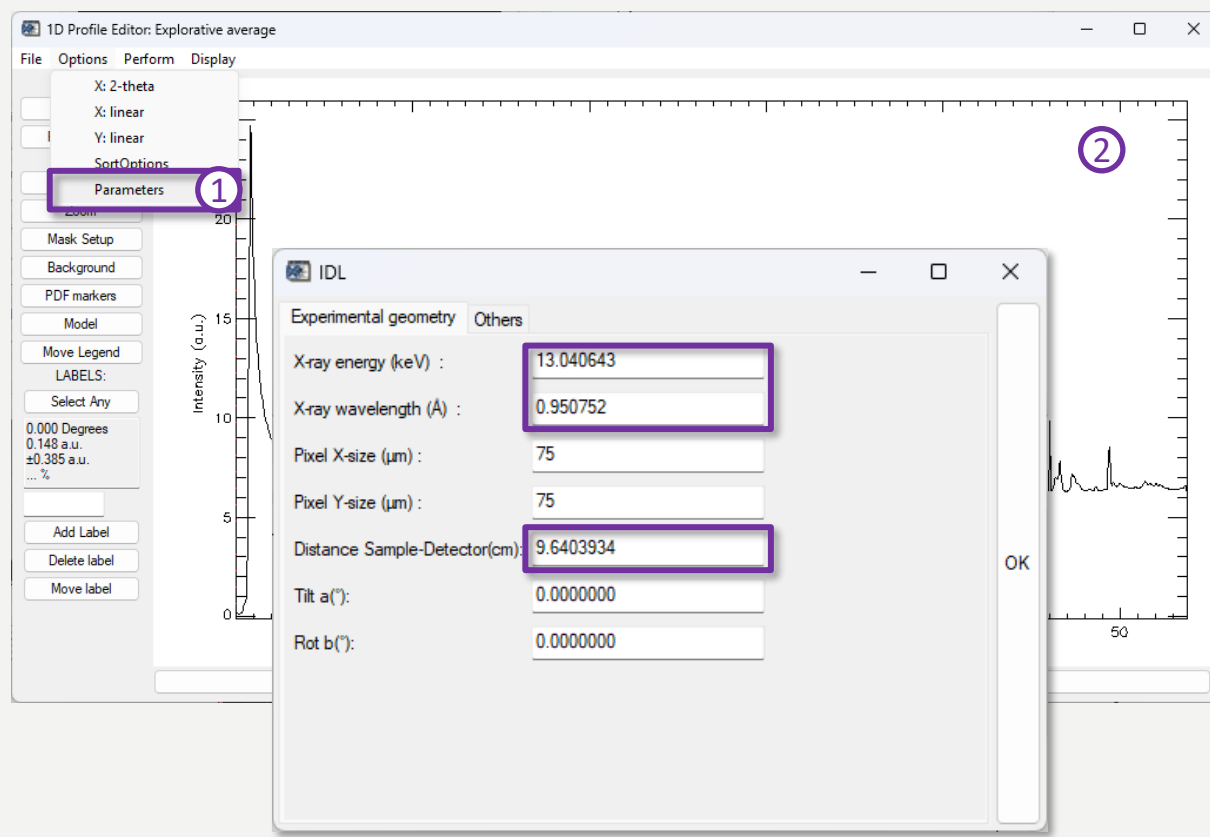
- Save/Load Mask (contains experimental parameters and fit model)
- Load (Multiple) PDF

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

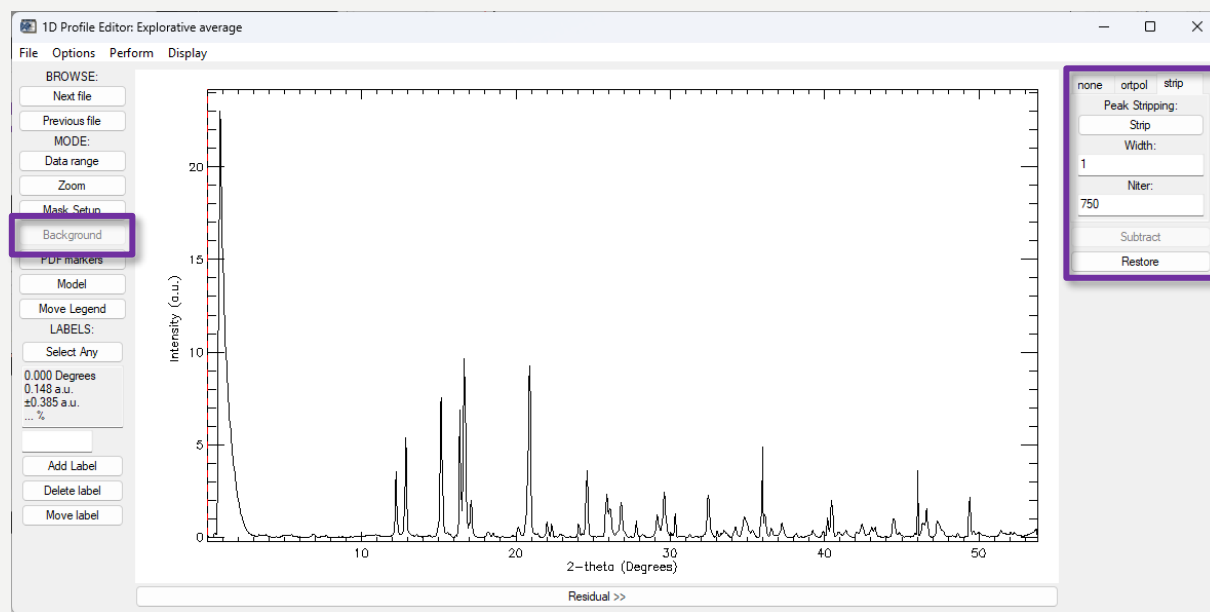
1. Set Experimental Geometry

- 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**

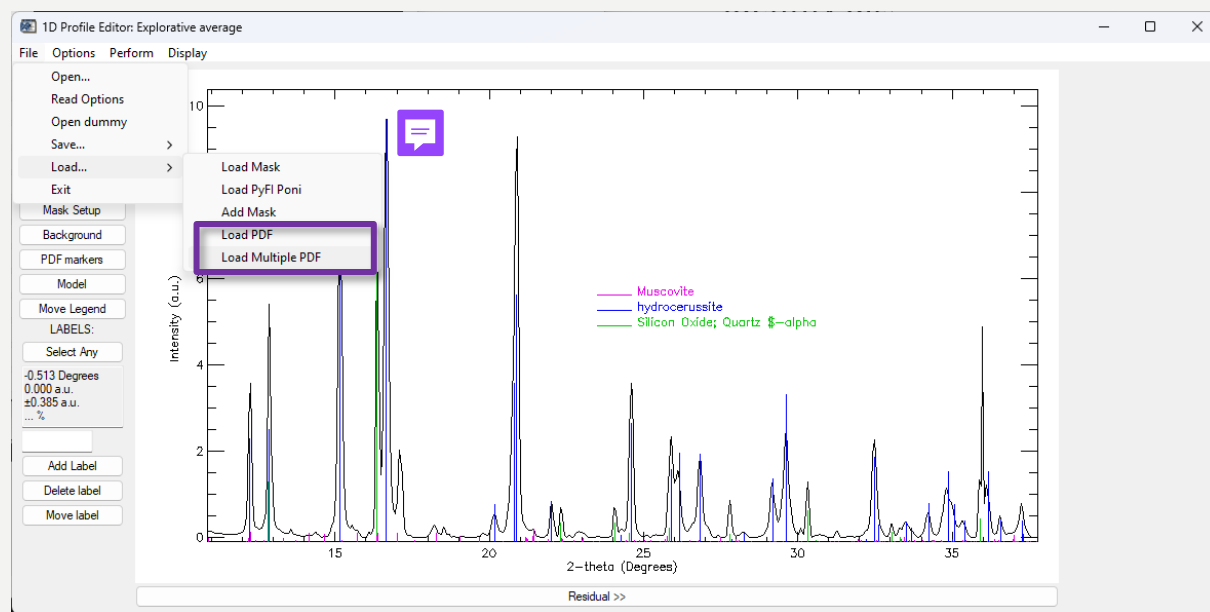
This is necessary the first time that you fill in the values of step 1 or load these values from an existing mask file.



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

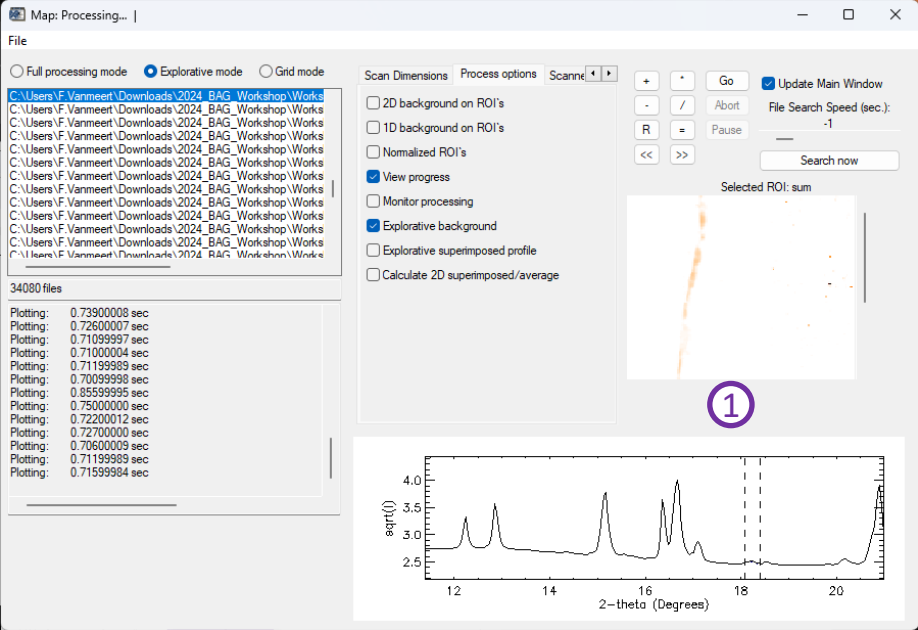
Identifying compounds

- 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).



PDF markers

Model

Move Legend

LABELS:

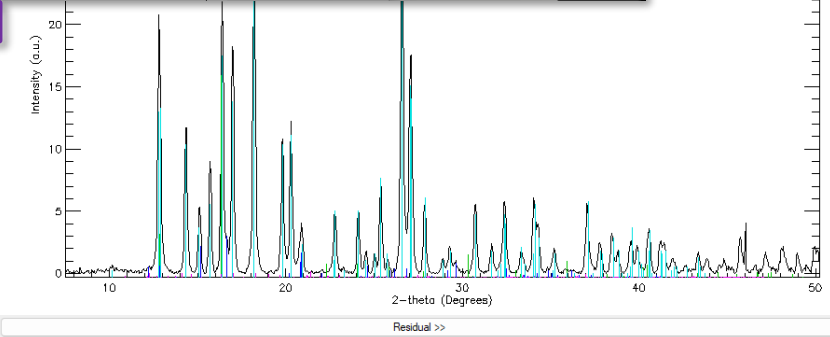
Select Any

6.936 Degrees
0.163 a.u.
±3.897 a.u.
... %

Add Label

Delete label

Move label

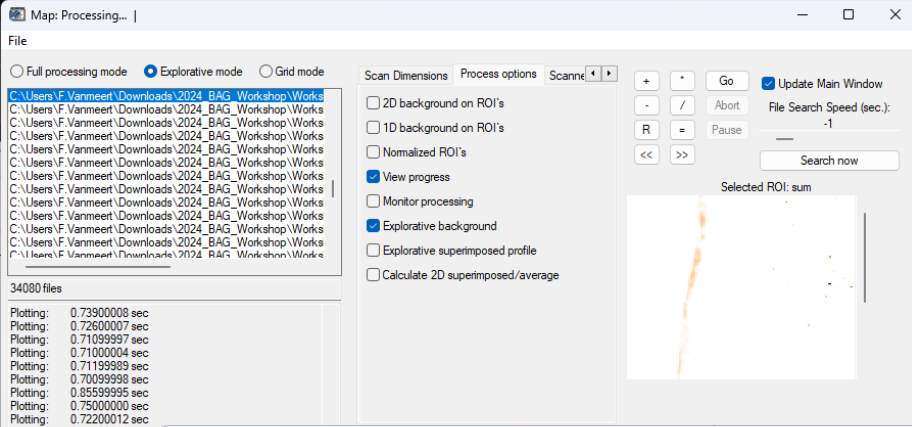


XRDUA BP + XRDUA 1D

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, ...).

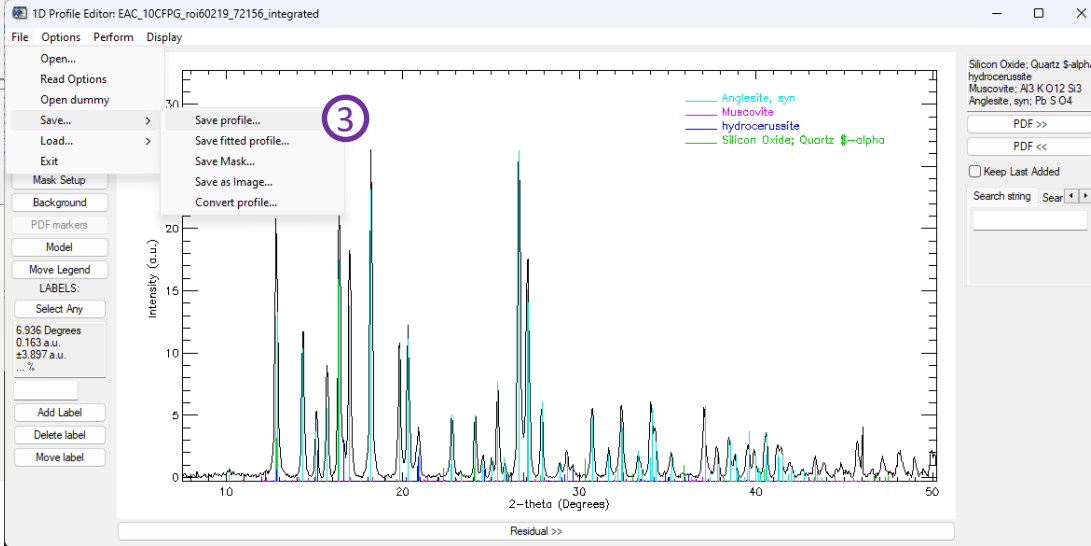
3. Identifying compounds



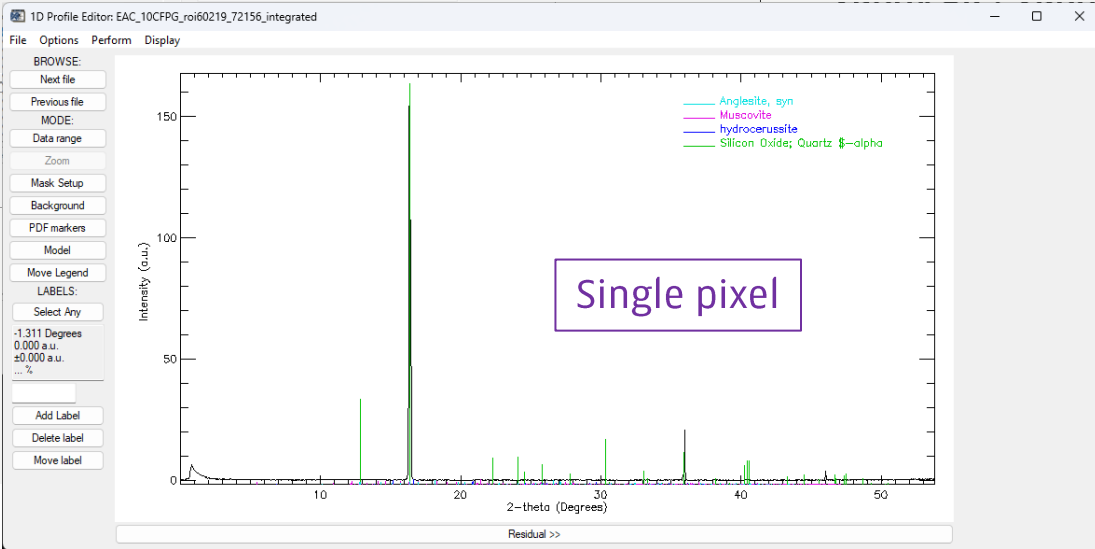
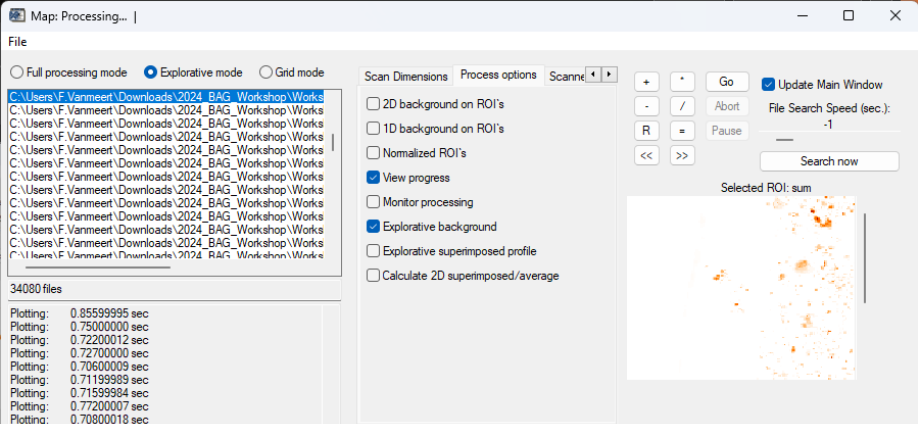
XRDUA BP + XRDUA 1D

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, ...).



3. Identifying compounds

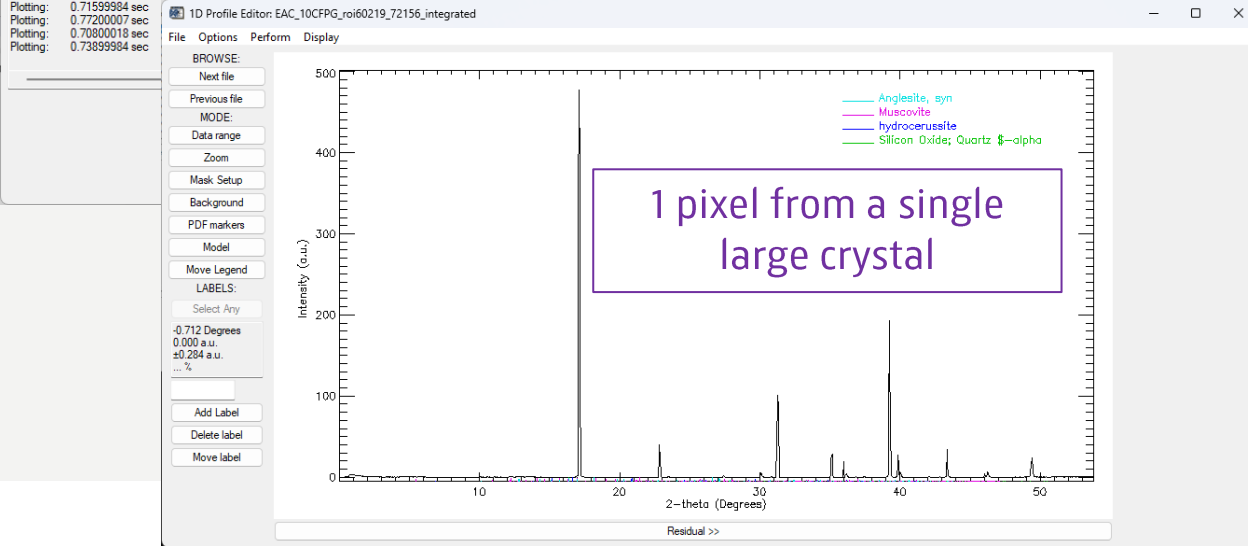
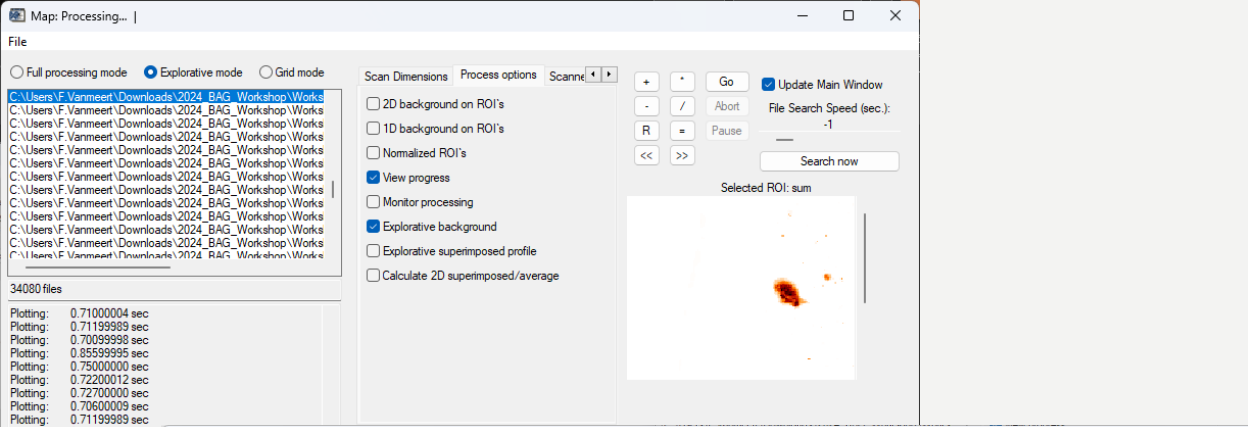


3. Identifying compounds

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!



3. Identifying compounds

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!

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

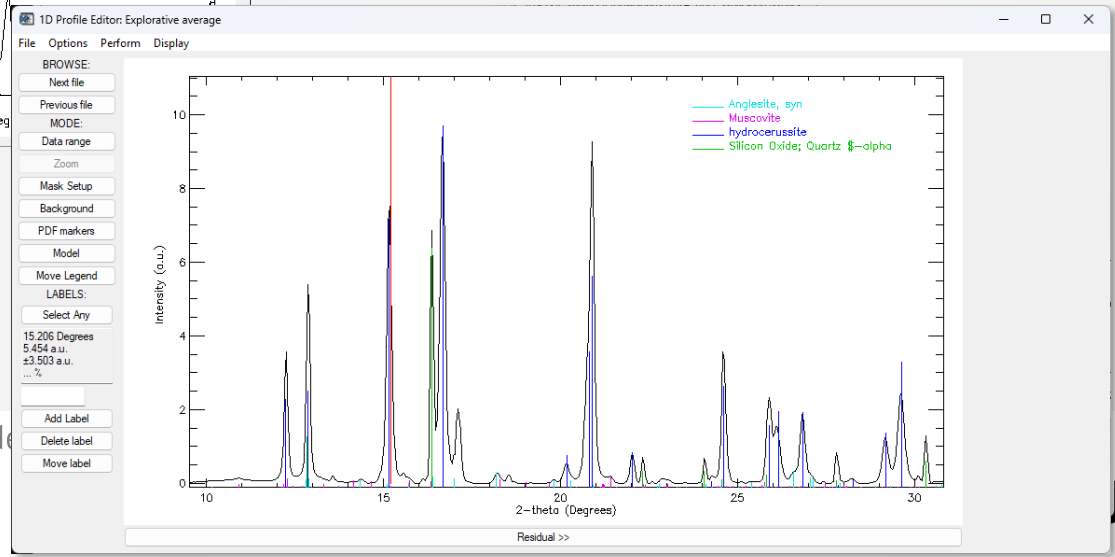
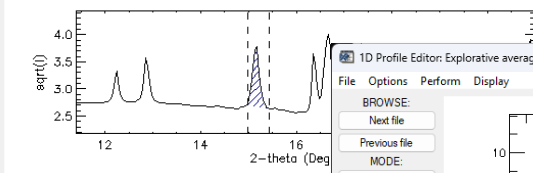
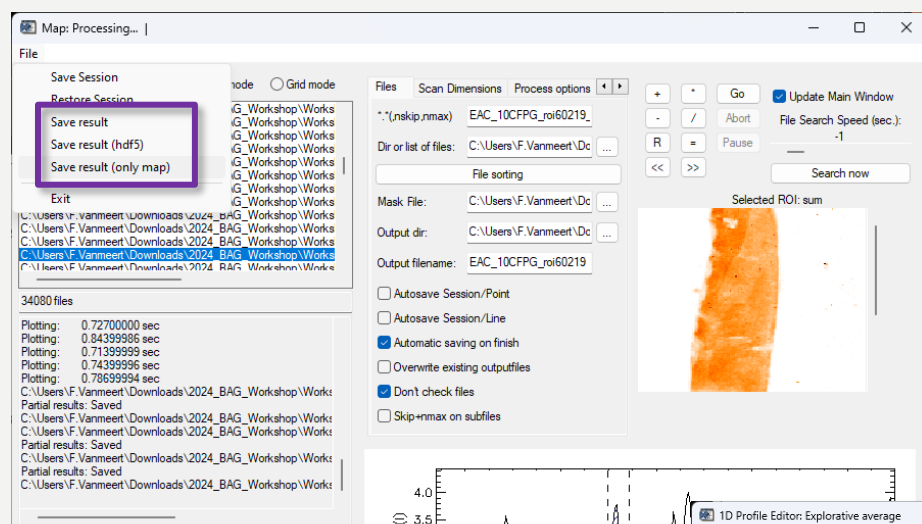
Identifying compounds

- 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 ;-)

XRDUA BP + XRDUA 1D

Saving ROI images

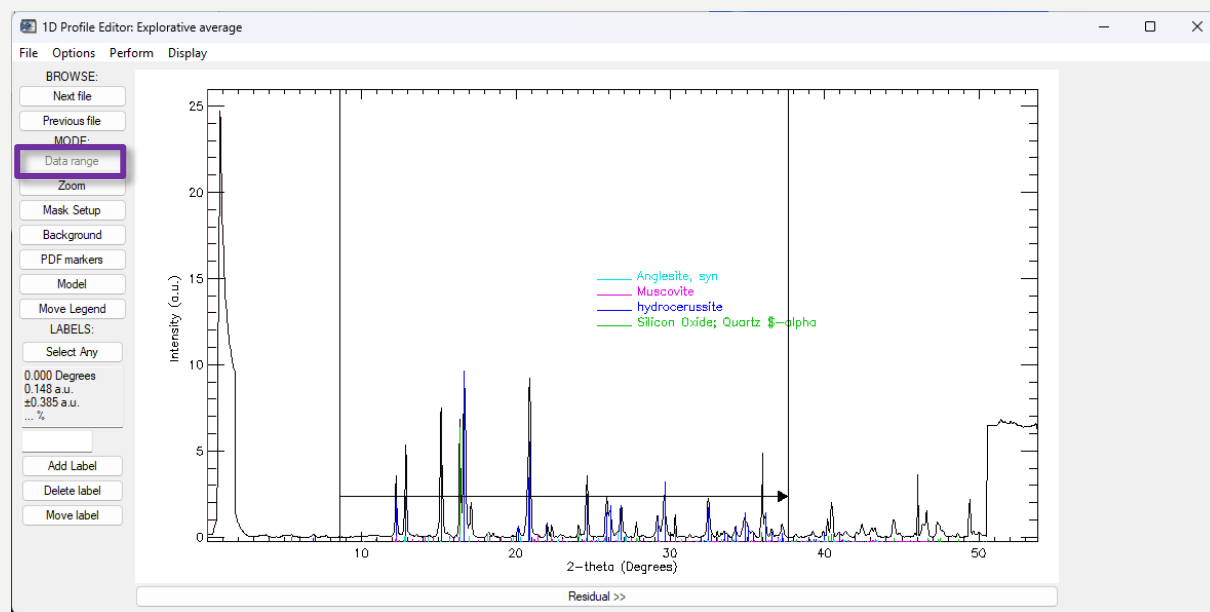
- Select ROI around peak of interest
- Check correct selection of peak in **XRDUA 1D** (when “Update Main Window” is checked)
- Save result (only map)



4. Saving ROI images

Fred

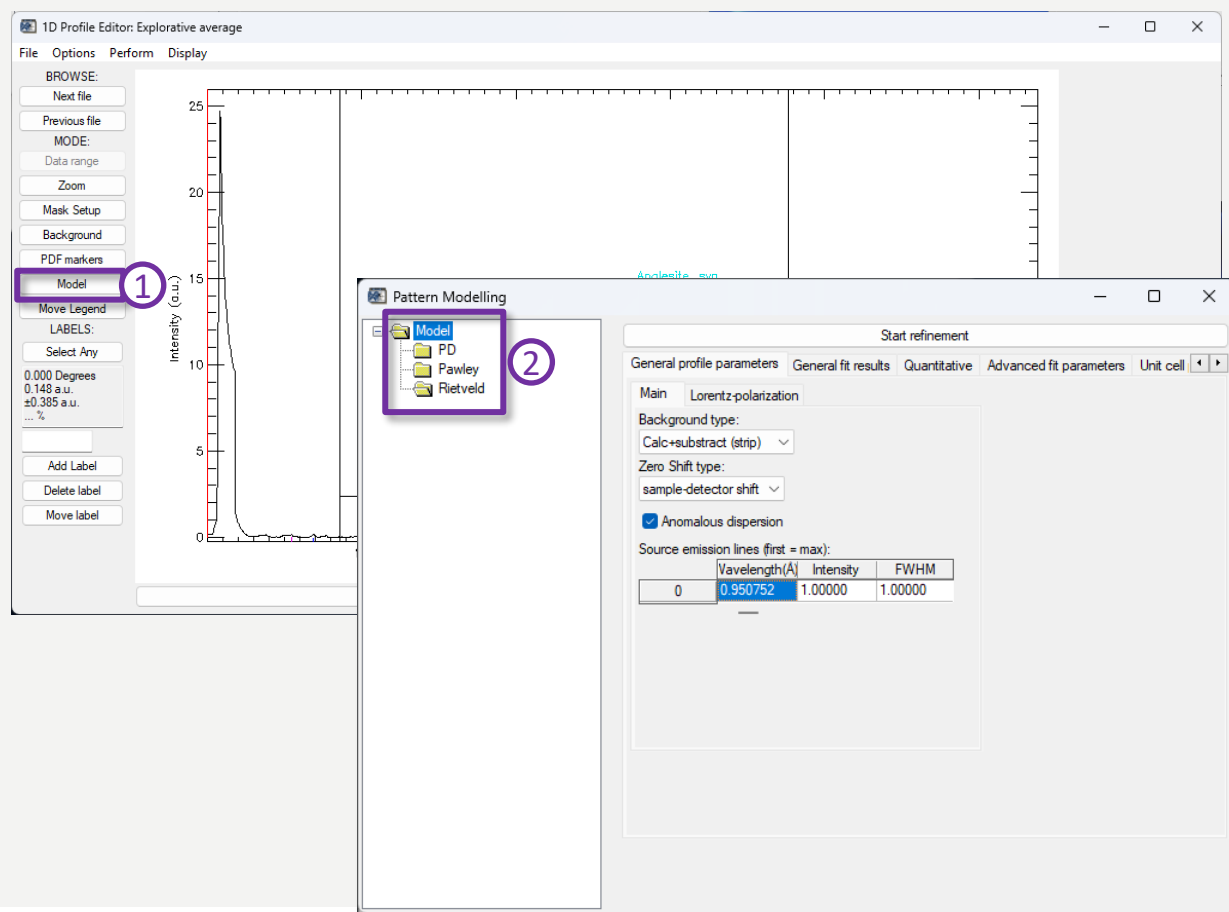
JM



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)

Frederik Vanmeert - 2024 Heritage BAG Workshop

5. Creating a Fit Model

RJKS MUSEUM

Map: Processing... |

File

Full processing mode
 Explorative mode
 Grid mode

C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works
 C:\Users\F.Vanmeert\Downloads\2024_BAG_Workshop\Works

34080 files (progress: 34080/34080)

Make Plotting window: 2.7539999 sec
 Plotting: 0.83799982 sec
 Image scaling 116.640%
 Make Plotting window: 3.0060000 sec
 Plotting: 0.88700008 sec
 Intensity scaling at 100.000%
 Make Plotting window: 3.9419999 sec
 Plotting: 1.0590000 sec
 Plotting: 0.91100001 sec
 Plotting: 0.85399985 sec
 Plotting: 0.84999990 sec
 Plotting: 0.82099986 sec
 Plotting: 0.89499998 sec

Scan Dimensions Process options Scanne:

2D background on ROI's
 1D background on ROI's
 Normalized ROI's
 View progress
 Monitor processing
 Explorative background
 Explorative superimposed profile
 Calculate 2D superimposed/average

 Update Main Window
 File Search Speed (sec.):
 -1

Selected ROI: sum

2 Click

3 ENTER

4 ENTER

sqrt(I)

2-theta (Degrees)

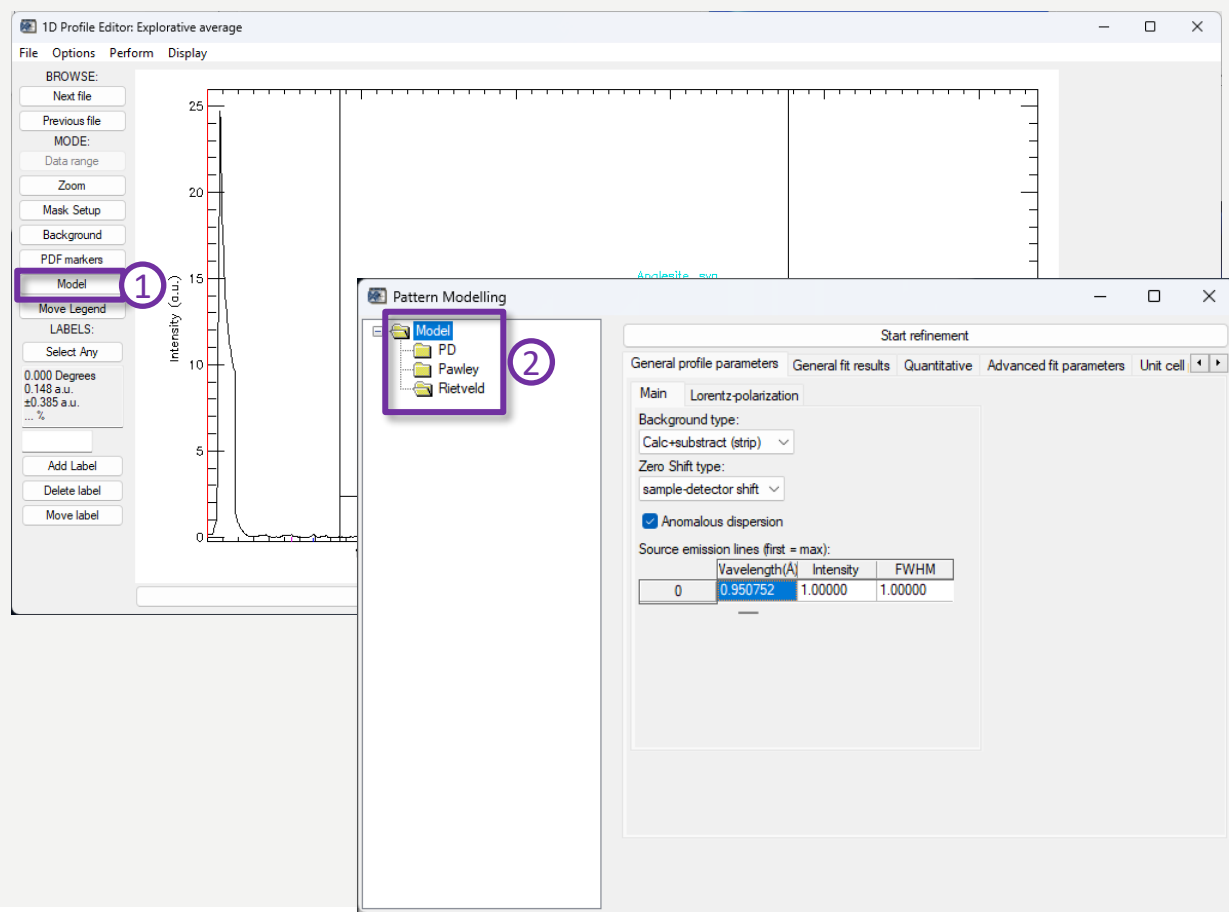
XRDUA BP

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

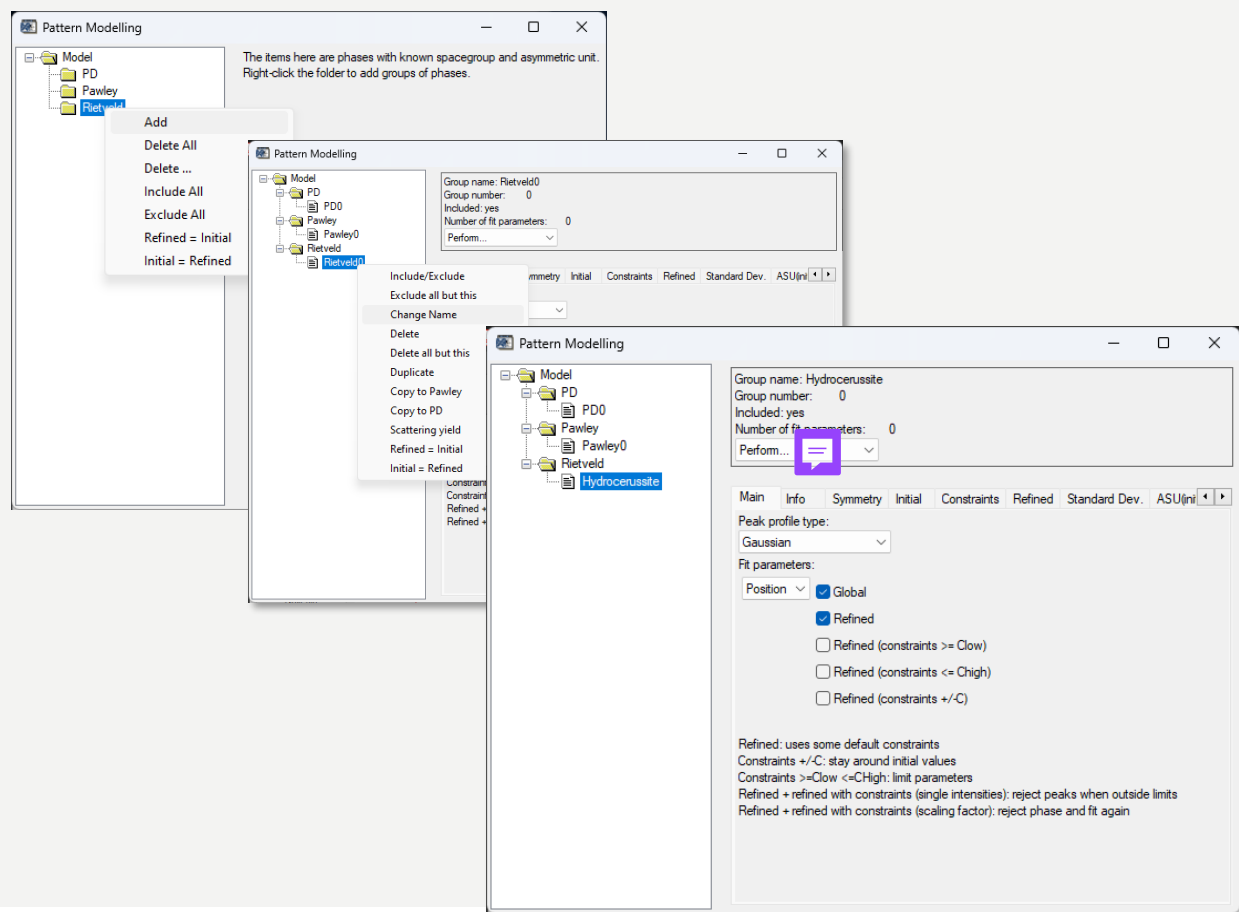
1. Model

- 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



- **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)



Model

Working with fitting groups

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



Frederik Vanmeert - 2024 Heritage BAG Workshop

5. Creating a Fit Model

RIJKS MUSEUM

Pattern Modelling

Model

- PD
- PDO
- Pawley
- Pawley0
- Rietveld
- Rietveld0
- Hydrocussite

Group name: Hydrocussite
 Group number: 0
 Included: yes
 Number of fit parameters: 3

Perform...
 Perform...
 Load Structure File
 Save Structure File
 Add ASU position

Delete Peaks With...
 Delete Small Peaks...

Reset Unit Cell
 Recalculate HKL

Save Peak param...
 Save FWHM...
 Save SNR...
 Save Peaks as PDF...

	k	l	m	FWHM_L	
0.00000	1.00000	6.00000	0.100000		
0.00000	-2.00000	6.00000	0.100000		
0.00000	6.00000	2.00000	0.100000		
0.00000	4.00000	6.00000	0.100000		
0.00000	-5.00000	6.00000	0.100000		
5	1.00000	0.00000	7.00000	6.00000	0.100000

Initial Constraints Refined Standard Dev. ASU(init) ASU(constr) ASU(ref) ASU(SD)

Ref = fixed

Global position parameters:

	dlist(mm)	a	b	c	alpha	beta	gamma
0	0.00000	5.24650	5.24650	23.7020	90.0000	90.0000	120.000

Global intensity parameters:

	scaling
0	0.00000

Global FWHM parameters:

	U	V	W	IG
0	0.00000	0.00000	0.0110000	0.00000

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

0.00000

This value will be refined by the model

5.24650

These values will NOT be refined by the model

0.10000

This site is an interface to a crystal structure database that includes every structure published in the American Mineralogist, The Canadian Mineralogist, European Journal of Mineralogy and Physics and Chemistry of Minerals, as well as selected datasets from other journals. The database is maintained under the care of the Mineralogical Society of America and the Mineralogical Association of Canada, and financed by the National Science Foundation.

hydrocerussite Mineral

Author

Chemistry Search

Cell Parameters and Sym

Diffraction Search

General Search Search Tips

Search Reset

Logic interface AND OR

Viewing (About File Formats) amc long form amc short form cif

Download amc cif diffraction data

People



Number of Files downloaded since Apr 1, 2003: 1180206959

Data Last Updated: January 08, 2024

Web Page Last Updated: July 31, 2018

This page has been accessed 4640665 times.

Also see our [complete list of minerals](#) and [complete list of authors](#).

This material is based upon work supported by the National Science Foundation under Grant Nos. EAR-0112782, and EAR-0621250. Any conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the National Science Foundation.

Should the use of the database require a citation, then please use: Downs, R.T. and Hall-Wallace, M. (2003) The American Mineralogist Crystal Structure Database. American Mineralogist 88, 247-250. ([pdf file](#))

Contact [Robert T. Downs](#) for suggestions and corrections.

Hydrocerussite

Siidra O, Nekrasova D, Depmeier W, Chukanov N, Zaitsev A, Turner R

Acta Crystallographica B74 (2018) 182-195

Hydrocerussite-related minerals and materials: structural principles, chemical variations and infrared spectroscopy

Locality: synthetic

_database_code_amcscd 0020727

5.257 5.257 23.636 90 90 120 R-3m

atom	x	y	z	occ	Uiso	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Pb1	0	0	.21570	.0271	.0249	.0249	.0314	.0125	0	0	0
Pb2	.9153	-.9153	.00191 1/6	.0286	.0337	.0337	.0279	.0238	-.0015	.0015	
C	1/3	2/3	.2337	.031	.012	.012	.068	.006	0	0	
O	.8083	-.8083	-.0979	.038	.032	.032	.063	.026	-.001	.001	
OH	0	0	.3124	.070	.090	.090	.030	.045	0	0	

[Download AMC data \(View Text File\)](#)

[Download CIF data \(View Text File\)](#)

[Download diffraction data \(View Text File\)](#)

[View JMOL 3-D Structure \(permalink\)](#)

Hydrocerussite

Martinetto P, Anne M, Dooryhee E, Walter P, Tsoucaris G

Acta Crystallographica C58 (2002) i82-i84

Synthetic hydrocerussite, 2PbCO₃*Pb(OH)₂ by X-ray powder diffraction

Locality: synthetic

_database_code_amcscd 0010324

5.2465 5.2465 23.702 90 90 120 R-3m

atom	x	y	z	occ	Uiso
Pb1	0	0	.21510	.0172	
Pb2	.9158	-.9158	.0016 1/6	.0215	
C	0	0	.4304	.028	
O1	.8568	-.8568	.4318	.028	
OH2	-.293	.293	.0200 1/3	.010	

[Download AMC data \(View Text File\)](#)

[Download CIF data \(View Text File\)](#)

[Download diffraction data \(View Text File\)](#)

[View JMOL 3-D Structure \(permalink\)](#)

Download in:

Multiple datasets can be concatenated into a single downloadable file by selecting the datasets and then clicking

Multiple datasets can be downloaded as individual files inside a ZIP archive by selecting the datasets and then clicking

Total number of retrieved datasets: 4

View in [amc](#)longform, download in [amc](#)

[Return to AMCSD Home Page](#)

Pattern Modelling

Model

- PD
 - PDO
 - Pawley
 - Pawley0
 - Rietveld
 - Hydrocussite

Group name: Hydrocusite
 Group number: 0
 Included: yes
 Number of fit parameters: 3
 Perform... ③

Main Info Symmetry Initial Constraints Refined Standard Dev. ASU(init) ASU(constr) ASU(ref) ASU(SD)

Cell color: Gray = no parameter, Red = fixed ①

ASU parameters:

	Natoms	Name	Z	Wyck	x	y	z	SOF	Biso
0	1	Pb1	Pb2+	c (0, 0, x)	0.00000	0.00000	0.215100	1.00000	1.35810
1	1	Pb2	Pb2+	8h (x, x, y)	0.915800	0.0842000	0.00160000	0.166700	1.69760
2	1	C	C4+	c (0, 0, x)	0.00000	0.00000	0.430400	1.00000	2.21080
3	1	O1	O2-	8h (x, x, y)	0.856800	0.143200	0.431800	1.00000	2.21080
4	1	O-H2	O2-H1+	8h (x, x, y)	0.707000	0.293000	0.0200000	0.333300	0.789600

Unit cell properties:

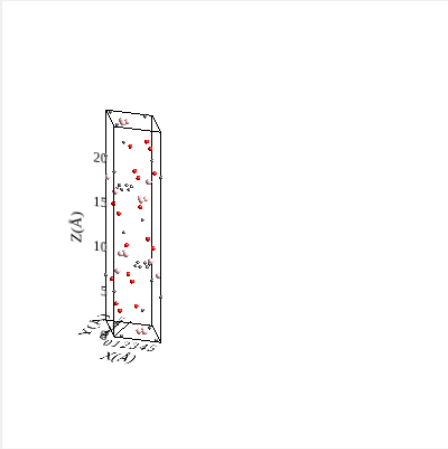
Charge: 0.00 ②

Standard weights:

- Pb2+: 9.00
- C4+: 6.00
- O2-: 24.00
- H1+: 6.00

Weight%:

- Pb2+: 80.1 (%)
- C4+: 3.1 (%)
- O2-: 16.5 (%)
- H1+: 0.3 (%)



+ - +Z -Z XY YZ XZ Reset Rebuild Axis on/off

Unconstrained Slow zoom Save Image...

Model

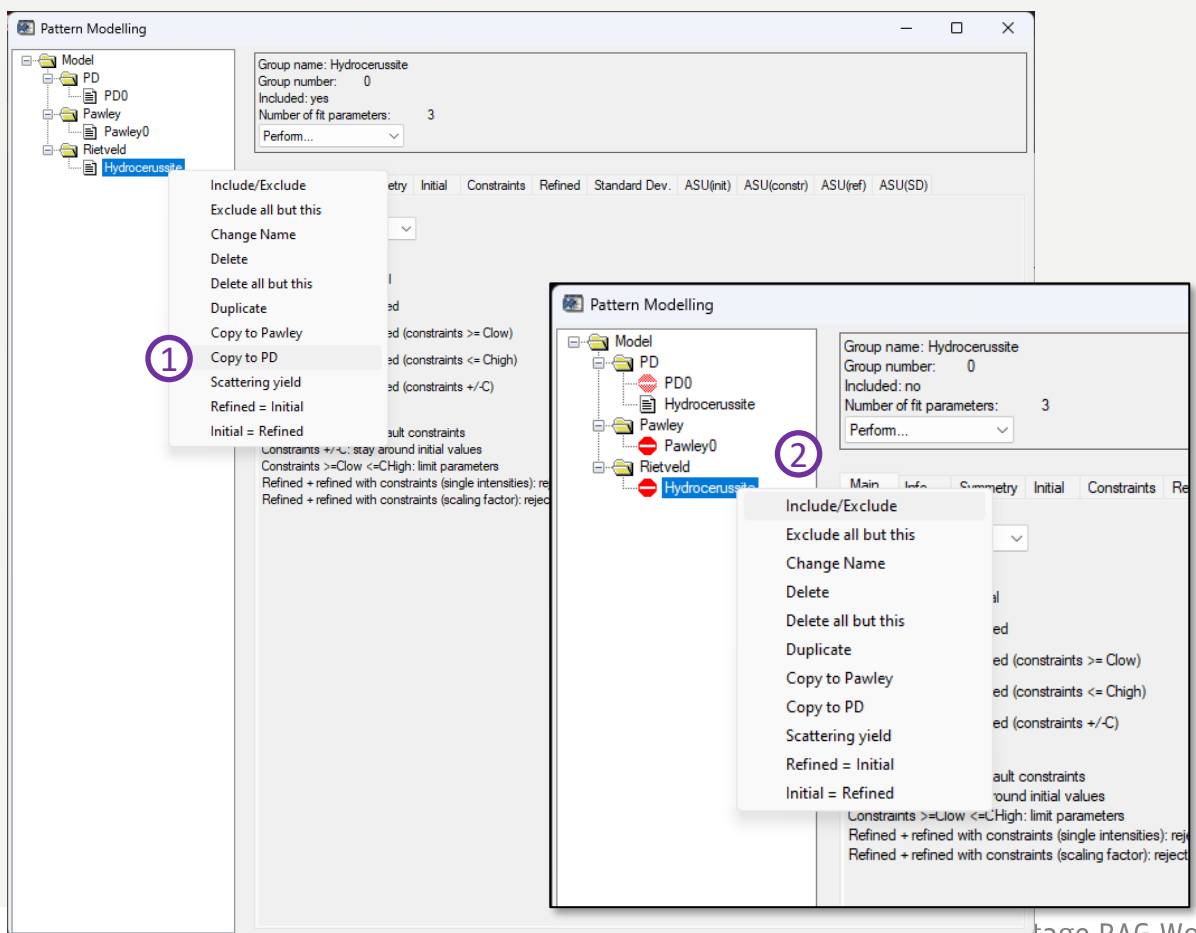
Rietveld Group

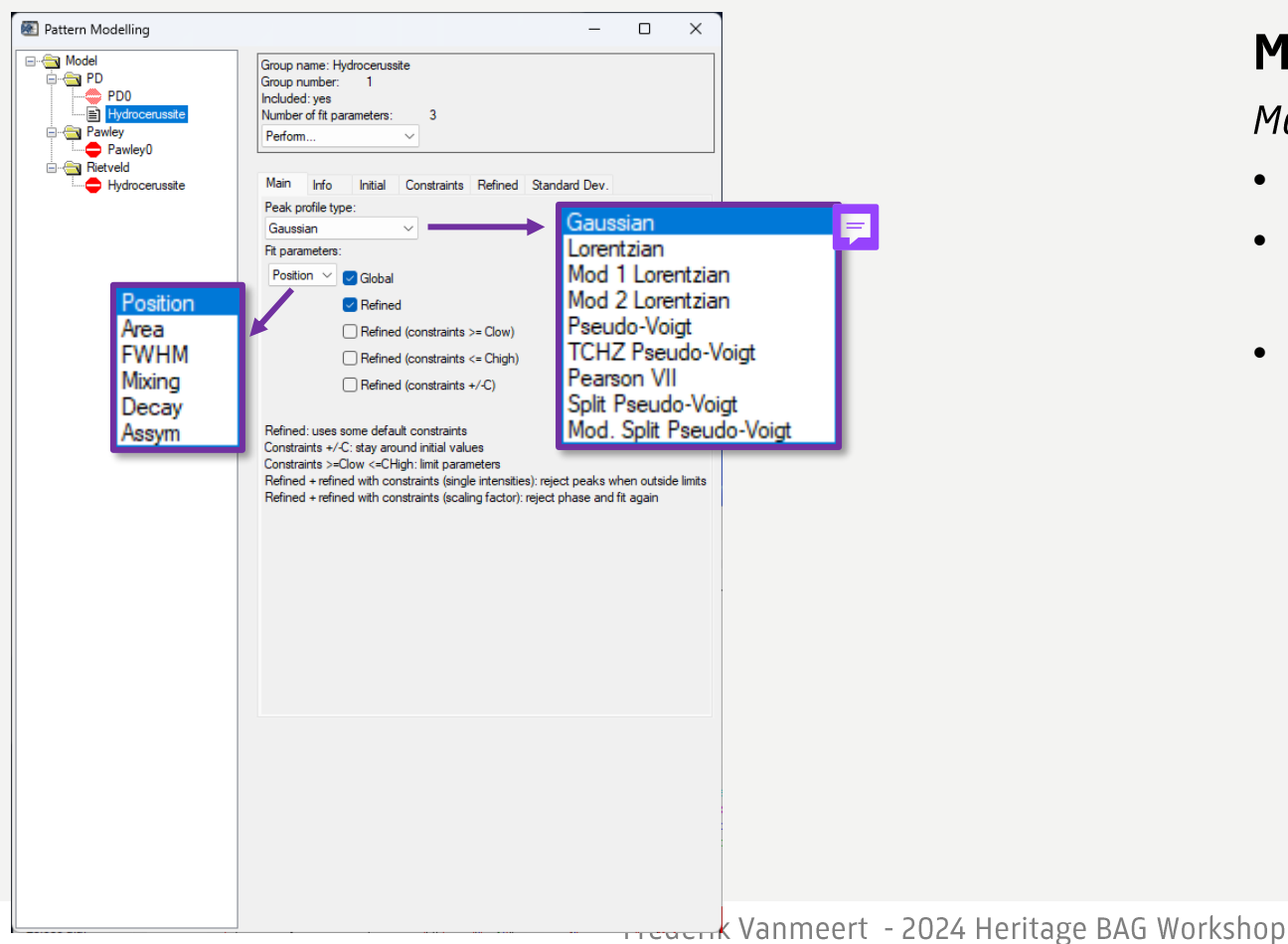
- ASU(init)
 - Atomic structure information
 - Charges are default, unless the structure is loaded from a XRDUA .cel file
- Set proper charges
 - Z, e.g.,
 - Pb: Pb2+
 - OH-: O2-H1+
 - H2O: O2-H1+2
 - Charge: 0.00
- You can modify and save the structure as a .cel file (Perform ...)

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

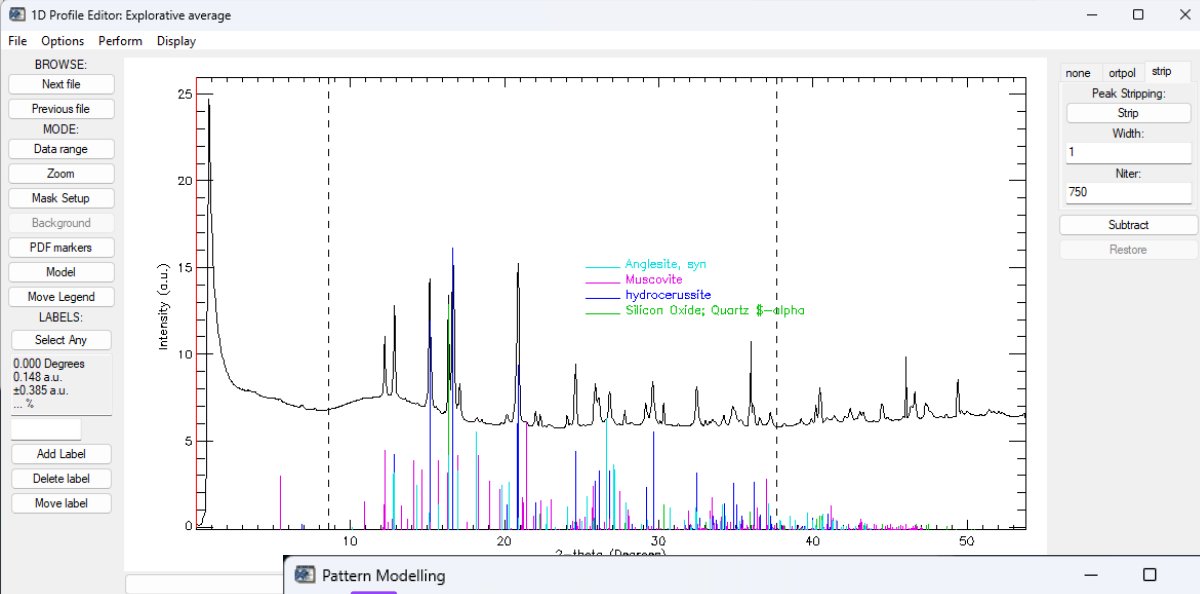




Model

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

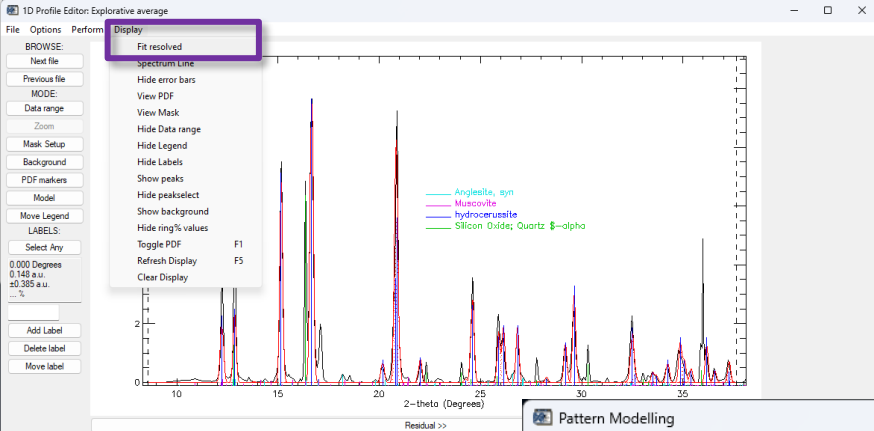


XRDUA 1D + Model

First Fitting


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


	Wavelength(Å)	Intensity	FWHM
0	0.950752	1.00000	1.00000



XRDUA 1D + Model


Find good starting values

1. Subtract background again 
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 distance 
- scaling
- W: Cagliotti theta independent peak width parameter

Group name: Hydrocerussite
 Group number: 1
 Included: yes
 Number of fit parameters: 3
 Perform...

Main Info Initial Constraints **Refined** Standard Dev.

Cell color: Gray = not used, Red = fixed 

Peak parameters:

	2-theta	I	FWHM_L
0	12.2307	98721.5	0.100000
1	12.8672	136051.	0.100000
2	13.8232	1164.13	0.100000
3	15.1522	545168.	0.100000
4	16.6657	906313.	0.100000
5	20.1680	93719.7	0.100000

Global position parameters:

	ddist (mm)	isodefom
0	0.00530383	0.00000

Global intensity parameters:

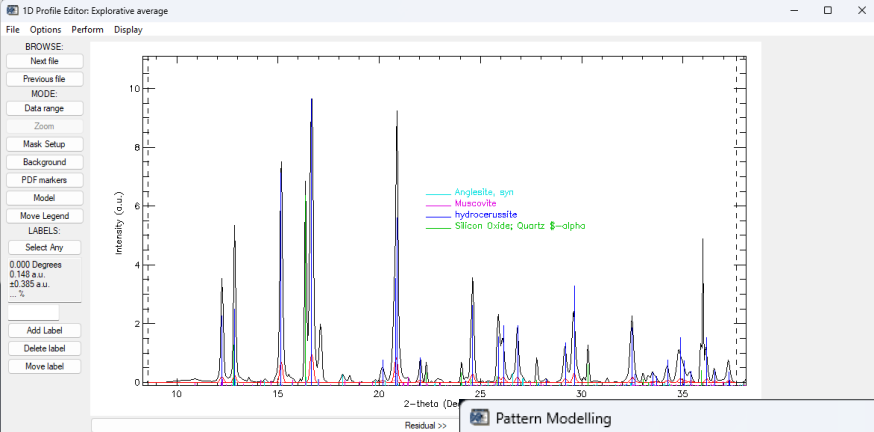
	scaling
0	8.70519e-08

Global FWHM parameters:

	U	V	W	IG
0	0.00000	0.00000	0.0325559	0.00000

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

Pattern Modelling

Model

- PD0
- Pawlk
- P
- Rietv
- H

Group name: Hydrocrussite
Group number: 1
Included: yes

Parameters: 3

Initial Constraints Refined Standard Dev.

ay = not used, Red = fixed

Parameters:	2-theta	I	FWHM_L
	12.2307	98721.5	0.100000
	12.8672	136051.	0.100000
2	13.8232	1164.13	0.100000
3	15.1522	545168.	0.100000
4	16.6657	906313.	0.100000
5	20.1680	93719.7	0.100000

Global position parameters:

	dlist (nm)	isodefom
0	0.00530383	1.00000

Global intensity parameters:

	scaling
0	8.70519e-09

Global FWHM parameters:

	U	V	W	IG
0	0.00000	0.00000	0.0325559	0.00000

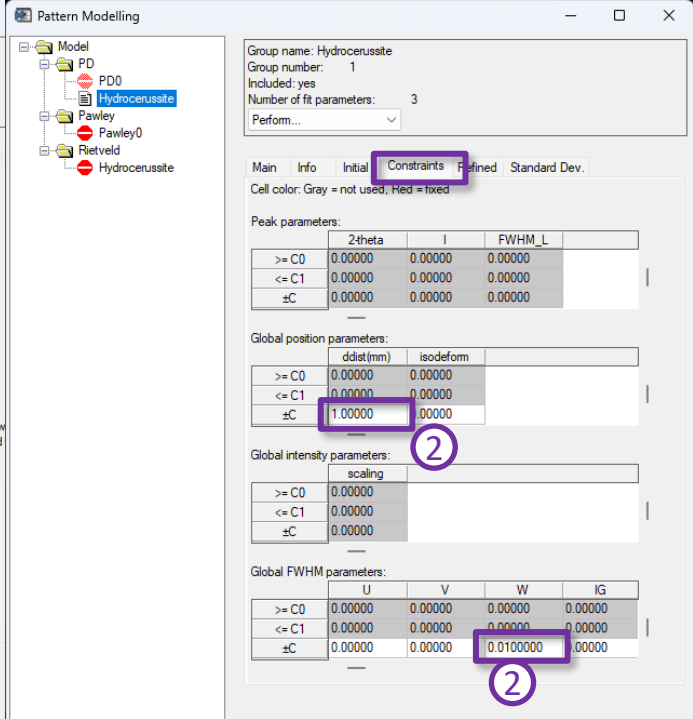
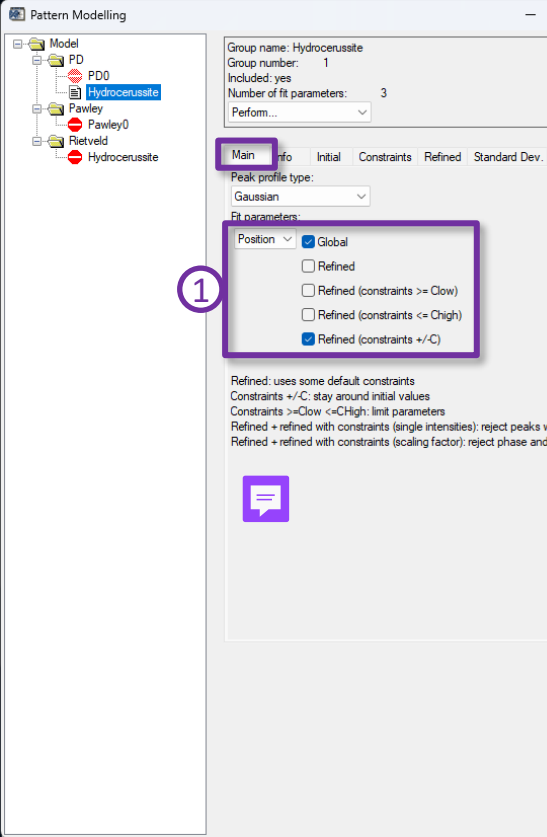
Context menu for PD0:


- Include/Exclude
- Exclude all but this
- Change Name
- Duplicate
- Delete
- Delete all but this
- Refined = Initial
- Initial = Refined

5. Creating a Fit

hop

RIJKS MUSEUM

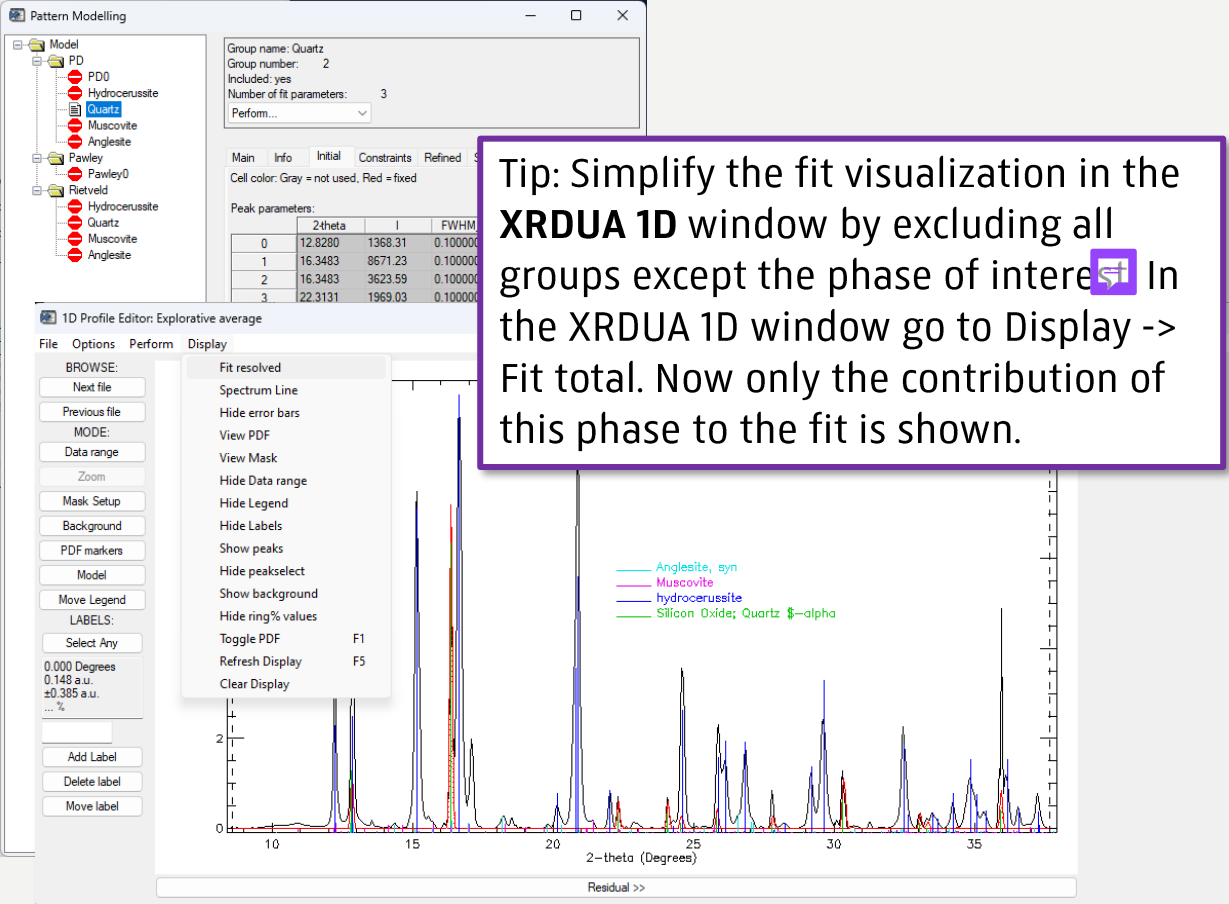


Tip: Rerun the fit to check that no mistake was made (do not forget to restore the background) 

XRDUA 1D + Model

Set constraints

- It's best to constrain the position and width to avoid unrealistic values
- Refined (constraints +/-C)
 - This constraints the value based on the initial value and a constant
 - 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



XRDUA 1D + Model

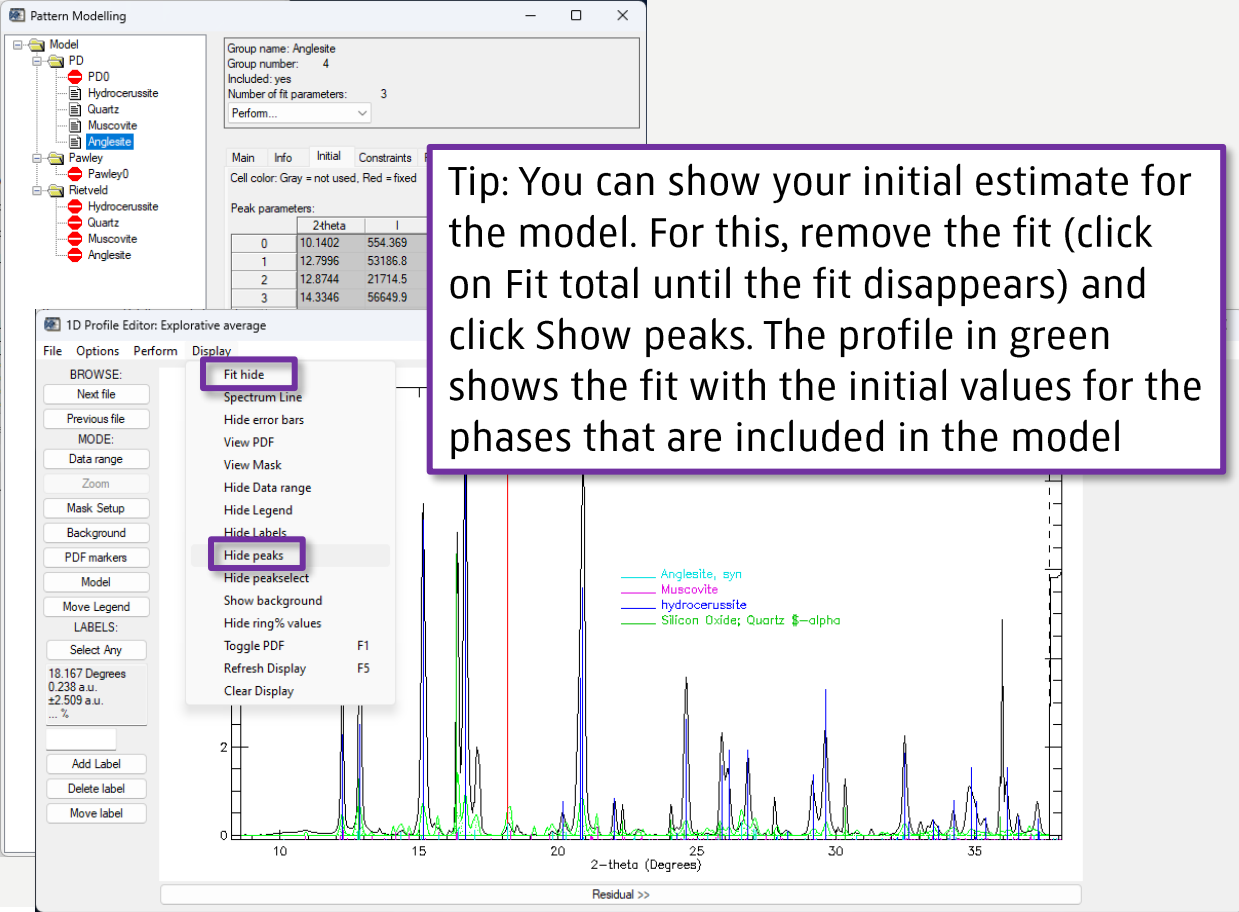
Add a new group for the next phase and repeat the procedure

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

Frederik Vanmeert - 2024 Heritage BAG Workshop

5. Creating a Fit Model

RJKS MUSEUM

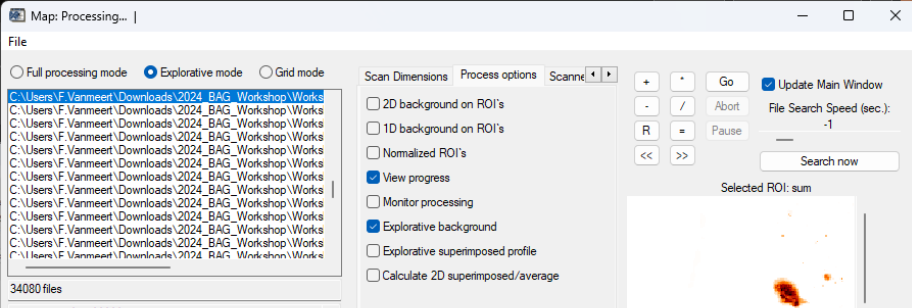


Tip: You can show your initial estimate for the model. For this, remove the fit (click on Fit total until the fit disappears) and click Show peaks. The profile in green shows the fit with the initial values for the phases that are included in the model

XRDUA 1D + Model

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

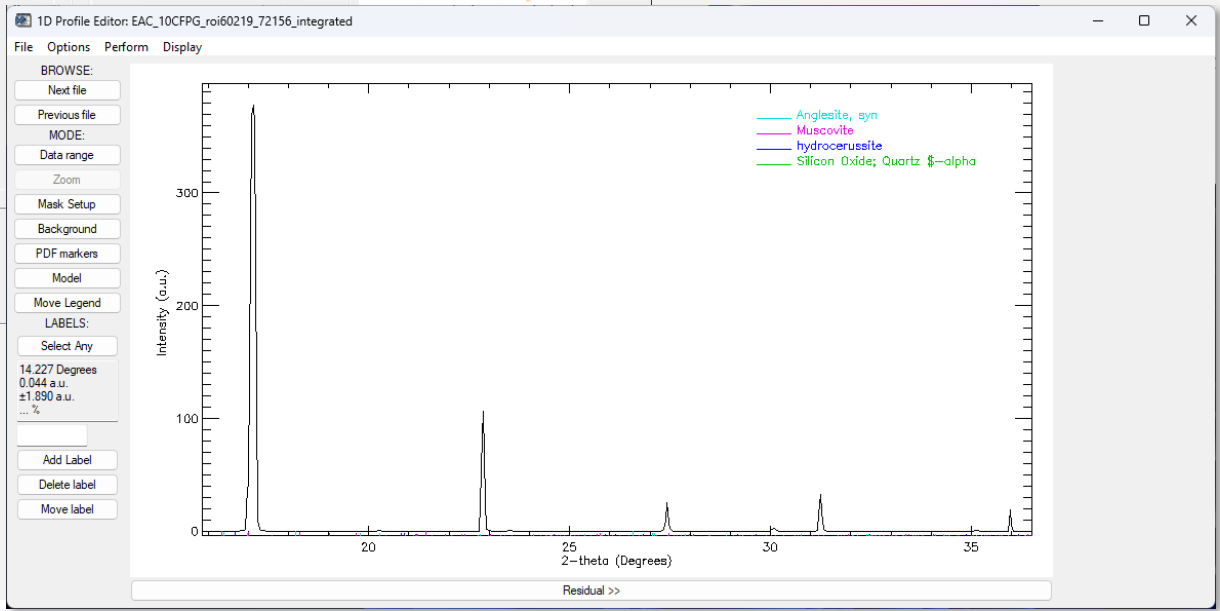


XRDUA 1D + Model

Unknown phase

34080 files

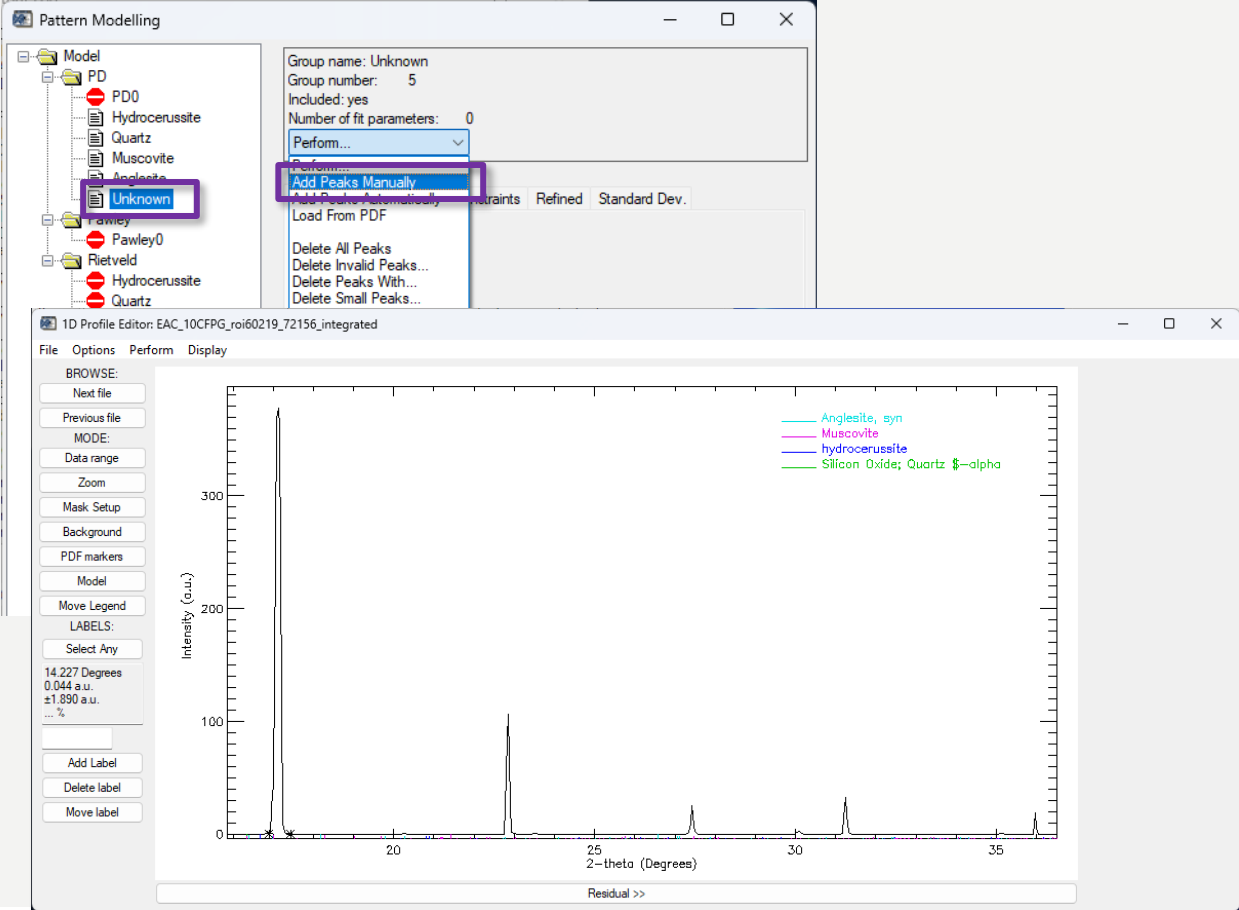
Plotting: 0.71000004 sec
 Plotting: 0.71199989 sec
 Plotting: 0.70099998 sec
 Plotting: 0.85599995 sec
 Plotting: 0.75000000 sec
 Plotting: 0.72200012 sec
 Plotting: 0.72700000 sec
 Plotting: 0.70600009 sec
 Plotting: 0.71199989 sec
 Plotting: 0.71599984 sec
 Plotting: 0.77200007 sec
 Plotting: 0.70800018 sec
 Plotting: 0.73899984 sec



Frederik Vanmeert - 2024 Heritage BAG Workshop

5. Creating a Fit Model

RIJKS MUSEUM



XRDUA 1D + Model

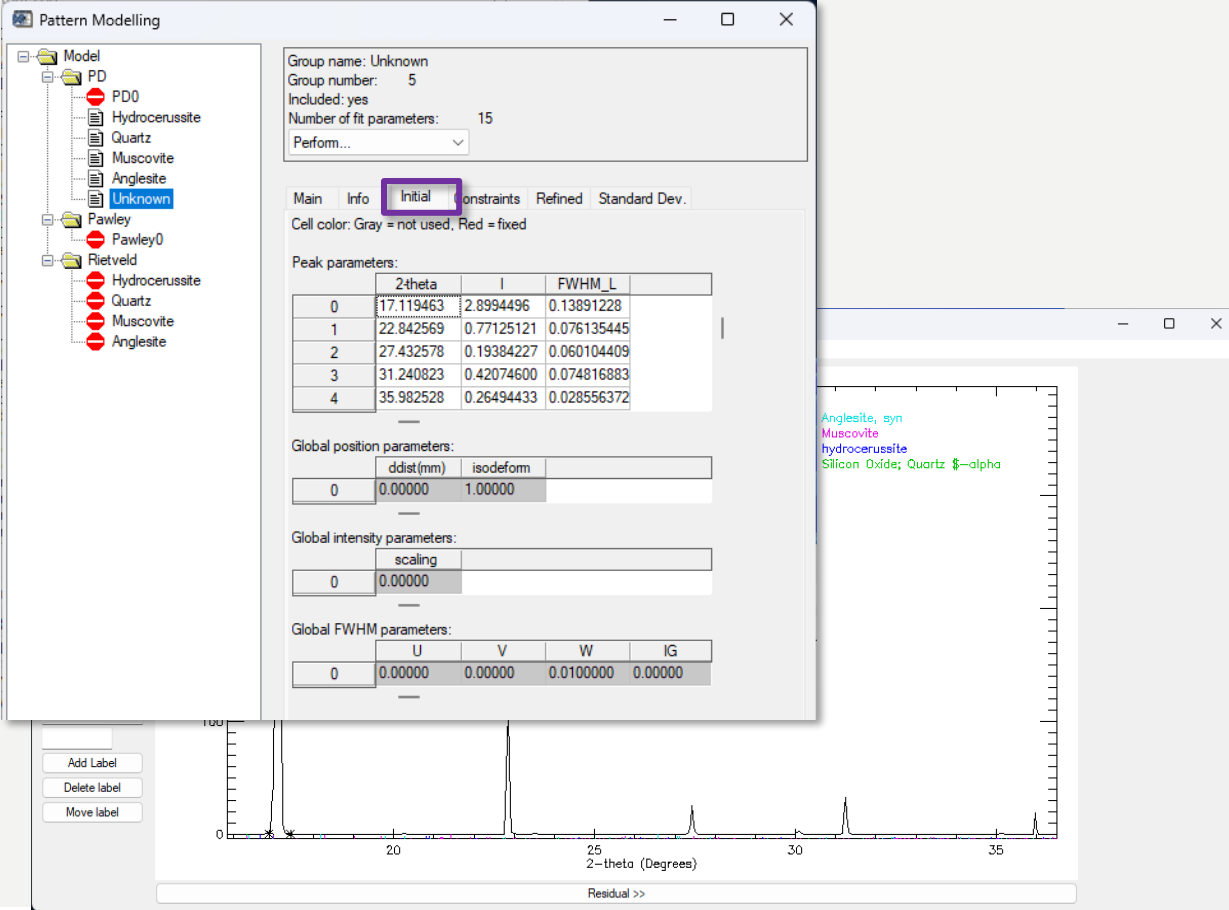
Unknown phase

- 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

Frederik Vanmeert - 2024 Heritage BAG Workshop

5. Creating a Fit Model

RJUKS MUSEUM



XRDUA 1D + Model

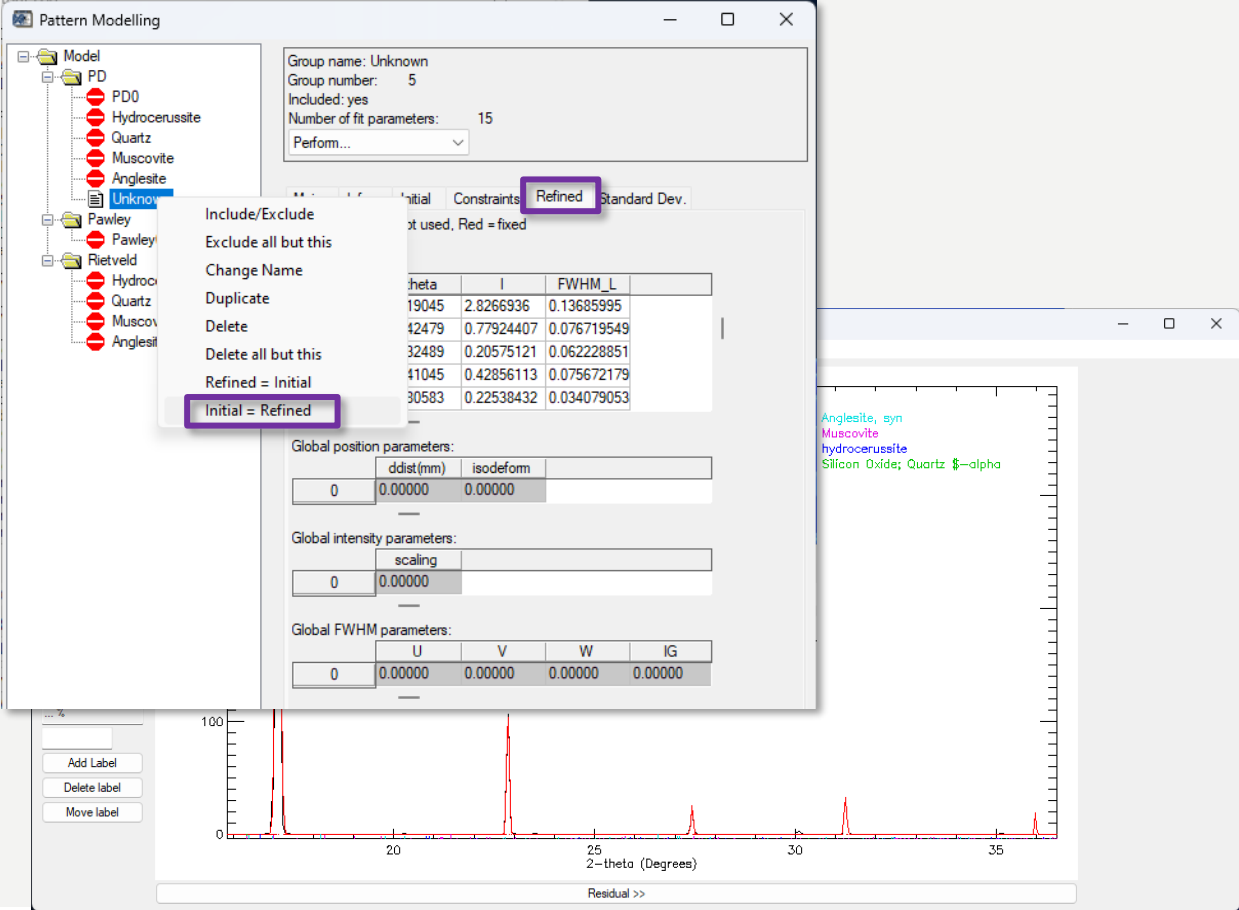
Unknown phase

- 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

Frederik Vanmeert - 2024 Heritage BAG Workshop

5. Creating a Fit Model

RJKS MUSEUM



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

Frederik Vanmeert - 2024 Heritage BAG Workshop

5. Creating a Fit Model

RJKS MUSEUM

Pattern Modelling

Group name: Unknown
Group number: 5
Included: yes
Number of fit parameters: 3

1

Main Info Initial Constr

Peak profile type:
Gaussian

Fit parameters:
Position Global
 Refined
 Refined (const...
 Refined (const...
 Refined (const...

Refined: uses some default const...
Constraints +/-C: stay around initi...
Constraints >=Clow <=CHigh: limit...
Refined + refined with constraints
Refined + refined with constraints

Model
PD
PDO
Hydrocerussite
Quartz
Muscovite
Anglesite
Unknown
Pawley
Pawley0
Rietveld
Hydrocerussite
Quartz
Muscovite
Anglesite

Pattern Modelling

Group name: Unknown
Group number: 4
Included: yes
Number of fit parameters: 6

2

Main Info Initial constraints Refined Standard Dev.

Cell color: Gray = not used, Red = fixed

Peak parameters:

	2 <theta< th=""> <th>I</th> <th>FWHM_L</th> </theta<>	I	FWHM_L
0	17.1190	2.82669	0.136860
1	22.8425	0.779244	0.0767196
2	27.4325	0.205751	0.0622289
3	31.2410	0.428561	0.0756722
4	35.9806	0.225384	0.0340791

Global position parameters:

	ddist(mm)	isodeform
0	0.00000	1.00000

Global intensity parameters:

	scaling
0	0.00000

Global FWHM parameters:

	U	V	W	IG
0	0.00000	0.00000	0.00000	0.00000

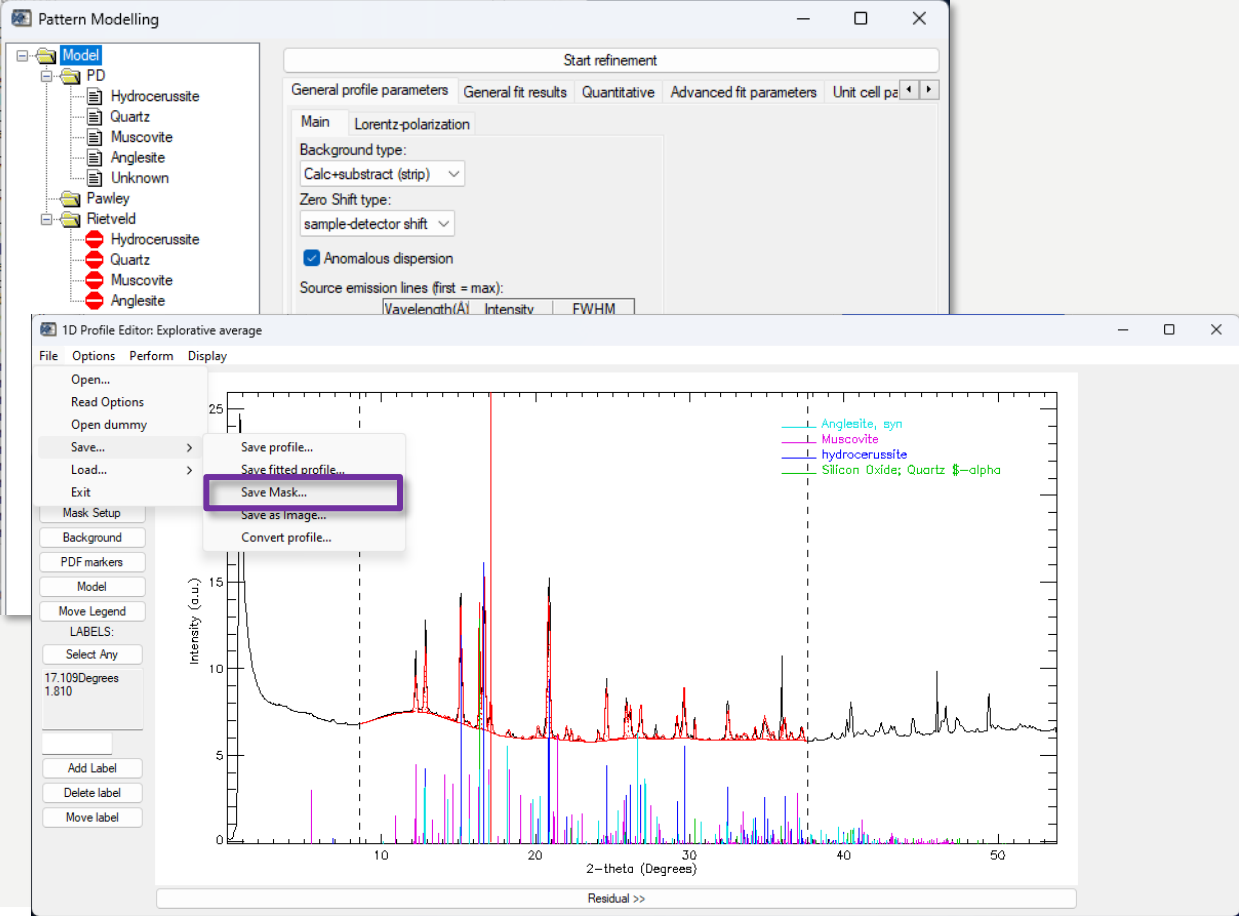
Note: We do not put the intensity to Global, since these values can strongly change for each signal depending on the hotspot intensity.

Model
PD
PDO
Hydrocerussite
Quartz
Muscovite
Anglesite
Unknown
Pawley
Pawley0
Rietveld
Hydrocerussite
Quartz
Muscovite
Anglesite

XRDUA 1D + Model

Unknown phase

- 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)
- 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 Position
 - 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**.

Frederik Vanmeert - 2024 Heritage BAG Workshop

5. Creating a Fit Model

RJKS MUSEUM

The screenshot shows the XRDUA BP software interface. The 'Full processing mode' radio button is selected (1). The 'Dir or list of files' field contains a directory path (2). The 'Mask File' field is set to a file path (3). The 'Update Main Window' checkbox is unchecked (4). The interface includes a file list, a 'Go' button, and a 'Search now' button. A plot of \sqrt{I} vs 2-theta (Degrees) is shown at the bottom, with a peak at approximately 17.5 degrees.

XRDUA BP

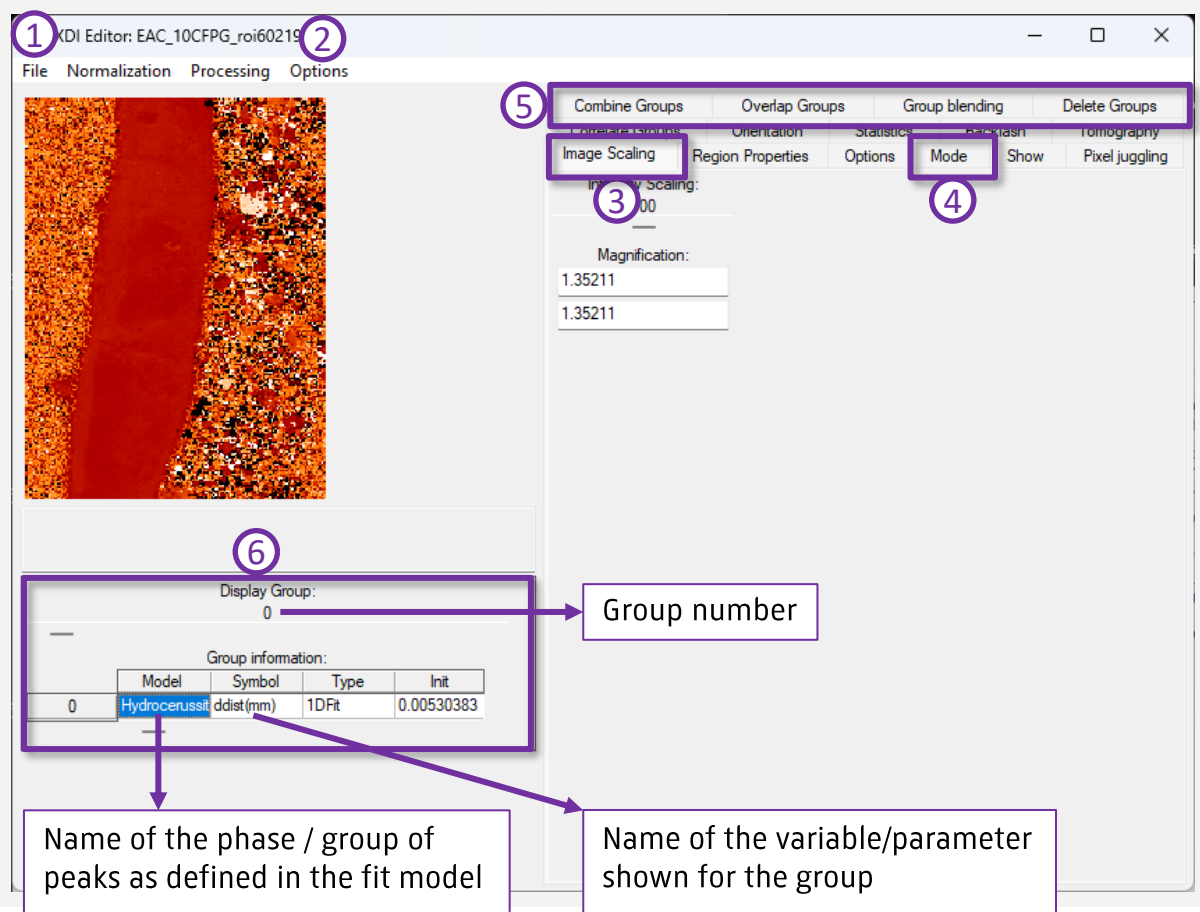
Full Processing with model

- 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

Frederik Vanmeert - 2024 Heritage BAG Workshop

6. Batch processing of fit model

RJKS MUSEUM



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

Frederik Vanmeert - 2024 Heritage BAG Workshop

File Normalization Processing Options

Combine Groups Overlap Groups Group blending Delete Groups
 Complete Groups Orientation Statistics Backlash Tomography
 Image Scaling Region Properties Options Mode Show Pixel juggling

Intensity Scaling: 40
 Magnification: 2
 2

Parameters
 Sort Options

1
 2

XDI parameters

Charsize (%size) : 1.00000
 Label index : 0
 Output rx : 4
 1D plot markers :
 Open formatted: unformatted
 Manual color table:
 Color index for borders : 0
 Color index for labels : 255
 HDF5 data : DiffTomo/NXdata/sinogr.
 HDF5 axis : DiffTomo/NXdata/xaxis

XLoadct

Done Help

Tables Options Function

Stretch Bottom 100
 Stretch Top 1.00000

Gamma Correction

0 - B-W LINEAR
 1 - BLUE/WHITE
 2 - GRN-RED-BLU-WHT
 3 - RED TEMPERATURE
 4 - BLUE/GREEN/RED/YELLOW
 5 - STD GAMMA-II
 6 - PRISM
 7 - RED-PURPLE
 8 - GREEN/WHITE LINEAR
 9 - GRN/WHT EXPONENTIAL
 10 - GREEN-PINK
 11 - BLUE-RED
 12 - 16 LEVEL
 13 - RAINBOW
 14 - STEPS
 15 - STERN SPECIAL

POSITION=[83,0], COORD=[0.00000mm,0.00000mm]
 FILEPOS,[83,0], FILENR.=83
 INT.=1.86639e-07

Display Group:
 1

Group information:

	Model	Symbol	Type	Init
0	Hydrocerusst	scaling	1DFit	8.70519e-09

XRDUA XDI

Looking at your results

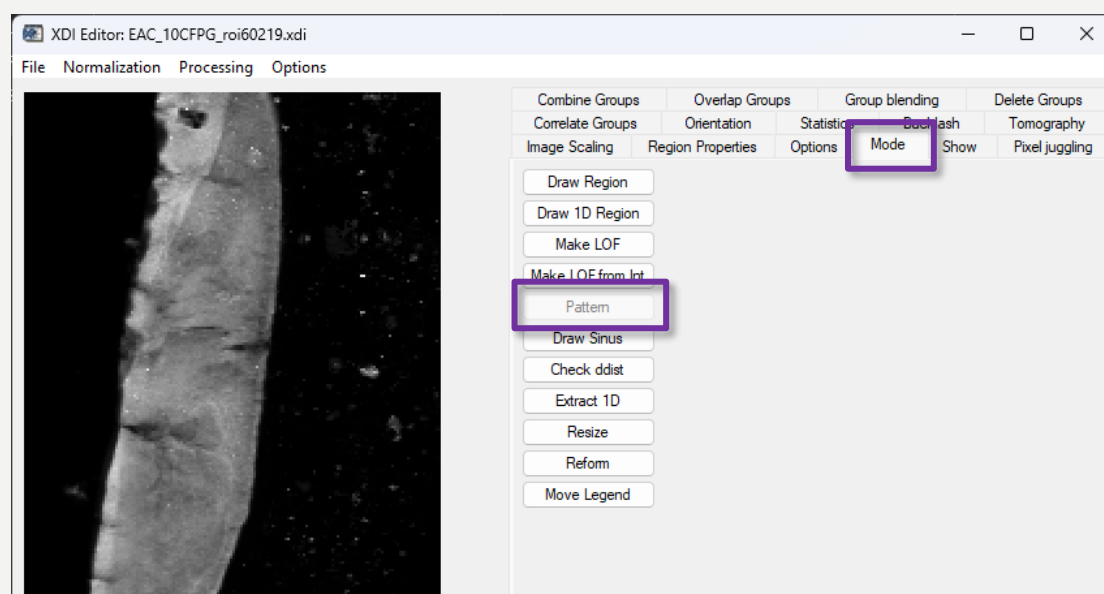
- Open .xdi file with **XRDUA XDI**
- 1. Change scaling (and intensity)
- 2. Option to change color
 - Go to Options → Parameters
 - Deselect Manual color table
 - Select color

Frederik Vanmeert

workshop

7. Results

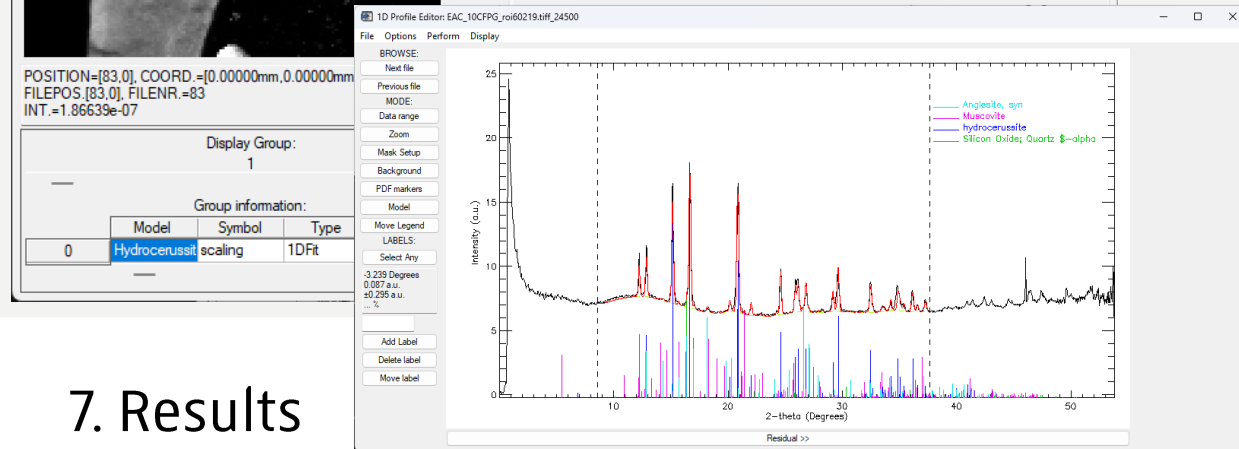
RJUKS MUSEUM



XRDUA XDI

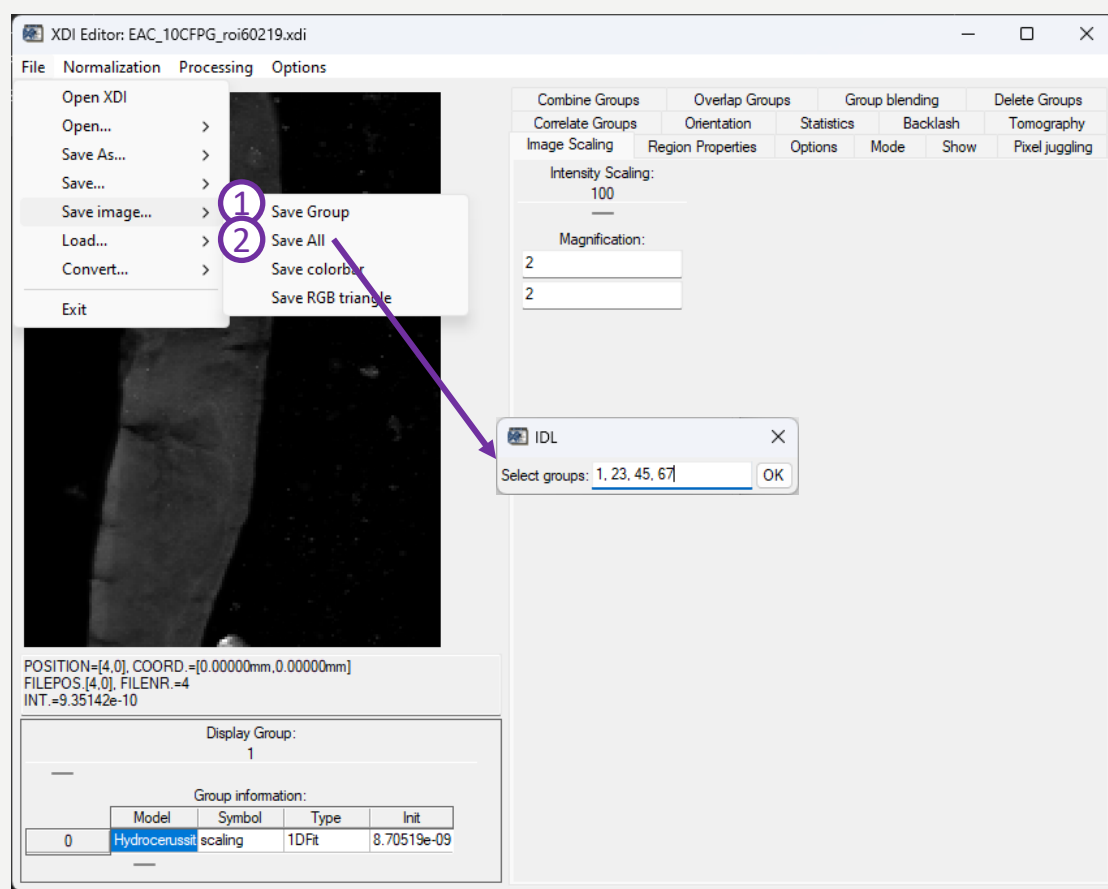
Verify results!

- 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!

7. Results



XRDUA XDI

Exporting results

- Results are exported using the current (intensity) scaling
- Single group
 - Go to the group that you want to save
 - Save Group
 - 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:

- [XRDU A Distribution Page](#)
Main XRDU A website with info, tutorials, and download link to latest version
- [XRDU A download | SourceForge.net](#)
Sourceforge page for XRDU A
- [NV5 | Customer Portal](#)
Portal to register and download IDL virtual machine needed to run XRDU A
- XRDU A 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

Frederik Vanmeert - 2024 Heritage BAG Workshop

Useful links

RIJKS MUSEUM