



• • • • • Testing • Research • Consulting

Workshop „Rietveld Refinement with Profex“

Lesson 4: Introduction to Profex - Phase Identification

Nicola Döbelin

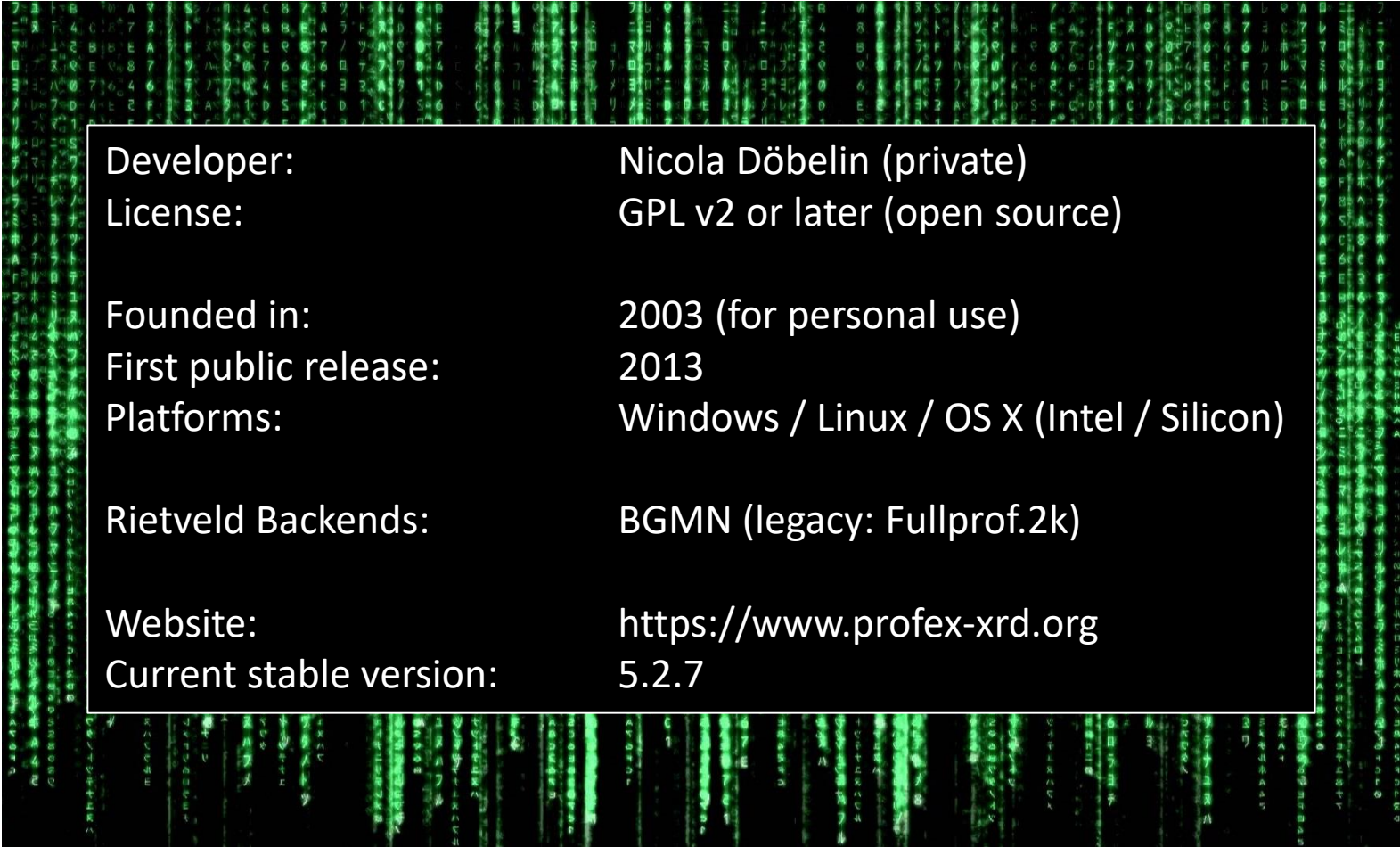
RMS Foundation, Switzerland

March 07-08, 2024

Forschungszentrum Jülich, Germany

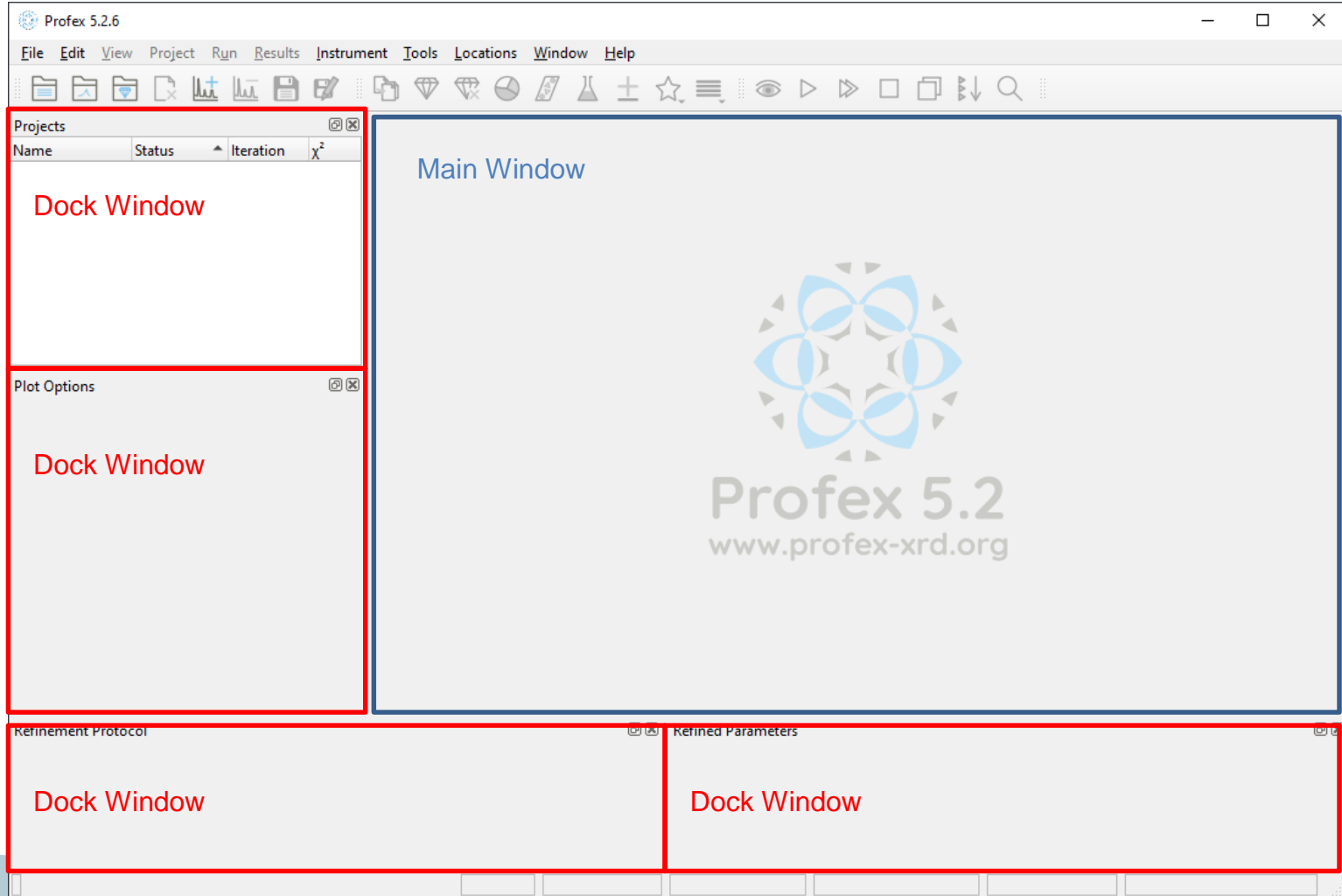


Facts about Profex



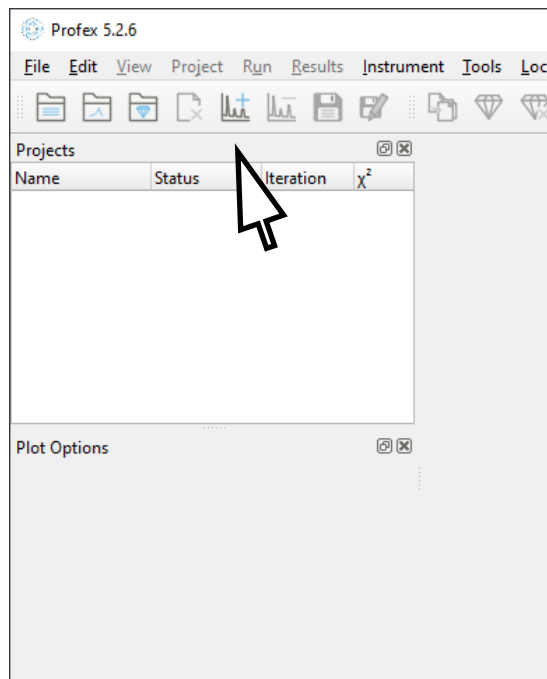
Developer:	Nicola Döbelin (private)
License:	GPL v2 or later (open source)
Founded in:	2003 (for personal use)
First public release:	2013
Platforms:	Windows / Linux / OS X (Intel / Silicon)
Rietveld Backends:	BGMN (legacy: Fullprof.2k)
Website:	https://www.profex-xrd.org
Current stable version:	5.2.7

Profex User Interface

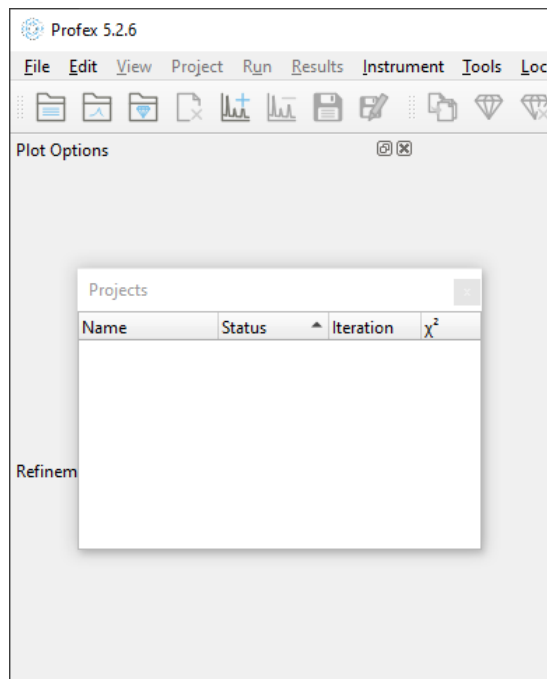


Profex User Interface

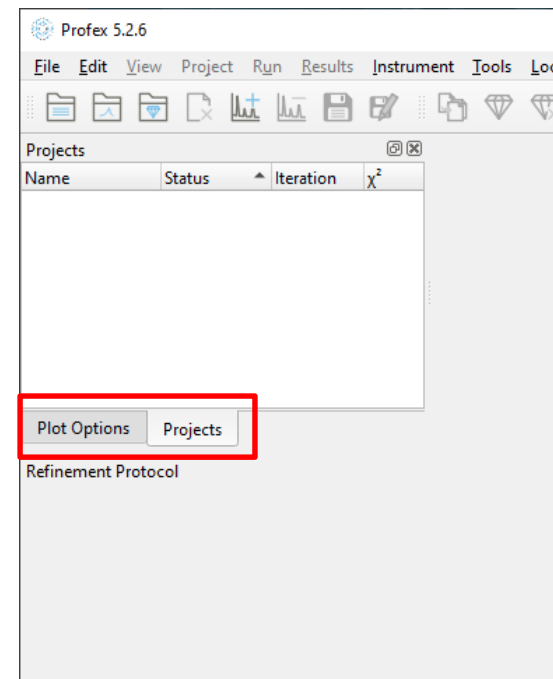
Docked



Floating

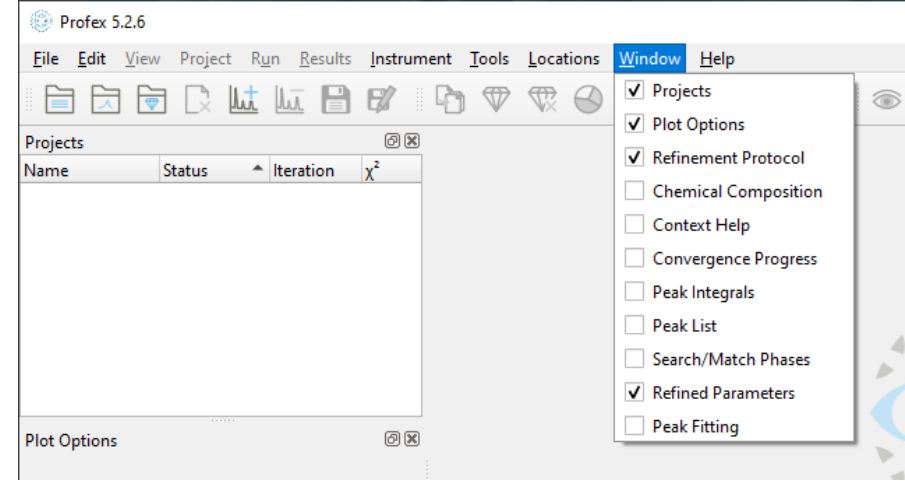
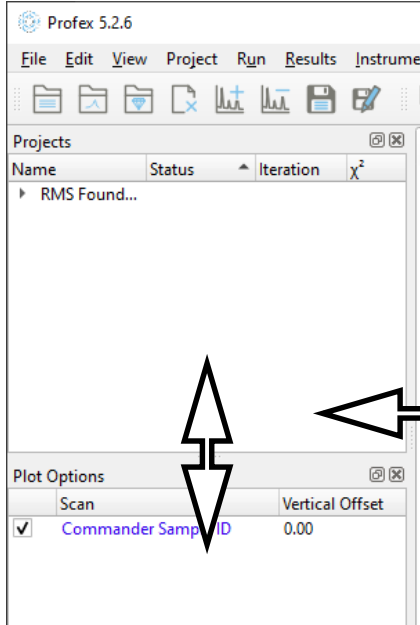


Stacked

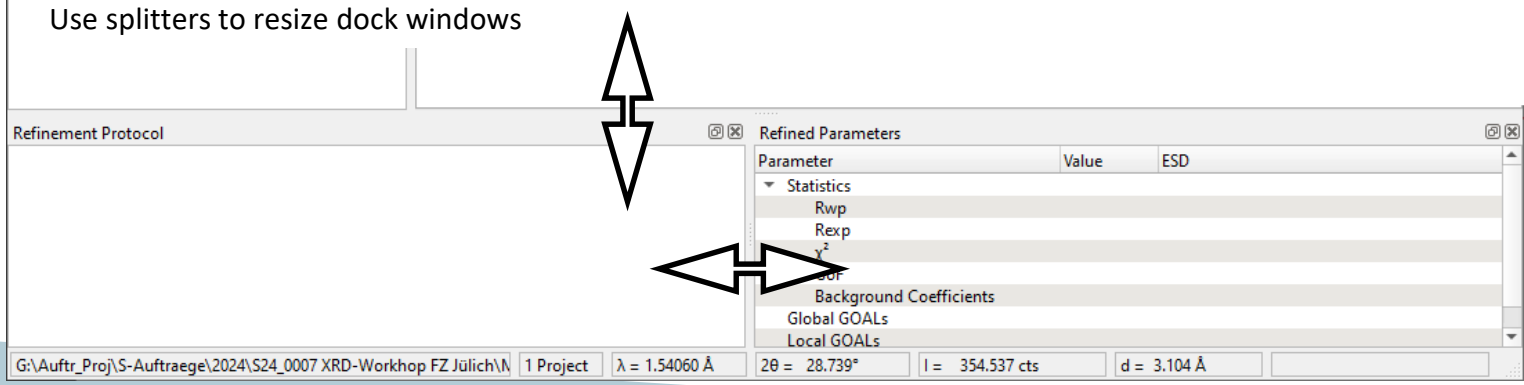


To re-arrange:
Grab title bar of dock windows with the mouse and drag.

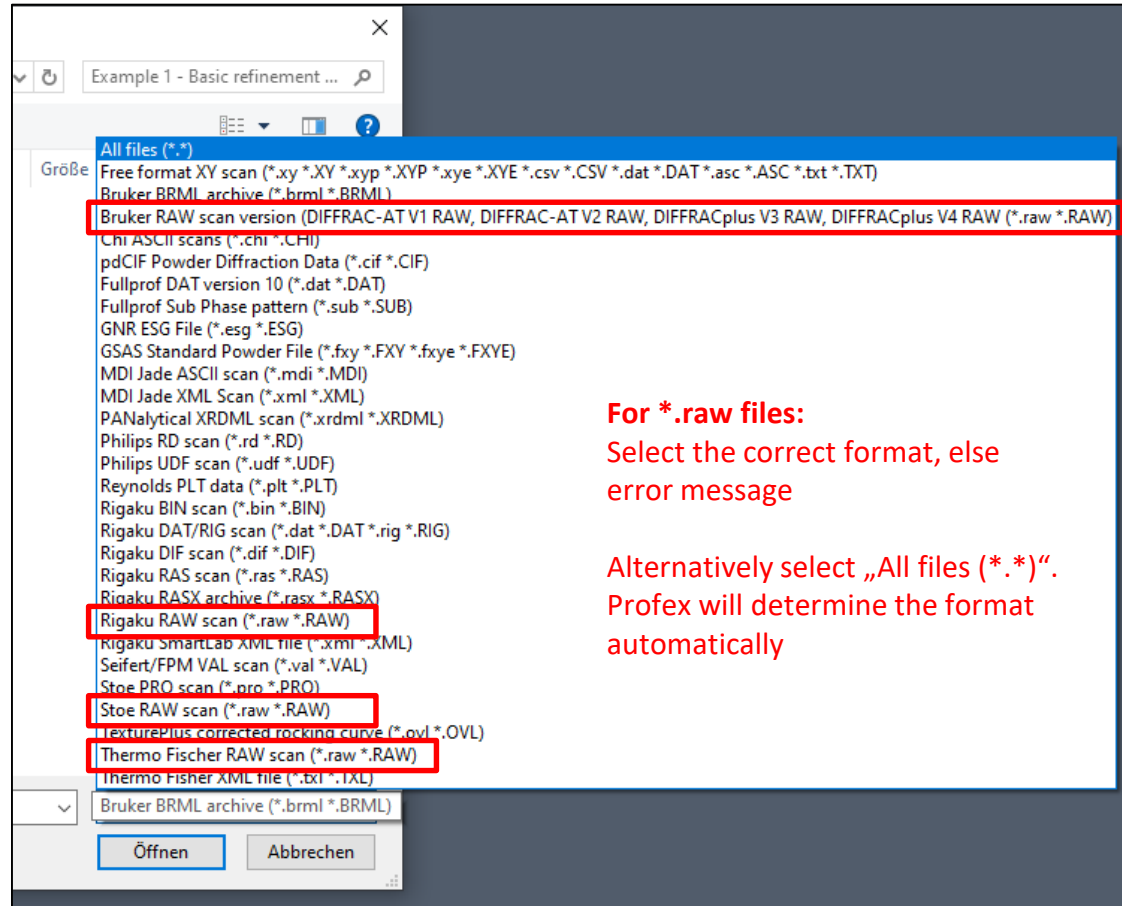
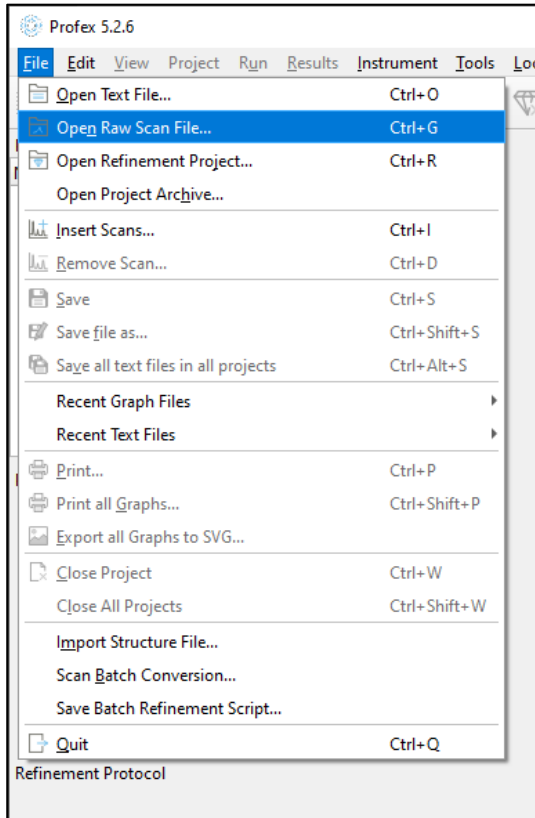
Profex User Interface



Closed dock windows can be opened from the „Window“ menu



Loading Scan Files



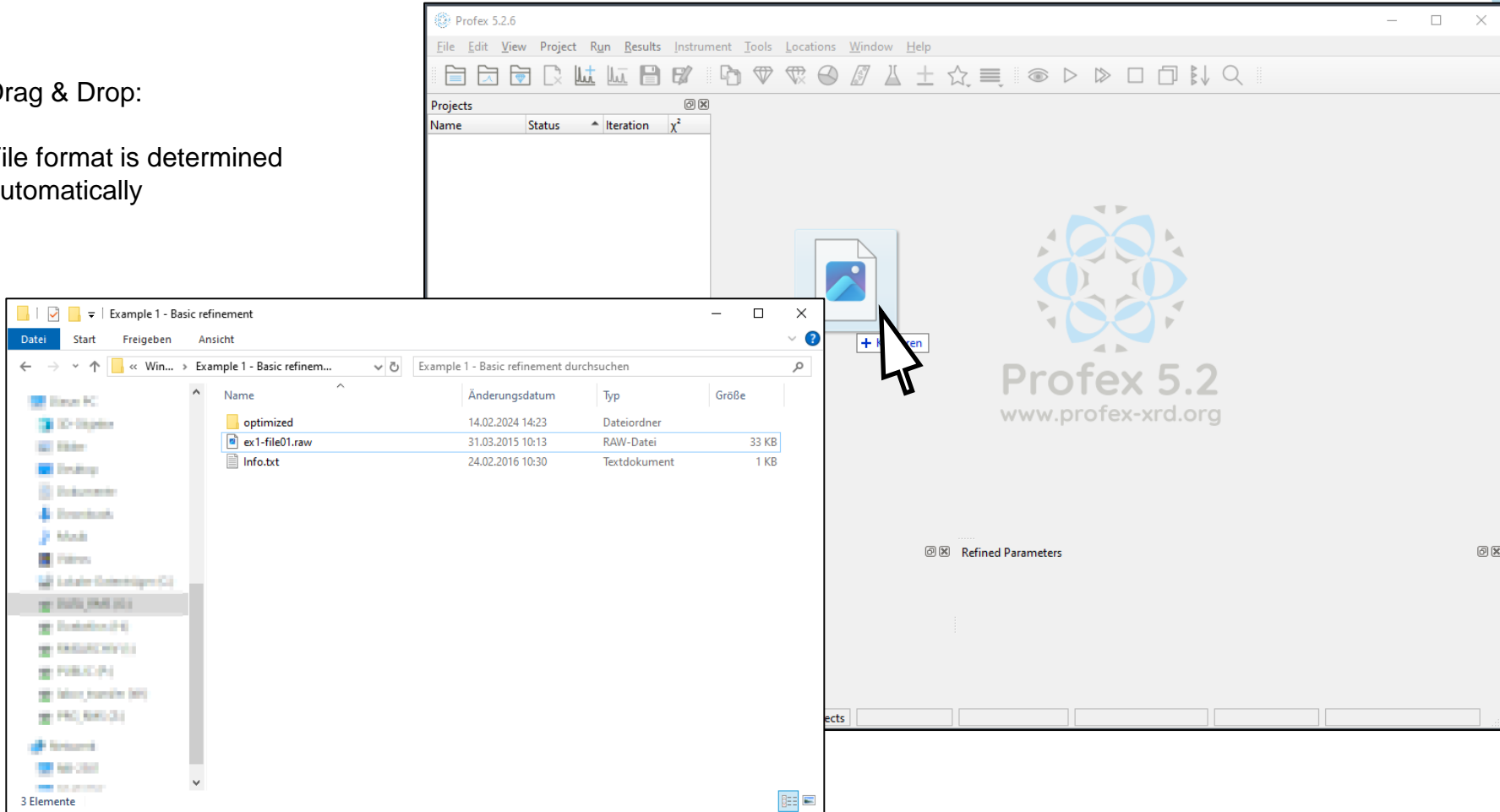
For *.raw files:
Select the correct format, else
error message

Alternatively select „All files (*.*)“.
Profex will determine the format
automatically

Loading Scan Files

Drag & Drop:

File format is determined automatically



Loading Scan Files

The screenshot displays the Profex 5.2.6 software interface. The main window shows a diffraction scan plot titled "RMS Forschung 50% HA ink 1000deg Amy WJ" with the file name "ex1-file01.raw". The plot shows Intensity [counts] on the y-axis (0 to 30000) and Diffraction Angle [2θ] on the x-axis (10.00 to 80.00). The plot features a blue line representing the scan data, with a prominent peak at approximately 32 degrees 2θ .

On the left side, there are two panels highlighted with a red border:

- Projects:** A table with columns "Name", "Status", and "Iteration". The entry "ex1-file01" is selected and highlighted in blue. The text "Refinement projects" is overlaid in red.
- Plot Options:** A table with columns "Scan" and "Vertical Offset". The entry "RMS Forschung 50% HA in..." is checked, and the vertical offset is 0.00. The text "Scans in the current project" is overlaid in red.

At the bottom, the "Refined Parameters" panel is visible, showing a table with columns "Parameter", "Value", and "ESD". The parameters listed are:

Parameter	Value	ESD
Statistics		
Rwp		
Rexp		
χ^2		
GoF		
Background Coefficients		
Global GOALS		
Local GOALS		

The status bar at the bottom shows: "G:\Auftr_Proj\S-Auftraege\2024\S24_0007 XRD-Workshop FZ Jülich\E 1 Project $\lambda = 1.54060 \text{ \AA}$ $2\theta = 0.000^\circ$ $I = 0.000 \text{ cts}$ $d = 0.000 \text{ \AA}$ ".

Loading Scan Files

The screenshot displays the Profex 5.2.6 software interface. The main window shows a diffraction scan plot titled "RMS Forschung 50% HA ink 1000deg Amy WJ". The y-axis is labeled "Intensity [counts]" and ranges from 0 to 30000. The x-axis is labeled "Diffraction Angle [°2θ]" and ranges from 10.00 to 80.00. A prominent peak is visible at approximately 30.00°. The plot is titled "ex1-file01.raw".

On the left side, there is a "Projects" panel with a table:

Name	Status	Iteration	χ^2
ex1-file01			

Below the projects panel is the "Plot Options" section with a table:

Scan	Vertical Offset
<input checked="" type="checkbox"/> RMS Forschung 50% HA in...	0.00

On the right side, a "Context Help" window is open, titled "Interacting with graphs". It lists mouse and keyboard actions:

Interacting with graphs

Mouse actions

Left Mouse Button	Zoom
Ctrl + Left mouse button	Drag view
Shift + Left mouse button	Select scan
Left double click	Load reference structure with strongest peak at click position
Ctrl + Left double click	Print current coordinates to refinement protocol console
Right mouse button	Reset zoom
Middle mouse button	Scale intensity of reference lines
Scroll wheel	Zoom horizontally
Ctrl + Scroll wheel	Zoom vertically

Keyboard actions

The graph must have keyboard focus.

Special cursors

Two red arrows point to the "Help" menu in the top toolbar and the "Context Help" window. A blue arrow points from the text below to the "Special cursors" section of the help window.

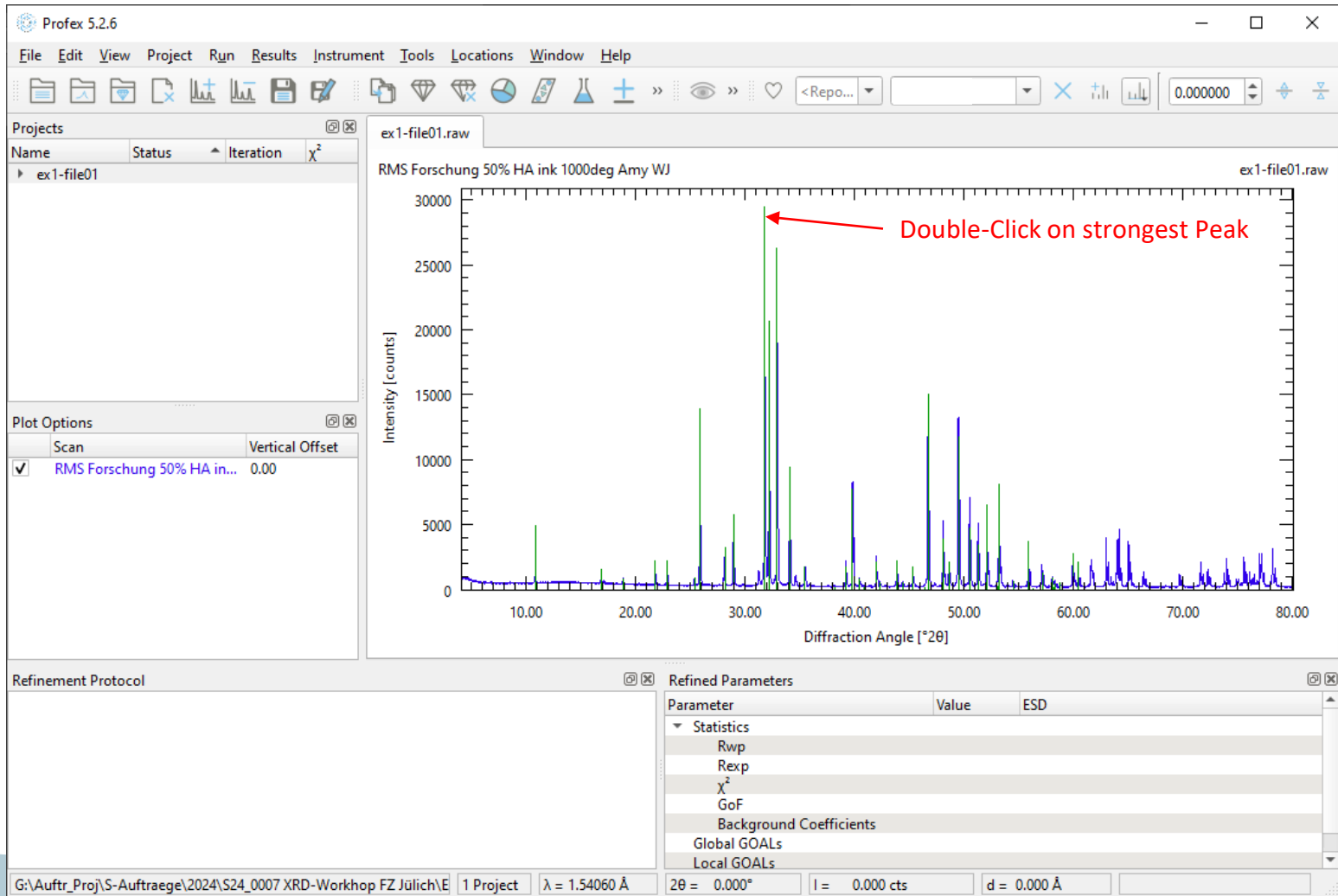
„Help → Mouse and Keyboard commands“

Phase Identification

3 Different approaches to phase identification in Profex

- 1: Double-click strongest peak
 - + Fast
 - - Can be unreliable
 - - Only searches internal database (~1000 phases)
- 2: Run full-pattern search-match in Profex
 - - Slow
 - + Reliable
 - - Only searches internal database (~1000 phases)
- 3: Run peak detection in Profex, import peak list to other search-match software
 - Can be slow
 - Requires other software
 - Searches COD or ICDD database (> 400'000 phases)

Phase Identification: Double Click on Strongest Peak

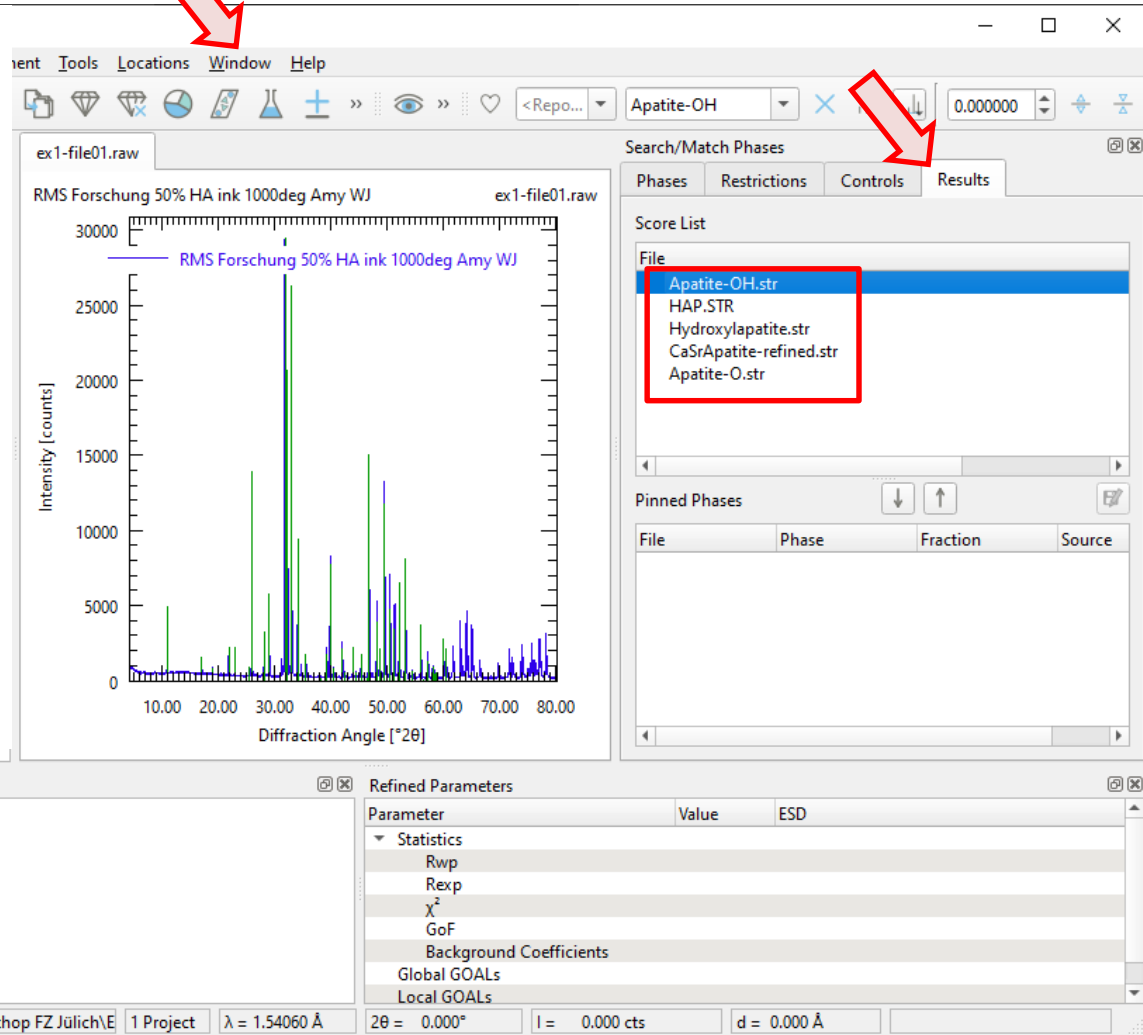


Phase Identification: Double Click on Strongest Peak

Best matching phases are also shown in Search-Match module:

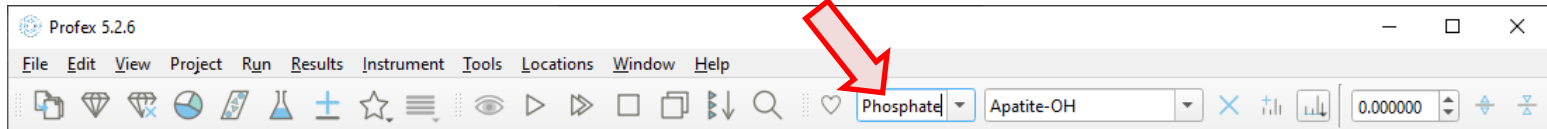
Window

- Search/Match Phases
- Results



Phase Identification: Double Click on Strongest Peak

Limit the search to a sub-directory to improve the hit rate



Double-click phase identification is often good enough to identify the strongest phase.

If not: Use Search/Match Module (Window → Search/Match Phases)

Full-Pattern Search/Match Module

The screenshot displays the Profex 5.2.6 interface. The main window shows an XRD pattern for 'ex1-file01.raw' with the title 'RMS Forschung 50% HA ink 1000deg Amy WJ'. The y-axis is 'Intensity [counts]' ranging from 0 to 30000, and the x-axis is 'Diffraction Angle [°2θ]' ranging from 10.00 to 80.00. A red arrow points to the 'Search/Match Phases' panel on the right, specifically to the 'Repositories' list. The 'Phases' tab is active, showing 0 phases under 'Favorites' and 1578 phases under 'Directories'. The 'Repositories' list is expanded to show the following table:

Repository	Number
<input checked="" type="checkbox"/> C:\RMSPRG\Profex-5.2.6-64bit\Structures	1
<input type="checkbox"/> SiC	26
<input checked="" type="checkbox"/> Phosphate	73
<input type="checkbox"/> Organic	12
<input type="checkbox"/> Minerals	100
<input type="checkbox"/> MetalsAlloysOxides	46
<input type="checkbox"/> MarsMineralCompendium	241
<input type="checkbox"/> Ceramics	101
<input type="checkbox"/> Cement	12
<input type="checkbox"/> Cement-PST	53
<input type="checkbox"/> BGMN	415
<input type="checkbox"/> Alumina-Titania-Zirconia-Yttria	17
<input type="checkbox"/> G:\Ressourcen\G036 XRD_Bruker_D8_Advance...	209
<input type="checkbox"/> C:\Users\doebelinn\AppData\Roaming\doebe...	69

The status bar at the bottom shows: G:\Auftr_Proj\S-Auftraege\2024\S24_0007 XRD-Workhop FZ Jülich\E 1 Project $\lambda = 1.54060 \text{ \AA}$ $2\theta = 0.000^\circ$ I = 0.000 cts d = 0.000 \AA

Full-Pattern Search/Match Module

Profex 5.2.6

File Edit View Project Run Results Instrument Tools Locations Window Help

ex1-file01.raw

RMS Forschung 50% HA ink 1000deg Amy WJ

Intensity [counts]

Diffraction Angle [2θ]

ex1-file01.raw

No Restrictions

Search/Match Phases

Phases Restrictions Controls Results

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18		
1	H																He		
2	Li	Be										B	C	N	O	F	Ne		
3	Na	...										Al	Si	P	S	Cl	Ar		
4	K	Ca	Sc	Ti	V	Cr	...	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr	
5	Rb	Sr	Y	Zr	Nb	...	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe	
6	Cs	Ba	*	Lu	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
7	Fr	Ra	**																
L			*	La	Ce	Pr	Nd	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb		
A			**	Ac	Th	Pa	U	Np	Pu	Bk	Cf						

Change all

Optional At least one Discard optional

Mandatory Discarded

G:\Auftr_Proj\S-Auftraege\2024\S24_0007 XRD-Workhop FZ Jülich\E 1 Project $\lambda = 1.54060 \text{ \AA}$ $2\theta = 69.241^\circ$ $I = 11390.325 \text{ cts}$ $d = 1.356 \text{ \AA}$

Full-Pattern Search/Match Module

The screenshot displays the Profex 5.2.6 software interface. The main window shows an XRD pattern for 'ex1-file01.raw' with the title 'RMS Forschung 50% HA ink 1000deg Amy WJ'. The plot shows Intensity (counts) on the y-axis (0 to 30000) and Diffraction Angle [2θ] on the x-axis (10.00 to 80.00). A prominent peak is visible at approximately 32 degrees 2θ . The 'Search/Match Phases' panel on the right is active, with the 'Controls' tab selected and highlighted by a red box. The controls include:

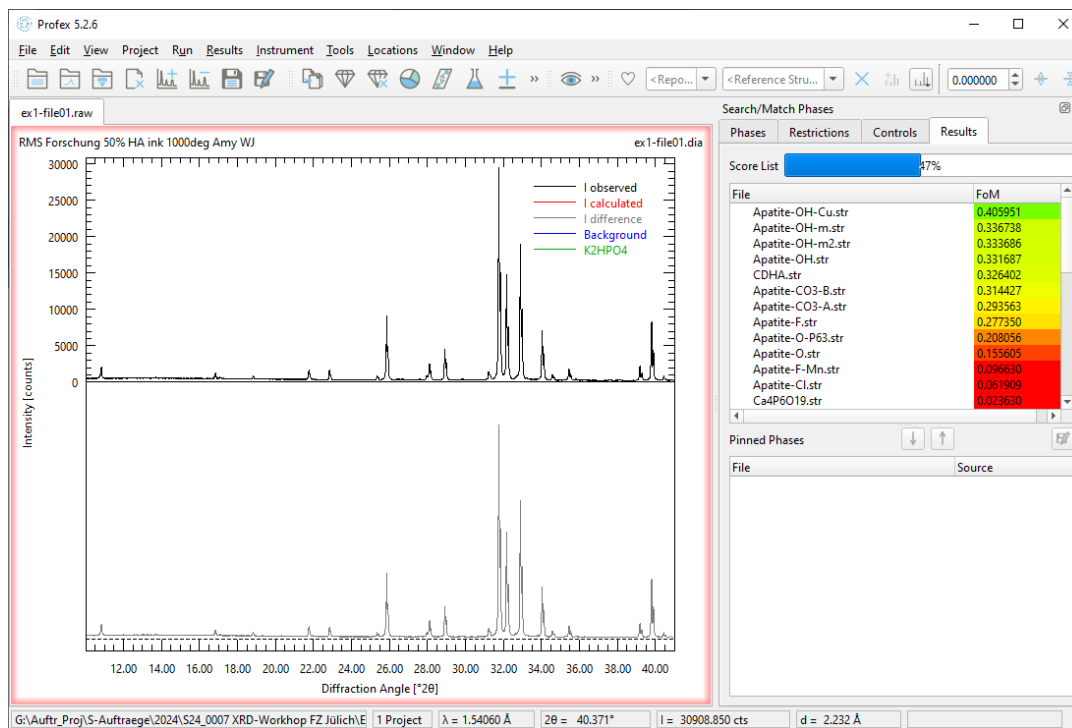
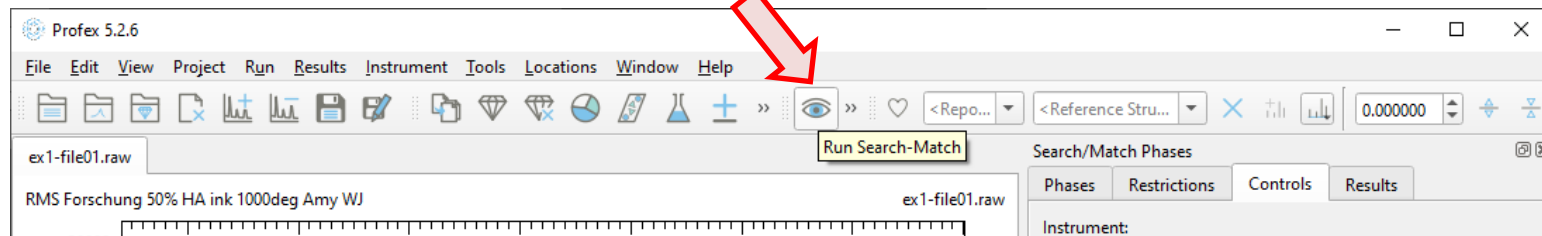
- Instrument: RMS-D8-ADS-15
- Instrument configuration: RMS-D8-ADS-15
- Characteristic radiation: CU
- Synchrotron radiation: 0.0182100 nm
- Refinement: Number of iterations: 10; Minimum angle: 10.00; Maximum angle: 41.00; Set number of background coefficients: 10
- Unit cell variability: Strict
- Allow anisotropic parameters:
- Refine sample height displacement:
- Sample properties: Crystallinity: High

Red text annotations are overlaid on the plot area:

- IMPORTANT: Adjust controls:
- Instrument: RMS-D8-ADS-15-LynxEyeXE
- Characteristic Radiation: CU
- Angular range: Include the first peak; Include the strongest peaks

The status bar at the bottom shows: G:\Auftr_Proj\S-Auftraege\2024\S24_0007 XRD-Workhop FZ Jülich\E 1 Project $\lambda = 1.54060 \text{ \AA}$ $2\theta = 0.000^\circ$ $I = 0.000 \text{ cts}$ $d = 0.000 \text{ \AA}$

Full-Pattern Search/Match Module



Full-Pattern Search/Match Module

The screenshot displays the Profex 5.2.7 interface. The main window shows a diffraction pattern plot titled "RMS Forschung 50% HA ink 1000deg Amy WJ" with the x-axis labeled "Diffraction Angle [°2θ]" and the y-axis labeled "Intensity [counts]". The plot shows a blue line representing the experimental data and a green line representing the reference pattern. A red arrow points to the "Help" menu in the top toolbar.

Text overlay on the plot area:

Residual search:
Select a matching phase and pin it.
Then run search-matching again

The right-hand panel, titled "Search/Match Phases", contains a "Score List" table. A red arrow points to the "Pinned Phases" section below the table.

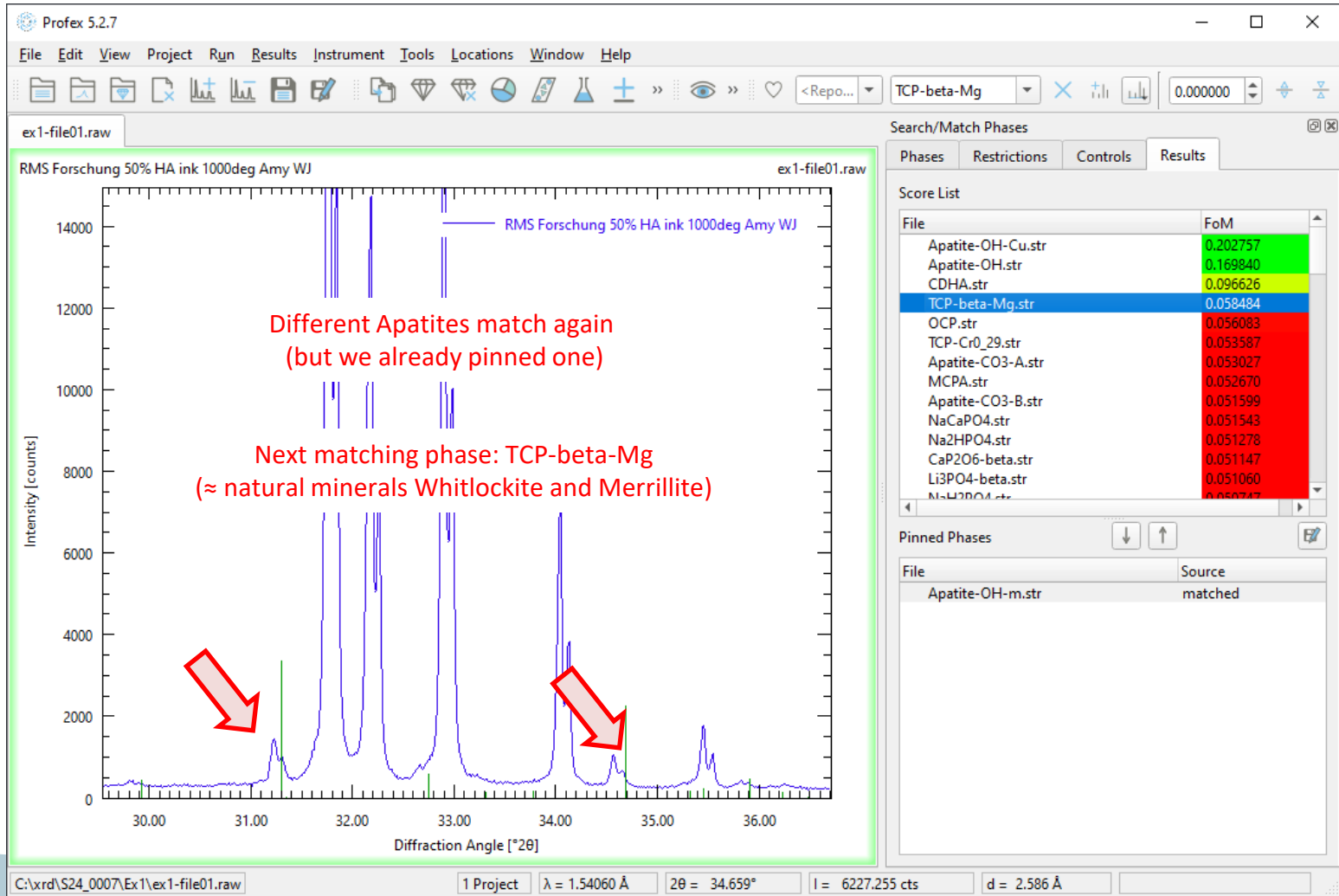
File	FoM
Apatite-OH-Cu.str	0.405951
Apatite-OH-m2.str	0.333686
Apatite-OH.str	0.331687
CDHA.str	0.326402
Apatite-CO3-B.str	0.314427
Apatite-CO3-A.str	0.293563
Apatite-F.str	0.277350
Apatite-O-P63.str	0.208056
Apatite-O.str	0.155605
Apatite-F-Mn.str	0.096630
Apatite-Cl.str	0.061909
OCP.str	0.044558
TCP-beta-Mg.str	0.034596
TCP-Mn-O-20.str	0.030122

The "Pinned Phases" section shows:

File	Source
Apatite-OH-m.str	matched

At the bottom of the window, the status bar displays: C:\xrd\S24_0007\Ex1\ex1-file01.raw | 1 Project | $\lambda = 1.54060 \text{ \AA}$ | $2\theta = 69.621^\circ$ | $I = 13052.696 \text{ cts}$ | $d = 1.349 \text{ \AA}$

Full-Pattern Search/Match Module



Preview: Create Rietveld Refinement Project

Preview

Profex 5.2.7

File Edit View Project Run Results Instrument Tools Locations Window Help

Generate default control file for instrument configuration: RMS-D8-ADS-15-LynxEyeXE

+ Add Phases - Remove Phases

Filter:

File Name	Phase
<input type="checkbox"/> Apatite-CO3-A.str	CO3ApatiteA
<input type="checkbox"/> Apatite-CO3-B.str	CO3ApatiteB
<input type="checkbox"/> Apatite-F.str	Fluorapatite
<input type="checkbox"/> Apatite-F-Mn.str	CaMnApatite
<input type="checkbox"/> Apatite-F-Sr.str	CaSrFApatite
<input type="checkbox"/> Apatite-O.str	Oxyapatite
<input type="checkbox"/> Apatite-OH.str	Hydroxyapatite
<input type="checkbox"/> Apatite-OH-Cu.str	CuHydroxyapatite
<input checked="" type="checkbox"/> Apatite-OH-m.str	HydroxylapatiteM
<input type="checkbox"/> Apatite-OH-m2.str	HydroxylapatiteMonoclinic2
<input type="checkbox"/> Apatite-O-P63.str	CalciumOxidePhosphate
<input type="checkbox"/> Ba3P2O8.str	Ba3P2O8
<input type="checkbox"/> Ca2P6O17.str	Ca2P6O17
<input type="checkbox"/> Ca3Cu3P4O16.str	Ca3Cu3P4O16
<input type="checkbox"/> Ca4P6O19.str	Ca4P6O19
<input type="checkbox"/> CaP2O6-alpha.str	alphaCaP2O6
<input type="checkbox"/> CaP2O6-beta.str	betaCaP2O6
<input type="checkbox"/> CaP4O11.str	CaP4O11
<input type="checkbox"/> CDHA.str	CDHA
<input type="checkbox"/> CPP-alpha.str	alphaCPP

Overwrite existing files

OK Cancel

Intensity [counts]

Diffraction Angle [°2θ]

1 Project λ = 1.54060 Å 2θ = 78.126° I = 30723.092 cts d = 1.222 Å

Results

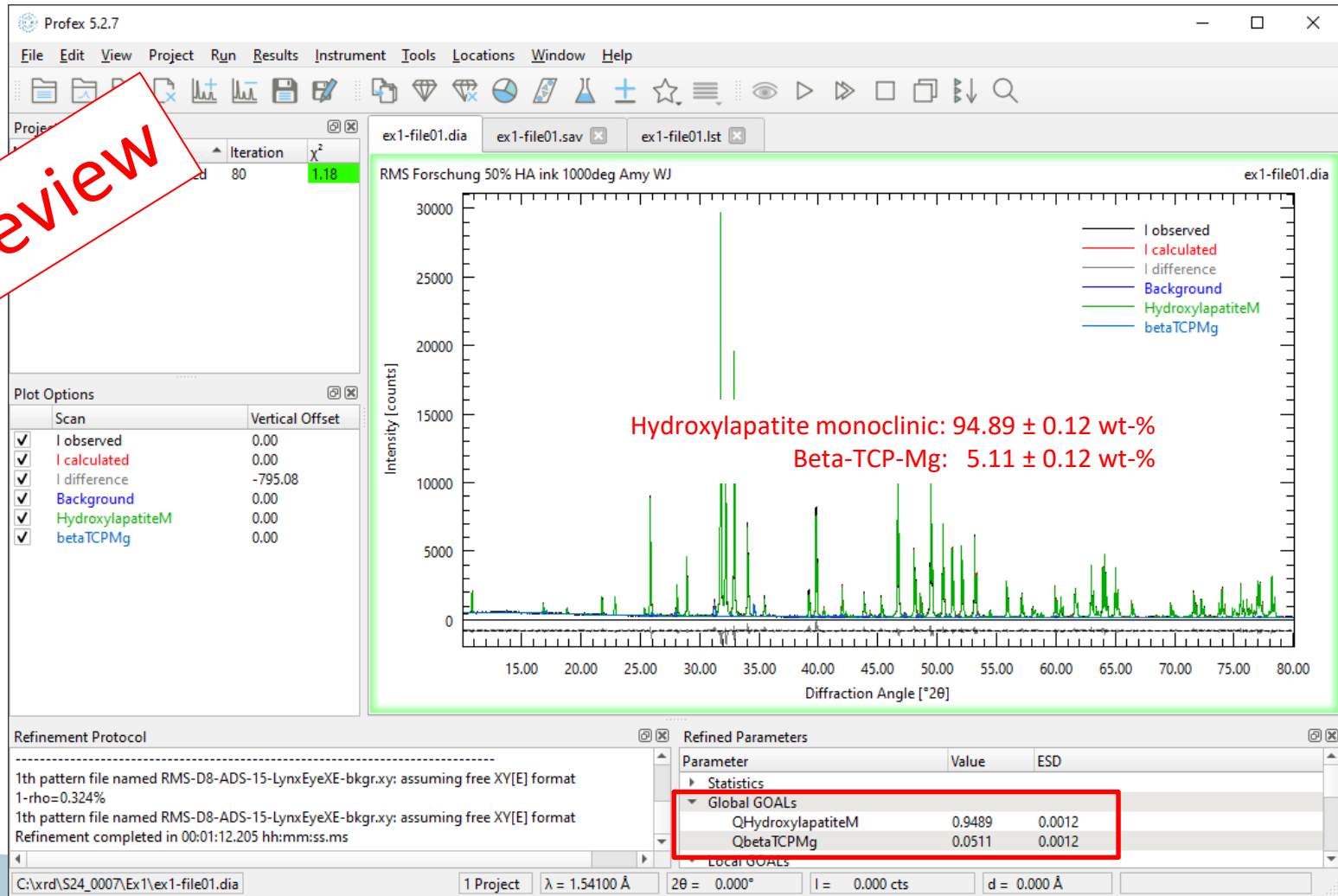
FoM
0.202757
0.169840
0.096626
0.056083
0.053587
0.053027
0.052670
0.051599
0.051543
0.051278
0.051147
0.051060
0.050747
0.050559

Source

project

Preview: Rietveld Refinement

Preview



Search-Match with 3rd Party Software

Use 3rd-party search/match software to search in large databases:

1. Run peak detection in Profex
(extremely reliable, but slow)
2. Export peak list
3. Import peak list in 3rd-party software
4. Run search/match on large database

2 different Structure Databases

Crystallography Open Database (COD)

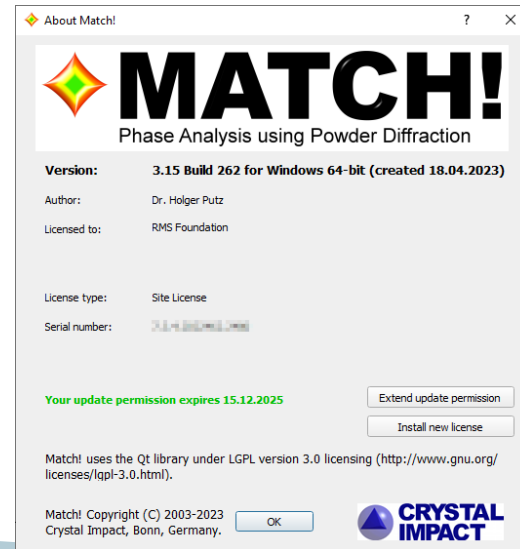
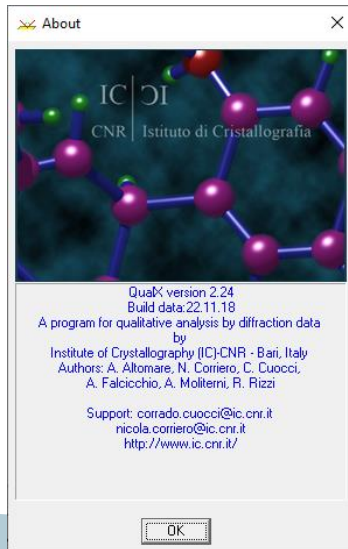
<http://www.crystallography.net/cod>

Open access

ICDD PDF-4+

<https://www.icdd.com/pdf-4-minerals/>

Commercial



Peak Detection

Profex 5.2.7

File Edit View Project Run Results Instrument Tools Locations Window Help

ex1-file01.raw

RMS Forschung 50% HA ink 1000deg Amy WJ

ex1-file01.raw

Phases: All phases

Phase	h	k	l	Angle (°2θ)	d (nm)	Inte
-------	---	---	---	-------------	--------	------

Select Instrument Configuration

Instrument Configuration File RMS-D8-ADS-15-Glass-LynxEyeXE

Wavelength

Characteristic CU

Synchrotron 0.070000 nm

OK Cancel

Intensity [counts]

Diffraction Angle [°2θ]

Refinement Protocol

C:\xrd\S24_0007\Ex1\ex1-file01.raw

1 Project $\lambda = 1.54060 \text{ \AA}$ $2\theta = 0.000^\circ$ $I = 0.000 \text{ cts}$ $d = 0.000 \text{ \AA}$

1. Open peak list (Window → Peak List)
2. Run → Peak detection
3. Select instrument and wavelength

Peak Detection

Profex 5.2.7

File Edit View Project Run Results Instrument Tools Locations Window Help

ex1-file01.raw

RMS Forschung 50% HA ink 1000deg Amy WJ

Intensity [counts]

Diffraction Angle [2θ]

RMS Forschung 50% HA ink 1000deg Amy WJ (ex1-file01.raw)
RMS Forschung 50% HA ink 1000deg Amy WJ peak data ()

Add missed peaks by
Ctrl + Double click on peak

Peak List

Phases: All phases

Phase	h	k	l	Angle (2θ)	d (nm)
11 RMS Forschung...	0	0	0	25.8696	0.344
12 RMS Forschung...	0	0	0	26.6806	0.333
13 RMS Forschung...	0	0	0	27.6079	0.322
14 RMS Forschung...	0	0	0	27.9768	0.318
15 RMS Forschung...	0	0	0	28.1186	0.317
16 RMS Forschung...	0	0	0	28.9337	0.308
17 RMS Forschung...	0	0	0	29.8178	0.299
18 RMS Forschung...	0	0	0	31.2298	0.286
19 RMS Forschung...	0	0	0	31.7733	0.281
20 RMS Forschung...	0	0	0	32.1863	0.277
21 RMS Forschung...	0	0	0	32.9108	0.271
22 RMS Forschung...	0	0	0	34.0566	0.263
23 RMS Forschung...	0	0	0	34.5837	0.259

Refinement Protocol

Q=956.81
terminating fast startup peak scanning
closing iteration..... Q=956.80
Peak detection completed in 00:00:11.907 hh:mm:ss.ms
2theta=31.229806 l=2035.453764 d=2.861753

C:\xrd\S24_0007\Ex1\ex1-file01.raw

1 Project $\lambda = 1.54060 \text{ \AA}$ $2\theta = 0.000^\circ$ $I = 0.000 \text{ cts}$ $d = 0.000 \text{ \AA}$

Export Peak List

Profex 5.2.7

File Edit View Project Run Results Instrument Tools Locations Window Help

ex1-file01.raw Peak List

RMS Forschung 50% HA ink 1000den Amv WI

Save hkl list to CSV

Organisieren Neuer Ordner

Es wurden keine Suchergebnisse gefunden.

Select file format „d values (*.dif *.DIF)

Filename: ex1-file01.dif

Dateityp: d values (*.dif *.DIF)

Speichern Abbrechen

h	k	l	Angle (°2 θ)	
0	0	0	25.861	
0	0	0	26.681	
0	0	0	27.601	
0	0	0	27.9768	0.318
0	0	0	28.1186	0.317
0	0	0	28.9337	0.308
0	0	0	29.8178	0.299
0	0	0	31.2298	0.286
0	0	0	31.7733	0.281
0	0	0	32.1863	0.277
0	0	0	32.9108	0.271
0	0	0	34.0566	0.263
0	0	0	34.5837	0.259

Refinement Protocol

Q=956.81

terminating fast startup peak scanning

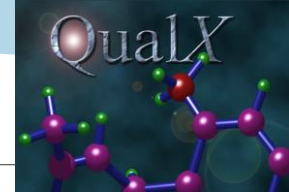
closing iteration..... Q=956.80

Peak detection completed in 00:00:11.907 hh:mm:ss.ms

2theta=31.229806 l=2035.453764 d=2.861753

C:\xrd\S24_0007\Ex1\ex1-file01.raw 1 Project $\lambda = 1.54060 \text{ \AA}$ $2\theta = 34.354^\circ$ $I = 3697.741 \text{ cts}$ $d = 2.608 \text{ \AA}$

Importing Peak List in QualX2



The screenshot illustrates the steps to import a peak list in QualX2:

- 1**: Click on the **File** menu and select **Import Diffraction Data**.
- 2**: In the **Open File** dialog, navigate to the folder containing the peak list file, **ex1-file01.xy**.
- 3**: In the **Set wavelength radiation** dialog, select the radiation type **Cu** from the list. The wavelength is set to **1.540560**.

The **Open File** dialog shows the following file list:

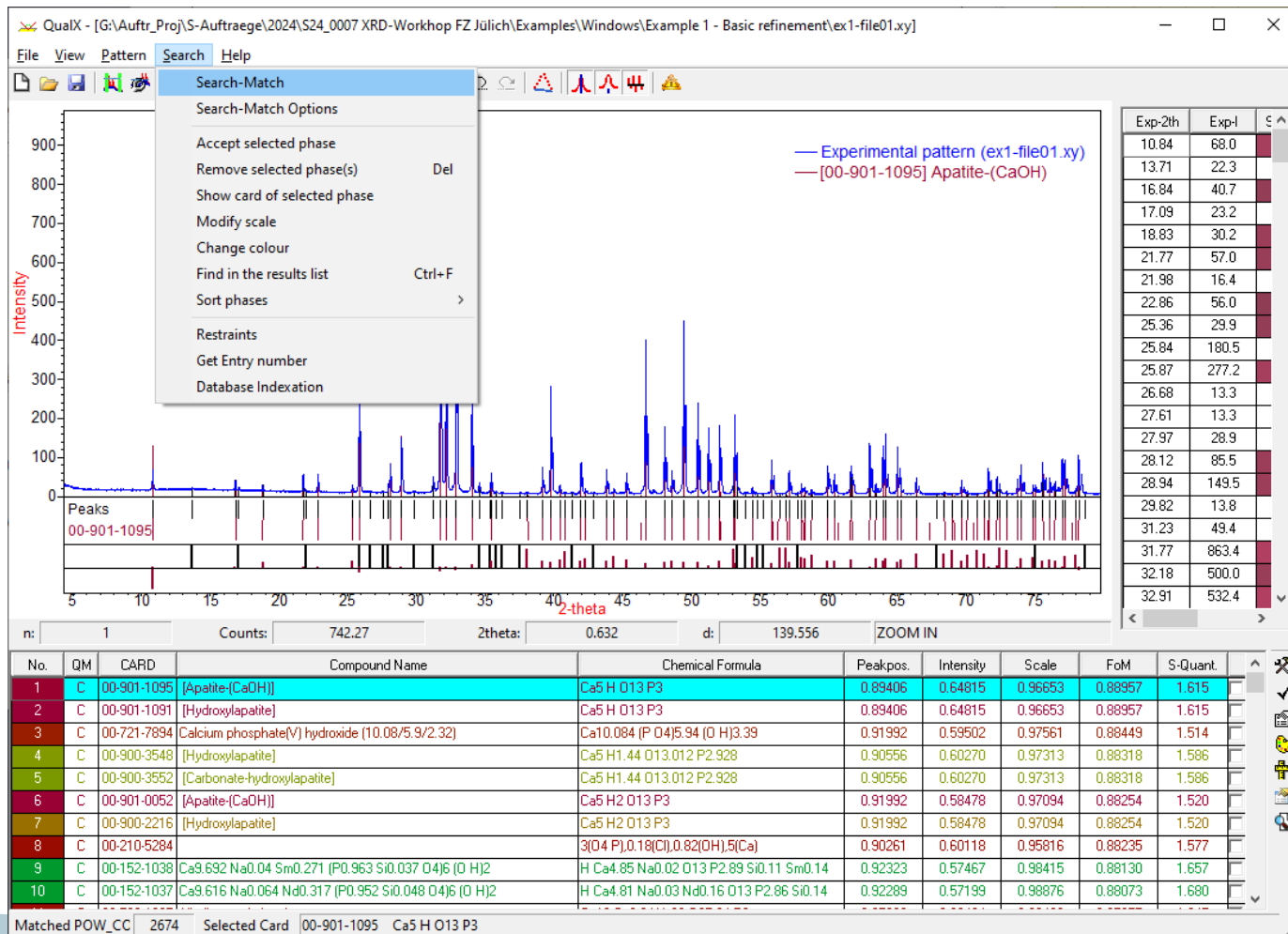
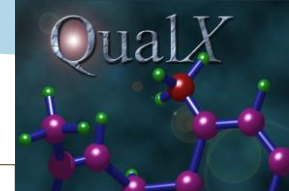
Name	Änderungsdatum	Typ	Größe
optimized	14.02.2024 14:23	Dateiordner	
ex1-file01.xy	15.02.2024 15:55	XY-Datei	134 KB
RMS-D8-ADS-15-LynxEyeXE-bkgr.xy	15.02.2024 15:56	XY-Datei	208 KB

The **Set wavelength radiation** dialog shows the following options:

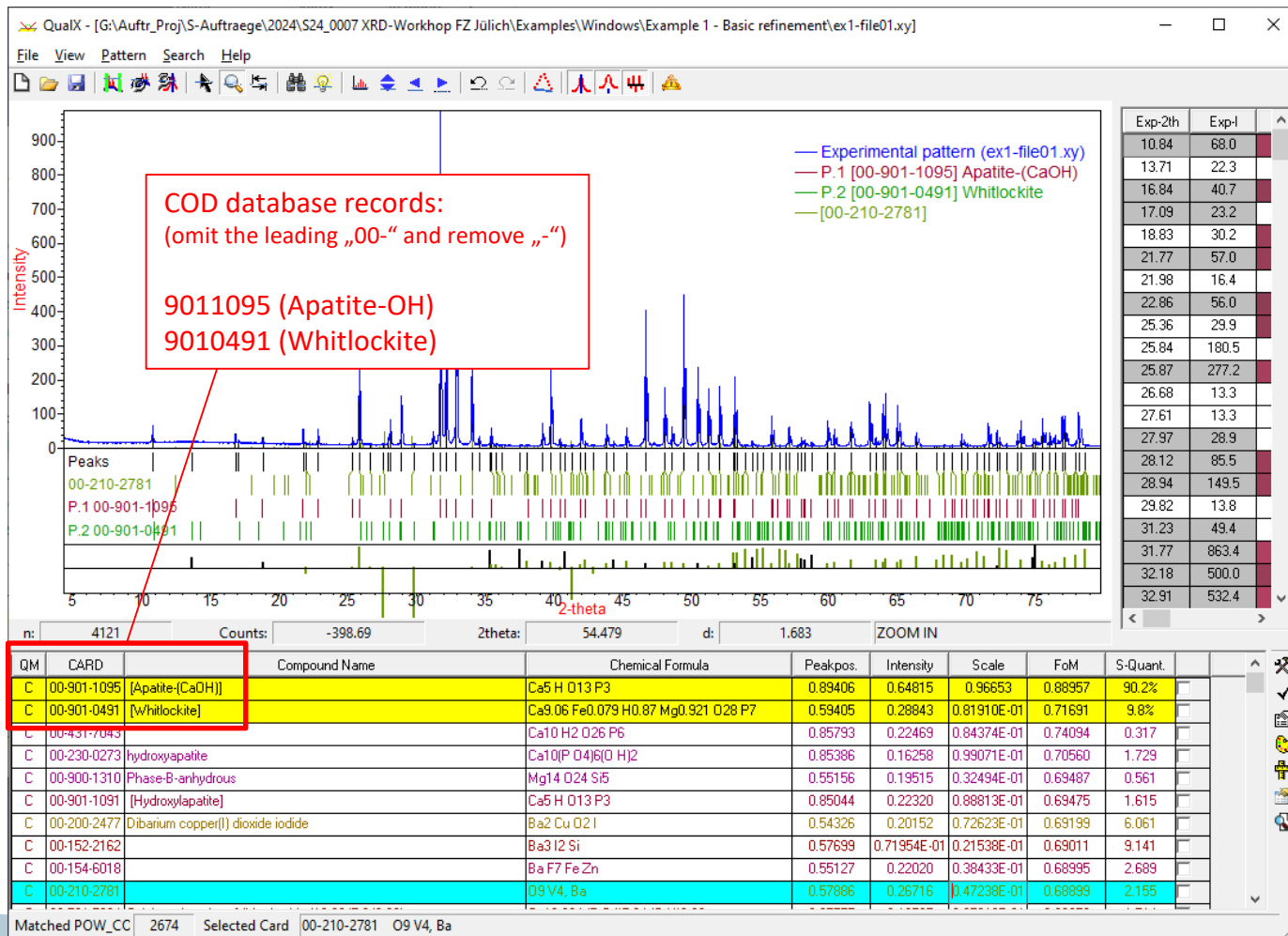
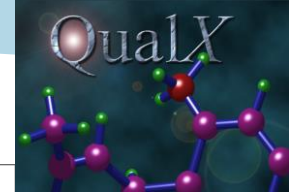
Select wavelength
User Defined
Cr
Fe
Co
Ni
Cu
Mo
Ag

The **lambda:** field is set to **1.540560**.

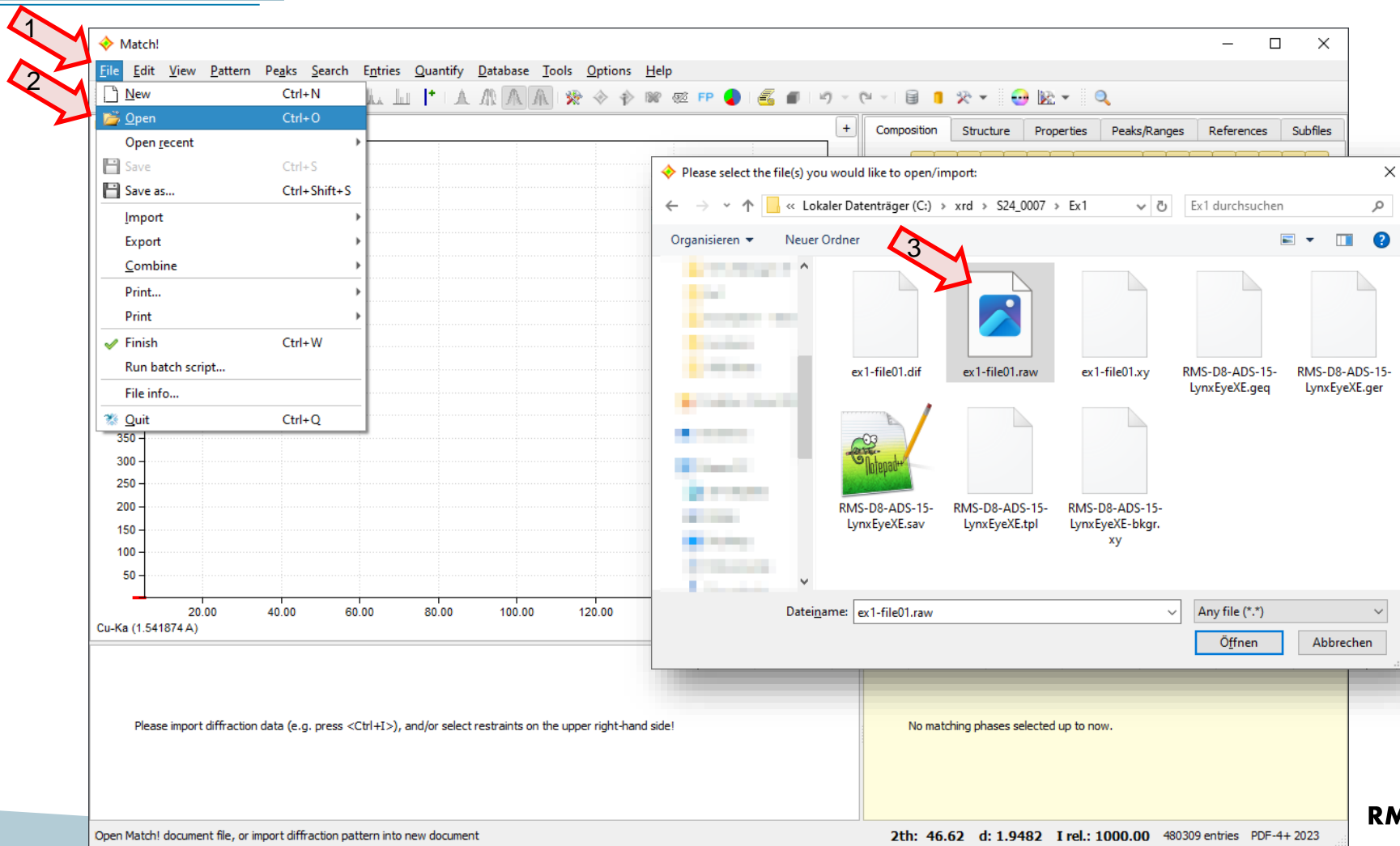
Search/Match in QualX2



Search/Match in QualX2



Importing Peak List in Match!



The screenshot shows the Match! software interface. The 'File' menu is open, with 'Open' selected. A file selection dialog is overlaid on the main window, showing a folder named 'Ex1' containing several files. The file 'ex1-file01.raw' is highlighted. The status bar at the bottom indicates '2th: 46.62 d: 1.9482 I rel.: 1000.00 480309 entries PDF-4+ 2023'.

1. Click on the 'File' menu.

2. Click on 'Open' in the File menu.

3. Select the file 'ex1-file01.raw' in the file selection dialog.

Importing Peak List in Match!

Match!*
File Edit View Pat Peaks Search Entries Quantify Database Tools Options Help

Peak searching
+ Add peak(s) Alt+K
Import from file
Find missing peaks
Delete
Delete all peaks Shift+Del
Activate
Deactivate
Mark uncorrelated peaks
Add as restraint(s)
Reset FWHM to default value
Scale relative intensities
Refill residuals
Add peaks to user database

Select the peak data file to import:
Lokaler Datenträger (C:) > xrd > S24_0007
Organisieren Neuer Ordner
ex1-file01.dif

Set Experimental Details
Please select the radiation type and wavelength applied in the diffraction experiment 'ex1-file01.dif'
Type of radiation
 X-rays Neutron
Wavelength
1.5418740 A (Cu-Ka)
Abscissa (value range: 1.22-8.15)
 theta [°] 2theta [°] d [Angstrom]
Help OK

Dateiname:
Peak list (2 columns: 2theta/d I)
Öffnen Abbrechen

Please run raw data processing (e.g. press <Ctrl+A>)!
No matching phases selected up to now.

Import peak data from a 2-column or Stoe PKS file, and add them to the current experimental pattern
2th: 67.46 d: 1.3884 I rel.: 510.39 480309 entries PDF-4+ 2023

Search/Match in Match!

Match!* _ □ ×

File Edit View Pattern Peaks Search Entries Quantify Database Tools Options Help

RMS Forschung 50% HA ink 1000deg Amy WJ
 Calc. (exp. peaks) (Rp=68.8%)
 Background
 [01-089-6437] Ca10.042 (P O4)5.952 (O H)2.292 Calcium Phosphate Hydroxide Hydroxylapatite, syn (93.0%)
 [04-009-2106] Ca2.59 Mg0.41 (P O4)2 Calcium Magnesium Phosphate Whitlockite, syn (7.0%)

ICDD PDF-4+ database records:
 01-089-6437 (Hydroxyapatite)
 04-009-2106 (Whitlockite)

Cu-Ka (1.541874 Å) 2theta

Color	Qual.	Entry	Formula	Cryst.	Candidate phase	P(2theta)	P(I/I0)	I scale fct.	I/Ic	FoM
D		00-002-0786	Ca3 (P O4)2	X	Calcium Phosphate (Whi...	0.0000	0.0000	1.0000	0.0000	0.0000
D		00-003-0713	Ca3 (P O4)2	X	Calcium Phosphate (Whi...	0.0000	0.0000	1.0000	0.0000	0.0000
D		00-006-0426	Ca3 (P O4)2	X	Calcium Phosphate (Whi...	0.0000	0.0000	1.0000	0.0000	0.0000
I		00-009-0169	Ca3 (P O4)2	H	Calcium Phosphate (Whi...	0.0000	0.0000	1.0000	0.0000	0.0000
B		00-013-0404	(Ca, Mg)...	H	Calcium Magnesium Phos...	0.0000	0.0000	1.0000	0.0000	0.0000
I		00-015-0389	H Ca8 Fe P...	M	Hydrogen Calcium Iron ...	0.0000	0.0000	1.0000	0.0000	0.0000
D		00-042-0577	Ca18 Mn2 ...	H	Calcium Manganese Hyd...	0.0000	0.0000	1.0000	0.0000	0.0000
D		00-042-0578	Ca18 Mg2 ...	H	Calcium Magnesium Hydr...	0.0000	0.0000	1.0000	0.0000	0.0000

Subfiles or compound classes:

<input type="checkbox"/> Battery materials	<input type="checkbox"/> ICSD patterns	<input type="checkbox"/> Organic
<input type="checkbox"/> Cement materials	<input checked="" type="checkbox"/> Inorganic	<input type="checkbox"/> Pearson's Crystal Data
<input type="checkbox"/> Ceramic	<input type="checkbox"/> Intercalate	<input type="checkbox"/> Pharmaceuticals
<input type="checkbox"/> Common phases	<input type="checkbox"/> Ionic conductors	<input type="checkbox"/> Pigments
<input type="checkbox"/> Corrosion products	<input type="checkbox"/> Merck	<input type="checkbox"/> Polymers
<input type="checkbox"/> CSD patterns	<input type="checkbox"/> Metals and alloys	<input type="checkbox"/> Superconducting mat.
<input type="checkbox"/> Education	<input checked="" type="checkbox"/> Minerals	<input type="checkbox"/> Zeolites
<input type="checkbox"/> Explosive	<input type="checkbox"/> NBS	
<input type="checkbox"/> Forensic	<input type="checkbox"/> NIST patterns	<input type="button" value="Clear all"/> <input type="button" value="Select all"/>

Database

PDF COD

User database

<input type="checkbox"/> IUCr journals	<input type="checkbox"/> PCOD	<input type="checkbox"/> CRYSTMET	<input type="checkbox"/> Own data
<input type="checkbox"/> AMCSO	<input type="checkbox"/> PCO	<input type="checkbox"/> CSD	
<input type="checkbox"/> COD	<input type="checkbox"/> ICSD	<input type="checkbox"/> PDF	<input type="button" value="Clear"/> <input type="button" value="All"/>

Preset: None / new set

Color	Entry	Formula	Cryst.	Matched phase	Quant.(%)
D	01-089-6437	Ca10.042 (P...	H	Calcium Phosphate H...	93.0
I	04-009-2106	Ca2.59 Mg0...	H	Calcium Magnesium P...	7.0

2theta: 4.00 d: 22.0902 I rel.: 1000.00 36 entries PDF-4+ 2023

RMS Foundation, Site License RMS

Matches in Match!

The screenshot displays the Match! software interface. The main window shows a powder diffraction pattern with the following data:

- RMS Forschung 50% HA ink 1000deg Amy WJ
- Calc. (exp. peaks) (Rp=68.8 %)
- Background

The x-axis is labeled "2theta" and ranges from 10.00 to 80.00. The y-axis is labeled "I rel." and ranges from 0 to 1000. The Cu-Ka radiation wavelength is 1.541874 Å.

The "Structure*" panel on the right is active, showing the following options:

- Crystal system(s):
 - Cubic
 - Tetragonal
 - Orthorhombic
 - Hexagonal/trigonal
 - Rhombohedral
 - Monoclinic
 - Triclinic/anorthic
 - Buttons: Clear all, Select all
- Space group: [Empty field]
- Unit cell parameters [Å] (min..max):
 - a: [Empty] [Reset]
 - b: [Empty] [Reset]
 - c: [Empty] [Reset]
 - alpha: [Empty] [Reset]
 - beta: [Empty] [Reset]
 - gamma: [Empty] [Reset]
 - Niggli-reduced cell
- Entries must contain full crystal structure (including atomic coordinates)

Below the Structure* panel, there is a message: "Activate to accept only entries that contain a full crystal structure (incl. atomic coordinates)".

At the bottom of the interface, there is a message: "Please run search-match by pressing <Ctrl+M>!".

The bottom status bar shows: 2th: 80.01 d: 1.1992 I rel.: 995.16 305478 entries PDF-4+ 2023

Rietveld Refinement requires full crystal structure information

Activate this option in Match!

Activate to accept only entries that contain a full crystal structure (incl. atomic coordinates)

Entries must contain full crystal structure (including atomic coordinates)

Importing new Structures to Profex

Importing from COD:

- Needs COD installed in Profex
- Needs internet connection

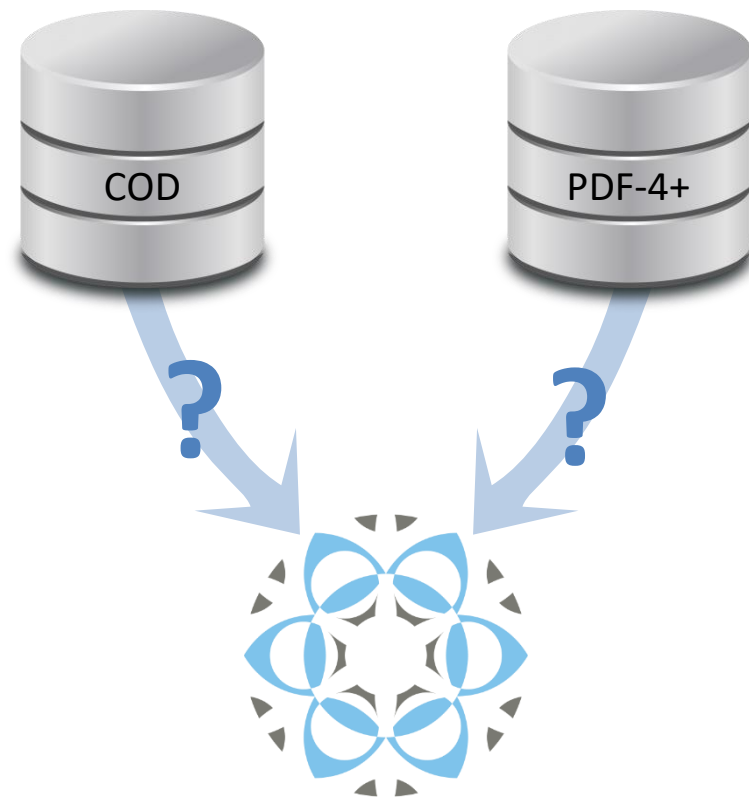
Importing from ICDD PDF-4+

- Needs valid license for PDF-4+
- Export structure from PDF-4+ as XML file

Note:

ICDD **PDF-2** does not contain crystal structure information.

→ Can't be used for Rietveld Refinement



Importing Structure Files from COD

The screenshot shows the Profex 5.2.7 software interface. The 'File' menu is open, and the 'Import Structure File...' option is highlighted. A red arrow labeled '1' points to the 'File' menu, and another red arrow labeled '2' points to the 'Import Structure File...' option. The main window displays the Profex 5.2 logo and the website address www.profex-xrd.org. The bottom of the window shows a 'Refinement Protocol' section with 'Refined Parameters' visible.

1

2

Profex 5.2.7

File Edit View Project Run Results Instrument Tools Locations Window Help

- Open Text File... Ctrl+O
- Open Raw Scan File... Ctrl+G
- Open Refinement Project... Ctrl+R
- Open Project Archive...
- Insert Scans... Ctrl+I
- Remove Scan... Ctrl+D
- Save Ctrl+S
- Save file as... Ctrl+Shift+S
- Save all text files in all projects Ctrl+Alt+S
- Recent Graph Files
- Recent Text Files
- Print... Ctrl+P
- Print all Graphs... Ctrl+Shift+P
- Export all Graphs to SVG...
- Close Project Ctrl+W
- Close All Projects Ctrl+Shift+W
- Import Structure File...**
- Scan Batch Conversion...
- Save Batch Refinement Script...
- Quit Ctrl+Q

Profex 5.2
www.profex-xrd.org

Refinement Protocol

Refined Parameters

Importing Structure Files from COD

The image shows a software interface with two overlapping dialog boxes. The background window is titled 'Import Structure Files' and has a menu open with three options: 'Open local file', 'Retrieve from COD Database', and 'Enter COD IDs'. A red arrow labeled '1' points to the 'Open local file' option, and another red arrow labeled '2' points to the 'Retrieve from COD Database' option. The foreground window is titled 'COD Structure Retrieval' and is divided into two sections: 'Structure' and 'Reference'. The 'Structure' section contains fields for 'COD ID', 'Mineral Name', 'Number of Elements' (with 'min' and 'max' dropdowns), 'Elements' (with '<include>' and '<exclude>' dropdowns), 'Space Group' (with '<Hermann-Mauguin symbol>' dropdown), and 'Temperature' (with a 'Restrict to:' checkbox and a 'Room temperature' dropdown). The 'Reference' section contains fields for 'Title', 'Authors', 'Journal', 'Volume', 'Year' (with 'oldest' and 'most recent' dropdowns), and 'DOI' (with '<10.1107/S2052520616015675>' dropdown). Below these sections is a 'Search' button and a green status indicator 'Database connected'. At the bottom of the dialog is a table with columns: 'COD ID', 'Mineral', 'Formula', 'Space Group', 'Year', and 'Bibliography'. The table is currently empty. At the bottom right of the dialog are 'OK' and 'Cancel' buttons. The background window also has a 'Messages' tab and an 'hkl Plot' tab, and a status bar at the bottom with a 'CuK α_1 ' dropdown and a 'Close' button.

Importing Structure Files from COD

COD Structure Retrieval

Structure

COD ID:

Mineral Name:

Number of Elements:

Elements:

Space Group:

Temperature: Restrict to:

Reference

Title:

Authors:

Journal:

Volume:

Year:

DOI:

Database connected

COD ID	Mineral	Formula	Space Group	Year	Bibliography
<input type="checkbox"/> 1011242	Hydroxylapatite	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1932	Hendricks, S B; ...
<input type="checkbox"/> 9001233	Hydroxylapatite	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1989	Hughes J M; ...
<input type="checkbox"/> 9002213	Hydroxylapatite	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1999	Wilson R M; Eli...
<input type="checkbox"/> 9002214	Hydroxylapatite	Ca ₅ H ₂ O ₁₃ P ₃	P 6 ₃ /m	1999	Wilson, R. M.; ...
<input type="checkbox"/> 9002215	Hydroxylapatite	Ca ₅ H ₂ O ₁₃ P ₃	P 6 ₃ /m	1999	Wilson, R. M.; ...
<input type="checkbox"/> 9002216	Hydroxylapatite	Ca ₅ H ₂ O ₁₃ P ₃	P 6 ₃ /m	1999	Wilson, R. M.; ...

COD Structure Retrieval

Structure

COD ID:

Mineral Name:

Number of Elements:

Elements:

Space Group:

Temperature: Restrict to:

Reference

Title:

Authors:

Journal:

Volume:

Year:

DOI:

Database connected

COD ID	Mineral	Formula	Space Group	Year	Bibliography
<input type="checkbox"/> 9011095	Apatite-(CaOH)	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1969	Sudarsanan, K.; ...

1. Enter mineral properties or COD ID
2. Click «Search»
3. Check the phases you want to download
4. Click «OK» to download

COD ID	Mineral	Formula	Space Group	Year	Bibliography
<input type="checkbox"/> 9011092	Hydroxylapatite	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1969	Sudarsanan, K.; ...
<input type="checkbox"/> 9011093	Hydroxylapatite	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1969	Sudarsanan, K.; ...
<input type="checkbox"/> 9011094	Hydroxylapatite	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1969	Sudarsanan, K.; ...
<input checked="" type="checkbox"/> 9011095	Apatite-(CaOH)	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1969	Sudarsanan, K.; ...
<input type="checkbox"/> 9011096	Apatite-(CaOH)	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1969	Sudarsanan, K.; ...
<input type="checkbox"/> 9011097	Apatite-(CaOH)	Ca ₅ H O ₁₃ P ₃	P 6 ₃ /m	1969	Sudarsanan, K.; ...

Importing Structure Files from COD

Import Structure Files

STR File **Source File**

```
Apatite-(CaOH)_COD_9011095.cif
1 #-----
2 #Date: 2023-11-10 21:21:08 +0200 (Fri, 10 Nov 2023) S
3 #Revision: 287519 S
4 #URL: file:///home/coder/svn-repositories/cod/cif/9/01/10/9011095.cif S
5 #-----
6 #
7 # This file is available in the Crystallography Open Database (COD),
8 # http://www.crystallography.net/. The original data for this entry
9 # were provided the American Mineralogist Crystal Structure Database,
10 # http://ruff.geo.arizona.edu/AMS/amcsd.php
11 #
12 # The file may be used within the scientific community so long as
13 # proper attribution is given to the journal article from which the
14 # data were obtained.
15 #
16 data_9011095
17 loop_
18 _publ_author_name
19 'Sudarsanan, K.'
20 'Young, R. A.'
21 publ_section title
```

Messages hkl Plot

Intensity [%]

Diffraction Angle [° 2θ]

$\rho = 3.1530 \text{ g/cm}^3$

CuK α_1 Close

Downloaded CIF file

Stick pattern and calculated density for verification

If conversion fails due to corrupted CIF file, no stick pattern is shown and errors are reported in «Messages» protocol.

Import Structure Files

STR File **Source File**

```
Apatite-(CaOH)_COD_9011095.cif
1 PHASE=ApatiteCaOH // cod9011095
2 Reference=cod9011095 //
3 Formula=Ca5_H_O13_P3 //
4 SpacegroupNo= 176 HermannMauguin=P6_3/m Setting= 1 UniqueAxis= c Lattice=Hexagonal //
5 PARAM= A= 0.942400_0.932976*0.951824 PARAM= C= 0.687900_0.681021^0.694779 //
6 RP= 4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT= SPHAR4 //
7 GOAL:ApatiteCaOH=GEWICHT*ifhensel(ifdef(d),exp(my*d^3/4),1) //
8 E=CA Wyckoff= f x=0.333333 y=0.666667 z=0.001500 TDS=0.006643
9 E=CA Wyckoff= h x=0.246800 y=0.993400 z=0.250000 TDS=0.005795
10 E=P Wyckoff= h x=0.398700 y=0.368500 z=0.250000 TDS=0.004658
11 E=O Wyckoff= h x=0.328400 y=0.484800 z=0.250000 TDS=0.008381
12 E=O Wyckoff= h x=0.587300 y=0.465100 z=0.250000 TDS=0.010435
13 E=O Wyckoff= i x=0.343700 y=0.257900 z=0.070200 TDS=0.013631
14 E=O(0.5000) Wyckoff= e x=0.000000 y=0.000000 z=0.195000 TDS=0.010878
15 E=H(0.5000) Wyckoff= e x=0.000000 y=0.000000 z=0.060800 TDS=0.029473
16
```

Messages hkl Plot

Intensity [%]

Diffraction Angle [° 2θ]

$\rho = 3.1530 \text{ g/cm}^3$

CuK α_1 Close

Converted BGMN STR file used by Profex

Messages hkl Plot

Filtering by symmetry operators:
--> No matching settings found. Skipping this test.

Running BGMN to verify the structure and calculate hkl line positions:
8 atoms found in spacegroup no -1 setting no -1
Line 9 (CA): No Wyckoff symbol found
Line 10 (CA): No Wyckoff symbol found

CuK α_1 Close

Saving STR Files

The screenshot illustrates the process of saving an STR file in a software application. The main window shows the 'Import STR Files' dialog with a list of files, including 'Apatite-(CaOH)_COD_9011095.cif'. The 'STR File' tab is active, displaying the following content:

```
1 PHASE=ApatiteCaOH // cod9011095
2 Reference=cod9011095 //
3 Formula=Ca5_H_O13_P3 //
4 SpacegroupNo=176 HermannMauguin=P6_3/m Setting=
5 PARAM=A=0.942400_0.932976^0.951824 PARAM=C=0.687
6 RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHAR
7 GOAL:ApatiteCaOH=GEWICHT*ifthenelse(ifdef(d),exp(my
8 E=CA Wyckoff=f x=0.333333 y=0.666667 z=0.001500 TDS=
9 E=CA Wyckoff=h x=0.246800 y=0.993400 z=0.250000 TDS=
10 E=P Wyckoff=h x=0.398700 y=0.368500 z=0.250000 TDS=0
11 E=O Wyckoff=h x=0.328400 y=0.484800 z=0.250000
12 E=O Wyckoff=h x=0.587300 y=0.465100 z=0.250000
13 E=O Wyckoff=i x=0.343700 y=0.257900 z=0.070200 TDS=0
14 E=O(0.5000) Wyckoff=e x=0.000000 y=0.000000 z=0.19500
15 E=H(0.5000) Wyckoff=e x=0.000000 y=0.000000 z=0.06080
16
```

The 'Save STR File' dialog box is overlaid on top, showing the file name 'HAp-Workshop2024.str' and the file type 'BGMN Structure Files (*.str *.STR)'. The 'Speichern' (Save) button is highlighted.

The main window also displays an 'hkl Plot' showing Intensity [%] versus Diffraction Angle [° 2θ]. The plot shows a series of peaks, with a legend indicating $\rho = 3.1530 \text{ g/cm}^3$. The 'Close' button is visible at the bottom right of the main window.

Saving STR Files

The screenshot displays the Profex 5.2.7 software interface. The main window shows an XRD pattern plot titled "RMS Forschung 50% HA ink 1000deg Amy WJ". The y-axis is labeled "Intensity [counts]" and ranges from 0 to 30000. The x-axis is labeled "Diffraction Angle [°2θ]" and ranges from 10.00 to 80.00. The plot shows a blue line representing the experimental data and a green line representing the fit. A red box highlights a dropdown menu in the top toolbar, which is currently set to "HAp-Workshop2024".

The interface includes several panels:

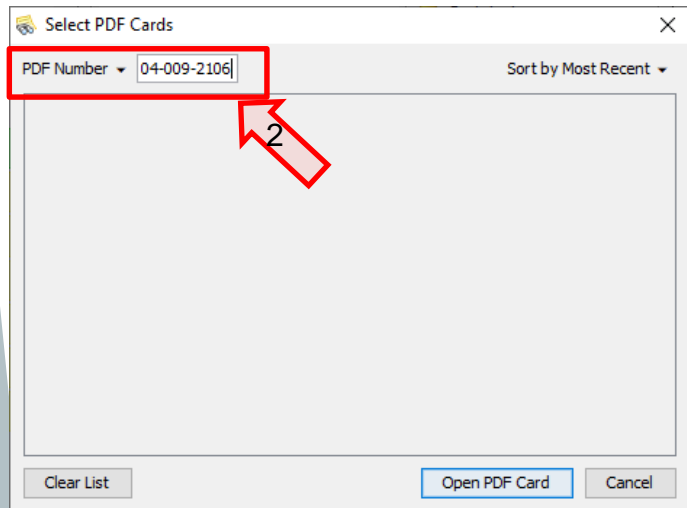
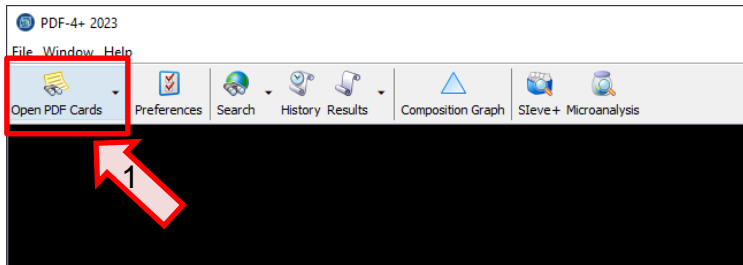
- Projects:** A table with columns for Name, Status, Iteration, and χ^2 . The current project is "ex1-file01".
- Plot Options:** A table with columns for Scan and Vertical Offset. The "Scan" option is checked, and the "Vertical Offset" is 0.00.
- Refinement Protocol:** A section for defining the refinement protocol.
- Refined Parameters:** A table showing the values and estimated standard deviations (ESD) for various parameters.

Parameter	Value	ESD
Statistics		
Rwp		
Rexp		
χ^2		
GoF		

The status bar at the bottom shows the following information:

- File path: C:\xrd\S24_0007\Ex1\ex1-file01.raw
- Project: 1 Project
- Wavelength: $\lambda = 1.54060 \text{ \AA}$
- 2θ: $2\theta = 0.000^\circ$
- Intensity: $I = 0.000 \text{ cts}$
- d-spacing: $d = 0.000 \text{ \AA}$

Exporting XML from PDF-4+



PDF-4+ 2023
File Window Help
Open PDF Cards Preferences Search History Results Composition Graph Sieve+ Microanalysis

Ca_{2.59}Mg_{0.41} (P O₄)₂ - 04-009-2106

File Plots
Export Print Toolbox Property Sheet
Temperature Series 2D Structure SAED Pattern Simulated Profile
3D Structure EBSD Pattern Raw Diffraction Data
Bonds Ring Pattern

X-ray Diffraction
Wavelength: Cu K α 1.54056 Å
 Neutron Diffraction
 Electron Diffraction

Simulated Profile (Calc)
 Raw Diffraction Data
Fixed Slit Intensity

2 θ (°)	d (Å)	I	h	k	l	*
10.965	8.06234	93	0	1	2	
13.720	6.44867	214	1	0	4	
14.266	6.20318	39	0	0	6	
17.155	5.16465	356	1	1	0	
18.594	4.76801	19	1	1	3	
20.404	4.34887	61	2	0	2	
21.511	4.12753	16	0	1	8	
22.032	4.03117	90	0	2	4	
22.381	3.96906	7	1	1	6	
25.907	3.43634	327	1	0	10	

Intensity
2 θ (°)

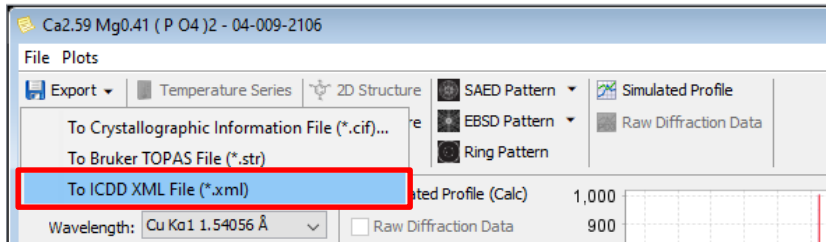
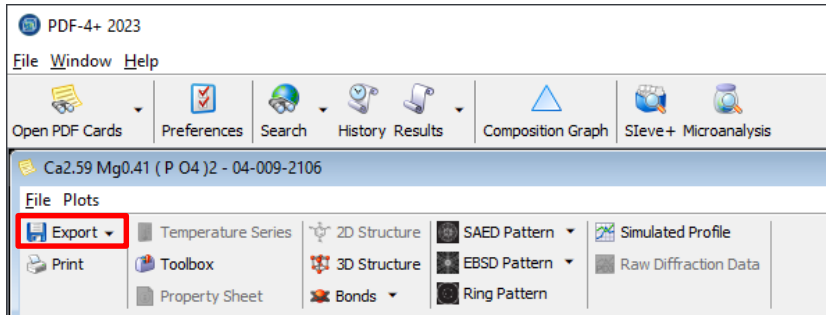
PDF
Status: Primary Quality Mark: Indexed
Experimental Environment: Ambient Temperature: 298.0 K (Assigned by ICDD editor) Pressure: -
Physical Phase: -
Chemical Formula: Ca_{2.59}Mg_{0.41}(P O₄)₂
Crystal Structural Formula: -
Structure Empirical Formula: Ca_{2.59}Mg_{0.41}O₈P₂
Refined Formula: Ca_{2.581}Mg_{0.419}O₈P₂
Classifications Weight %: Ca34.18 Mg3.28 O42.14 P20.40
Atomic %: Ca19.92 Mg3.15 O61.54 P15.38
Cross-references Compound Name: Calcium Magnesium Phosphate
References Mineral Name: Whitlockite, syn IMA No: -
Zeolite Name: -
Alternate Name: -
CAS Number: -
Entry Date: 09/01/2006
Modification Date: 09/01/2020 Modifications: Update

Ca_{2.59}Mg_{0.41} (P O₄)...

Exporting XML from PDF-4+ / Importing in Profex

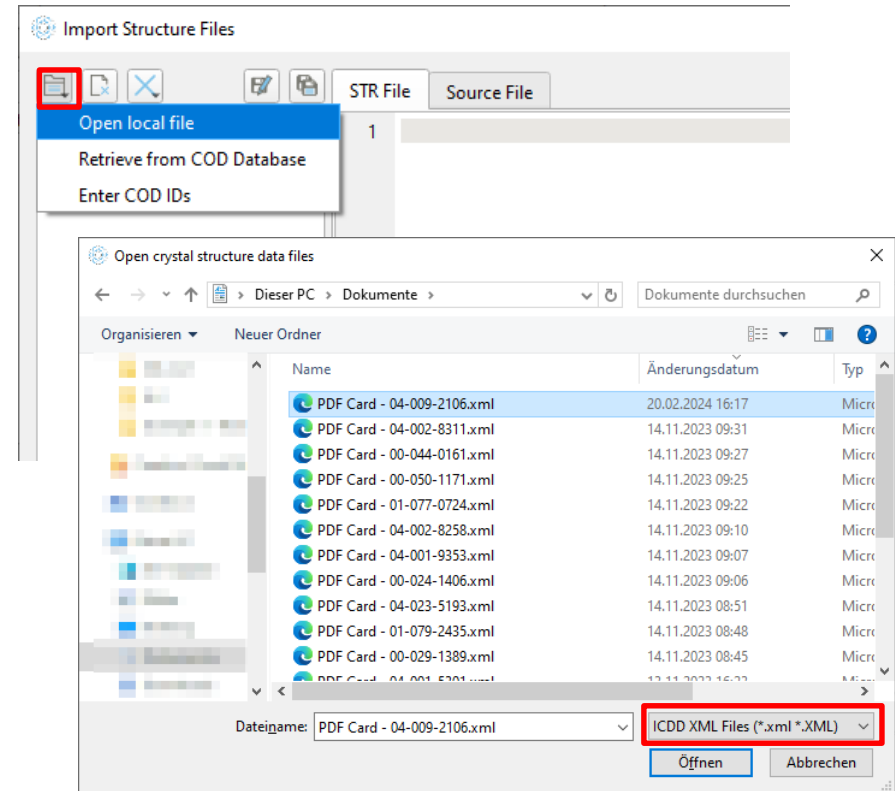
In PDF-4+:

On the PDF card, click Export → To ICDD XML File



In Profex:

File → Import Structure Files → Open local file
Set file format to „ICDD XML Files“



Saving STR Files

1. Check the stick pattern and density for plausibility
2. Save the STR file
3. Close the import dialog

Import Structure Files

STR File Source File

```
1 PHASE= CalciumMagnesiumPhosphate // 04-009-2106
2 Reference=04-009-2106 //
3 Formula=Ca2.59_Mg0.41_(P_O4)_2 //
4 SpacegroupNo=161 HermannMauguin=R3c Setting=1 UniqueAxis=c Lattice=Trigonal //
5 PARAM=A=1.032930_1.022601^1.043259 PARAM=C=3.721910_3.684691^3.759129 //
6 RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHARA //
7 GOAL:CalciumMagnesiumPhosphate=GEWICHT*ifthenelse(fdef(d),exp(my*d^3/4),1) //
8 E=CA Wyckoff=b x=0.724000 y=0.857100 z=0.167500 TDS=0.010000
9 E=CA Wyckoff=b x=0.618000 y=0.821500 z=0.965800 TDS=0.010000
10 E=CA Wyckoff=b x=0.727100 y=0.850600 z=0.060300 TDS=0.010000
11 E=(MG(0.4750),CA(0.0250)) Wyckoff=a x=0.000000 y=0.000000 z=0.916667 TDS=0.010000
12 E=(MG(0.9900),CA(0.0100)) Wyckoff=a x=0.000000 y=0.000000 z=0.737200 TDS=0.010000
13 E=P Wyckoff=a x=0.000000 y=0.000000 z=0.000000 TDS=0.010000
14 E=O Wyckoff=b x=0.007100 y=0.861900 z=0.986300 TDS=0.010000
15 E=O Wyckoff=a x=0.000000 y=0.000000 z=0.040200 TDS=0.010000
16 E=P Wyckoff=b x=0.689100 y=0.863500 z=0.868000 TDS=0.010000
17 E=O Wyckoff=b x=0.744200 y=0.920800 z=0.906300 TDS=0.010000
18 E=O Wyckoff=b x=0.759400 y=0.772700 z=0.854300 TDS=0.010000
19 E=O Wyckoff=b x=0.723800 y=0.023000 z=0.846000 TDS=0.010000
20 E=O Wyckoff=b x=0.518200 y=0.764200 z=0.866300 TDS=0.010000
21 E=P Wyckoff=b x=0.653500 y=0.846300 z=0.766700 TDS=0.010000
```

Messages hkl Plot

Intensity [%]

Diffraction Angle [° 2θ]

$\rho = 3.0780 \text{ g/cm}^3$

CuK α_1

Close

Profex 5.2.7

File Edit View Project Run Results Instrument Tools Locations Window Help

<Repository> Whitlockite-Workshop

Projects

Name Status Iteration χ^2

ex-1-file01

Plot Options

Scan Vertical Offset

RMS Forschung 50% HA ink... 0.00

Refinement Protocol

Refined Parameters

Parameter	Value	ESD
Statistics		
Rwp		
Rexp		
χ^2		
GoF		

C:\xrd\S24_0007\Ex1\ex-1-file01.raw 1 Project $\lambda = 1.54060 \text{ \AA}$ $2\theta = 0.000^\circ$ $l = 0.000 \text{ cts}$ $d = 0.000 \text{ \AA}$

Phase Identification: Summary

- ▶ All phases identified
 - ▶ double-click
 - ▶ search-match module
 - ▶ 3rd party software
- ▶ Missing structure files retrieved from COD or PDF-4+ (or other source)
- ▶ Retrieved structure files converted from CIF/XML format to STR
- ▶ Next: Rietveld refinement