RMS

Testing · Research · Consulting

BGMN/Profex User Meeting 2019

New Features in Profex 4
Part 1

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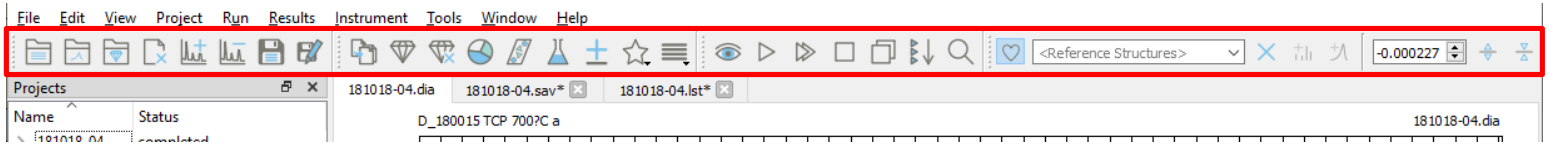
New Features in Profex 4

- GUI changes
- Search / Match
- Peak detection
- Interaction with QualX / Match!

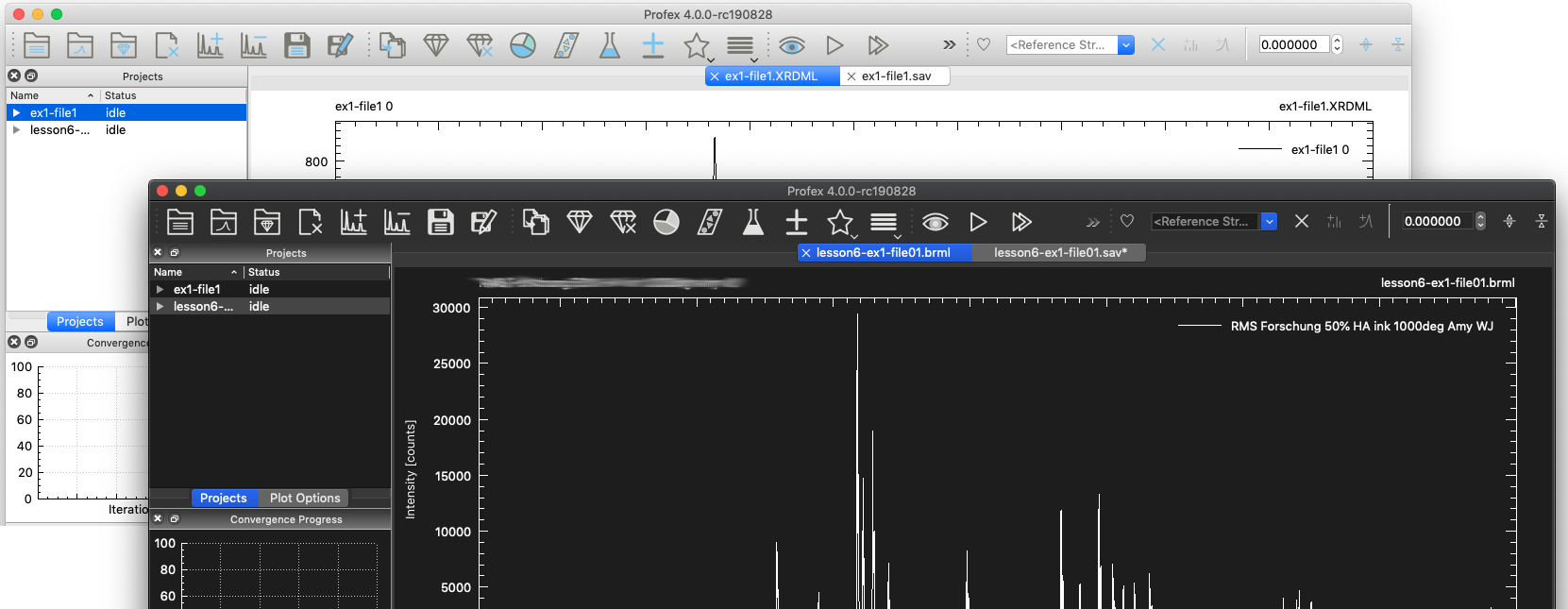
- Electron-density maps
- Drawing crystal structures with Vesta / Mercury
- Customizing report layouts

GUI Changes

New icon set:

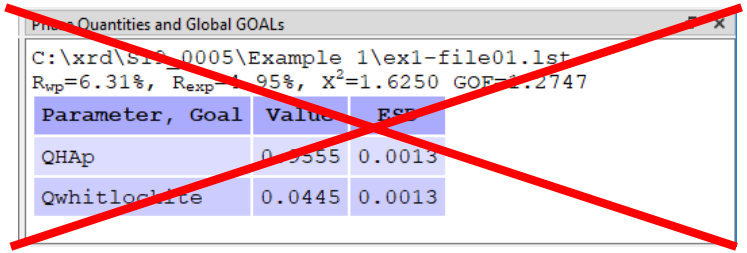


Support for dark mode on OS X:



GUI Changes

Summary tables:



Statistics, Global GOALs, and Local GOALs summarized in a tree structure

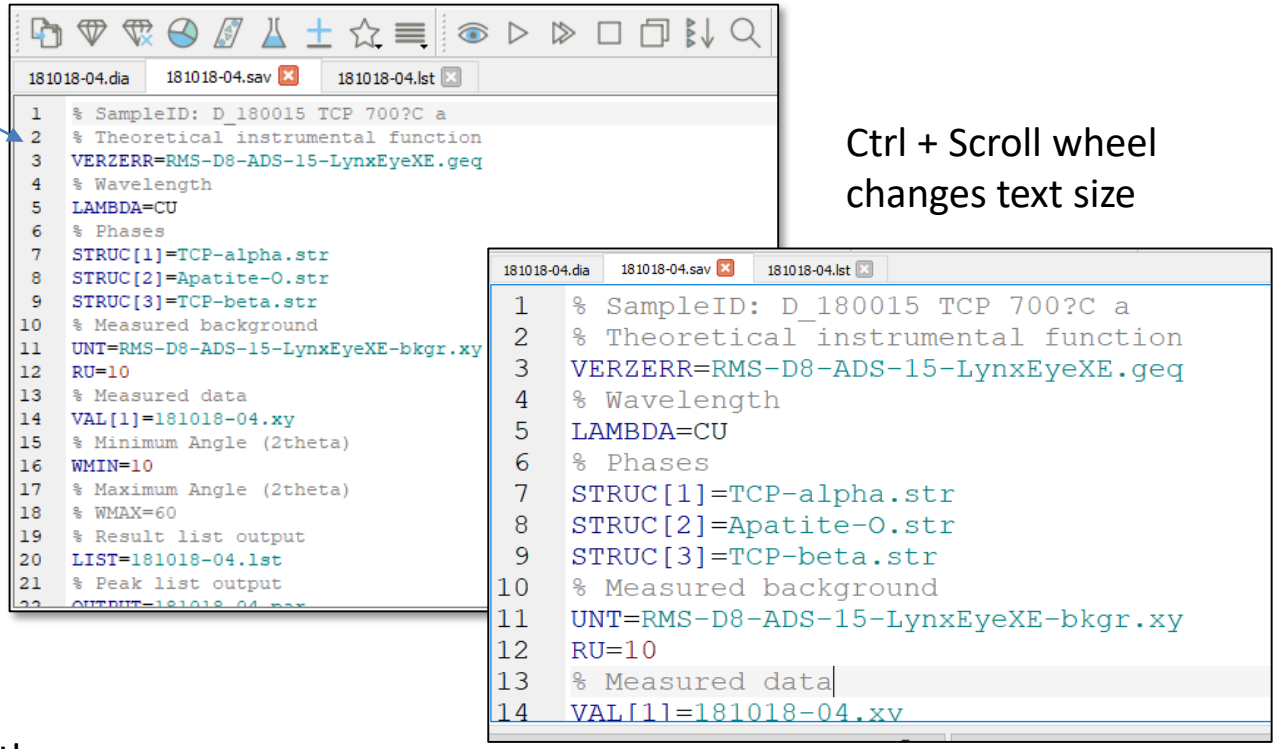
Refined Parameters

Parameter	Value	ESD
Statistics		
Rwp	8.03	
Rexp	7.31	
χ^2	1.21	
GoF	1.10	
Global GOALs		
QalphaTCP	0.6299	0.0029
QOxyapatite	0.0605	0.0016
QbetaTCP	0.3095	0.0030
Local GOALs		
alphaTCP		
A	1.28759	0.00009
B	2.7304	0.0001
C	1.52211	0.00007
BETA	126.231	0.004
GrainSize(1,1,1)	98.8	2.6
Oxyapatite		
A	0.94265	0.00009
C	0.68868	0.00009
GrainSize(1,1,1)	79.2	3.2
betaTCP		
A	1.04064	0.00005
C	3.7314	0.0002
GrainSize(1,1,1)	172.0	14.0

GUI Changes

Text editors:

Line numbers

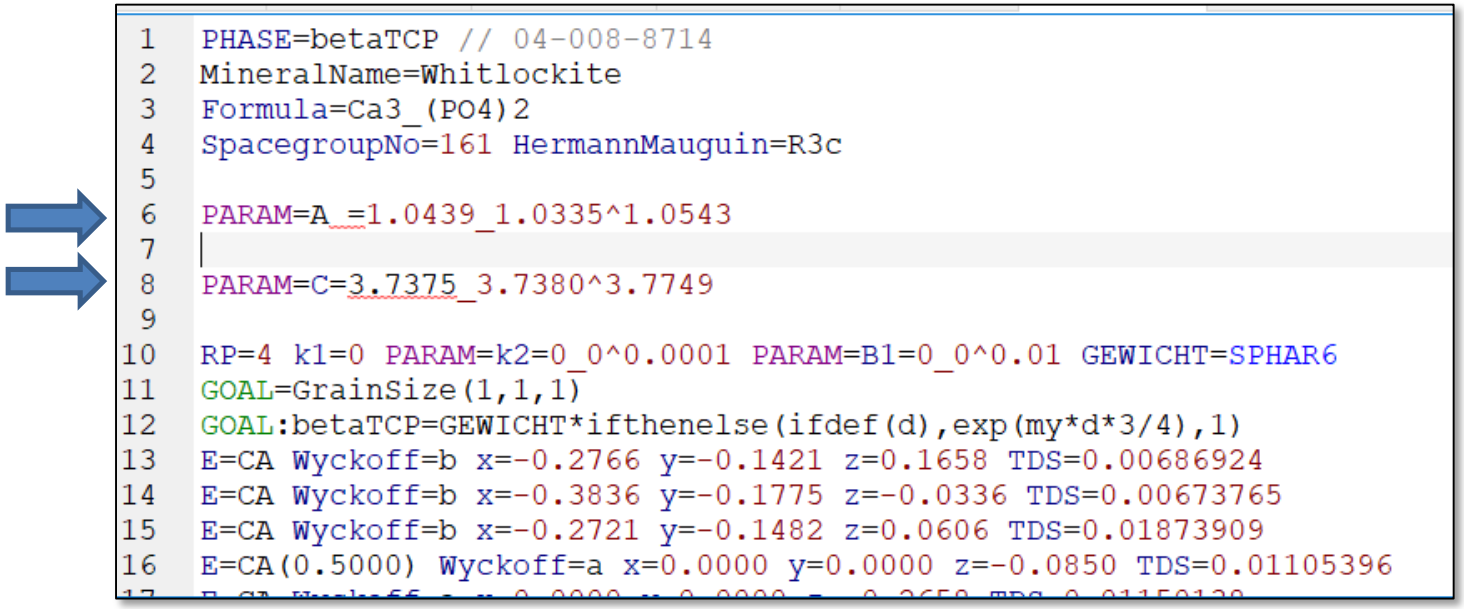


Ctrl + Scroll wheel changes text size

Change permanently:
«Edit → Preferences → Text editors»

GUI Changes

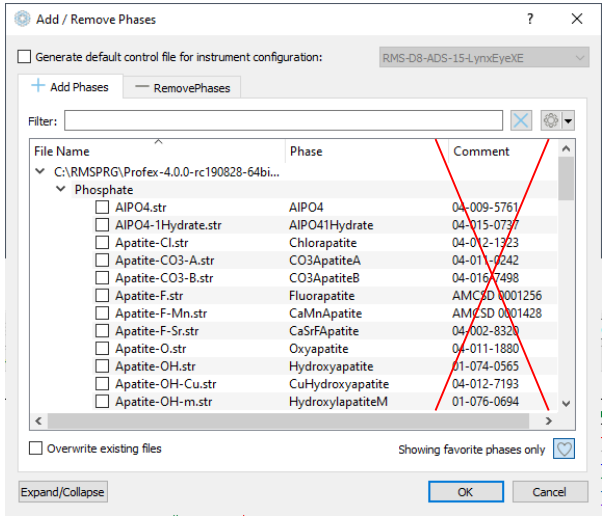
Spell checker underlines common mistakes:

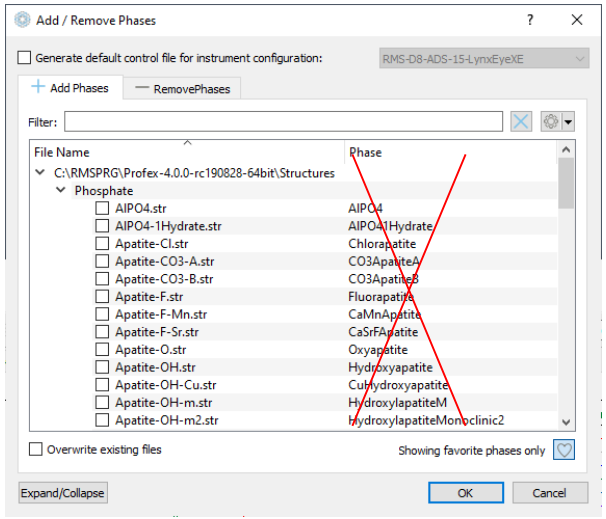


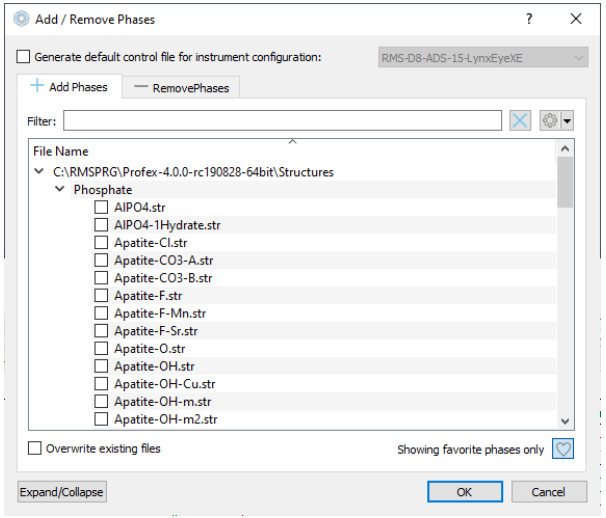
```
1  PHASE=betaTCP // 04-008-8714
2  MineralName=Whitlockite
3  Formula=Ca3_(PO4)2
4  SpacegroupNo=161 HermannMauguin=R3c
5
6  PARAM=A_=1.0439_1.0335^1.0543
7
8  PARAM=C=3.7375_3.7380^3.7749
9
10 RP=4 k1=0 PARAM=k2=0_0^0.0001 PARAM=B1=0_0^0.01 GEWICHT=SPHAR6
11 GOAL=GrainSize(1,1,1)
12 GOAL:betaTCP=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1)
13 E=CA Wyckoff=b x=-0.2766 y=-0.1421 z=0.1658 TDS=0.00686924
14 E=CA Wyckoff=b x=-0.3836 y=-0.1775 z=-0.0336 TDS=0.00673765
15 E=CA Wyckoff=b x=-0.2721 y=-0.1482 z=0.0606 TDS=0.01873909
16 E=CA(0.5000) Wyckoff=a x=0.0000 y=0.0000 z=-0.0850 TDS=0.01105396
```

GUI Changes

Show / hide certain table columns
(for less information clutter)







Right-click on the table header

New Modules: Search/Match and Peak Detection

Previous versions:

Method	Strengths	Limitations
Double-clicking strongest peak	• Very fast	• Fails in case of texture • Internal STR repository only
Scrolling through reference structures	• Largely unaffected by texture	• Manual search (tedious) • Internal STR repository only

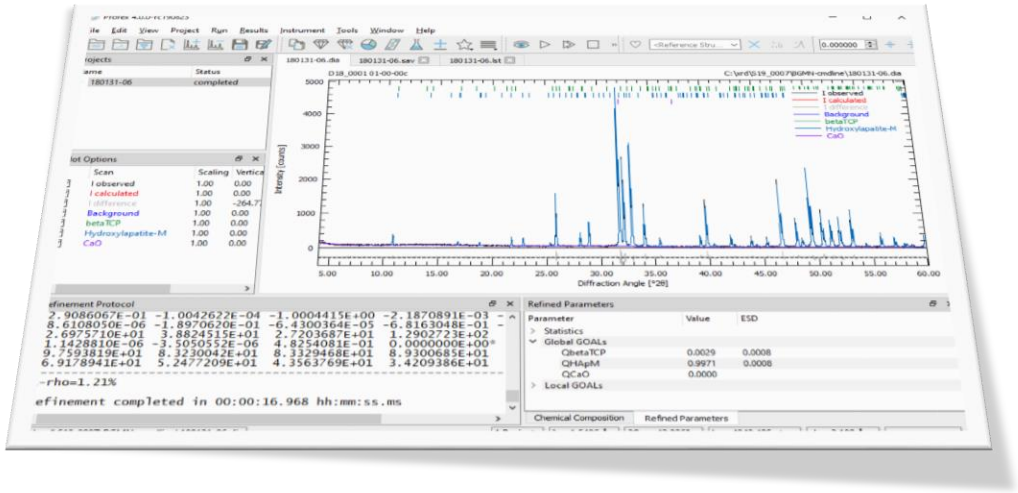
Profex 4:

Method	Strengths	Limitations
Full-profile search-match (derived from [1])	• Good hit rate	• Slow • Internal STR repository only
Peak detection, export to 3rd party software	• Searches COD and PDF databases	• Requires 3rd party software

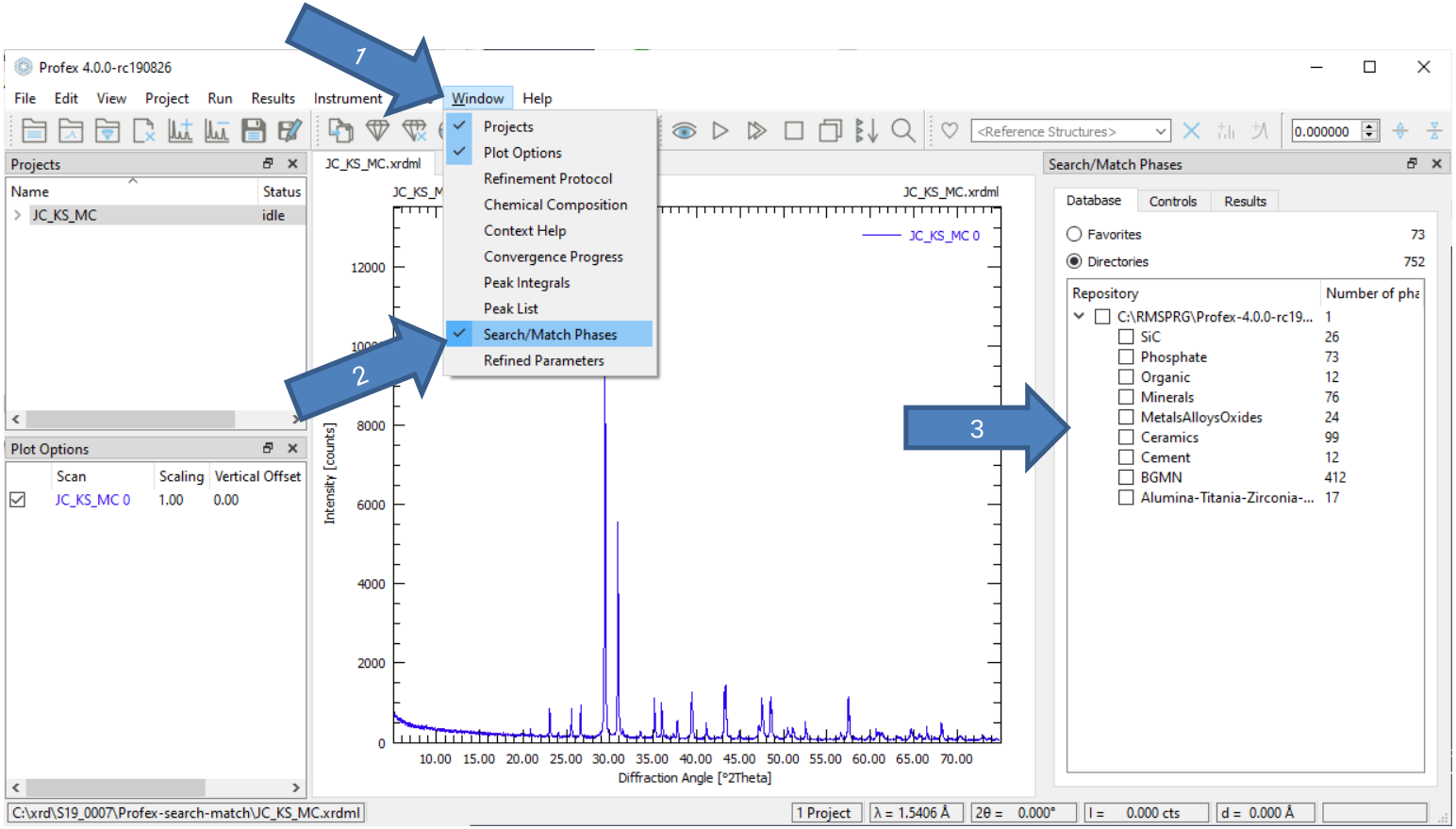
[1] Lutterotti, L., et al., «Full-profile search-match by the Rietveld method», J. Appl. Cryst. 52, 2019, 1-12.

Full-Profile Search-Match

- Refines each structure file separately
- Uses a restricted refinement strategy
  (no texture, no anisotropic parameters, limited number of iterations)
- Scores the structure files by the refinement statistics (Figure of Merit, FoM)
  (see Profex User Manual Part 2)
- Requires a correct instrument configuration



Full-Profex Search-Match

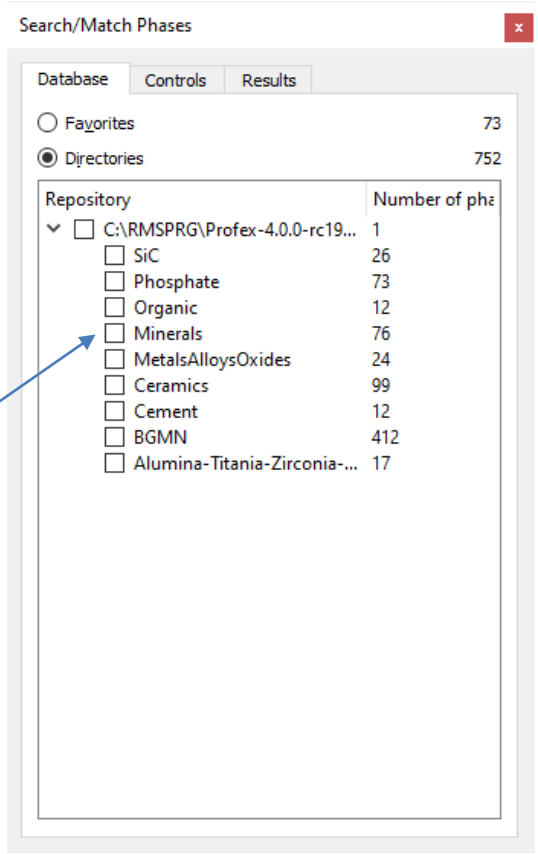


Full-Profile Search-Match

Page «Database»:

- Select the subset of the Profex structure file Repository to be searched.
- Searching among all 752 structures will take a long time…

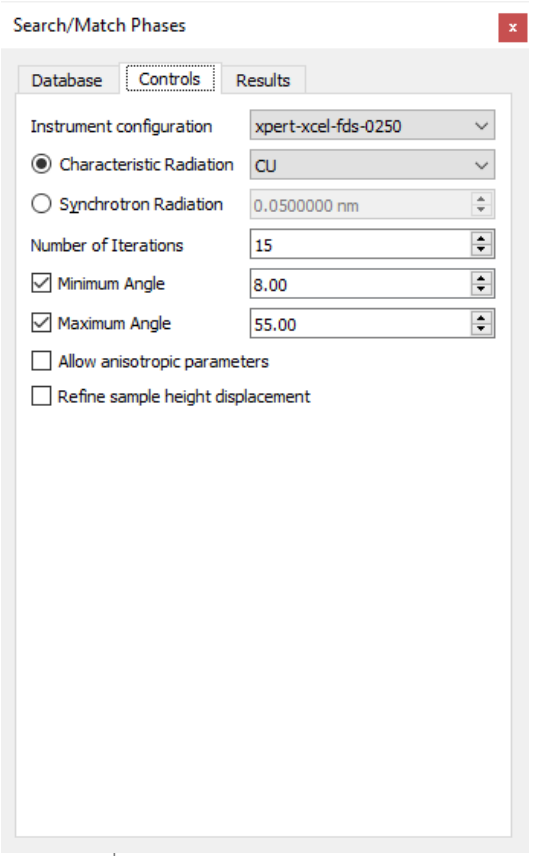
For our example: Check «Minerals»



Full-Profile Search-Match

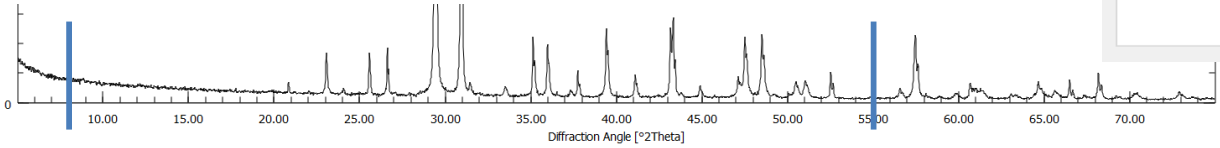
Page «Controls»:

- Select the instrument configuration used to measure the dataset.
- Select the wavelength distribution.
- Specify the number of iterations and angular range to be used for phase matching.



For our example:

Instrument = xpert-xcel-fds-0250
Radiation = CU
Number of iterations = 15
Minimum angle = 8.0
Maximum angle = 55.0

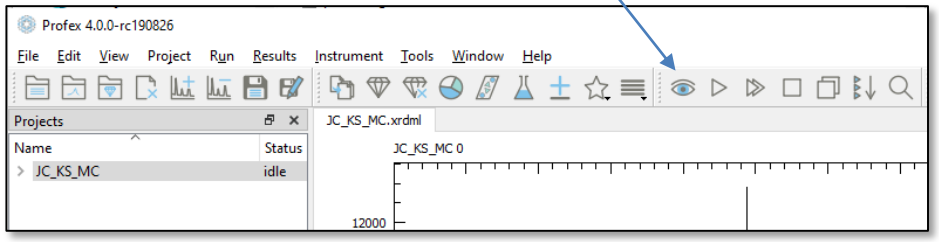


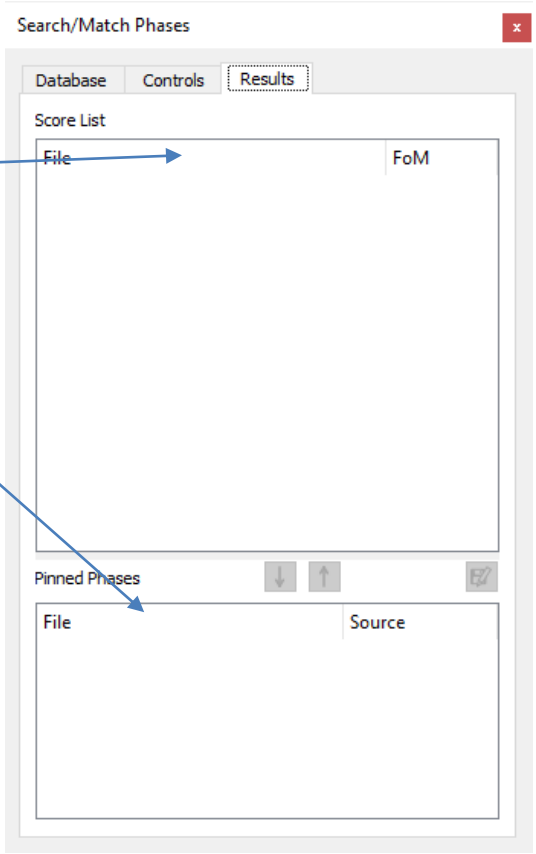
Full-Profile Search-Match

Page «Results»:

- No results yet
- Columns «Phase» and «Fraction» can be hidden (right mouse button)

Start Search/Matching:

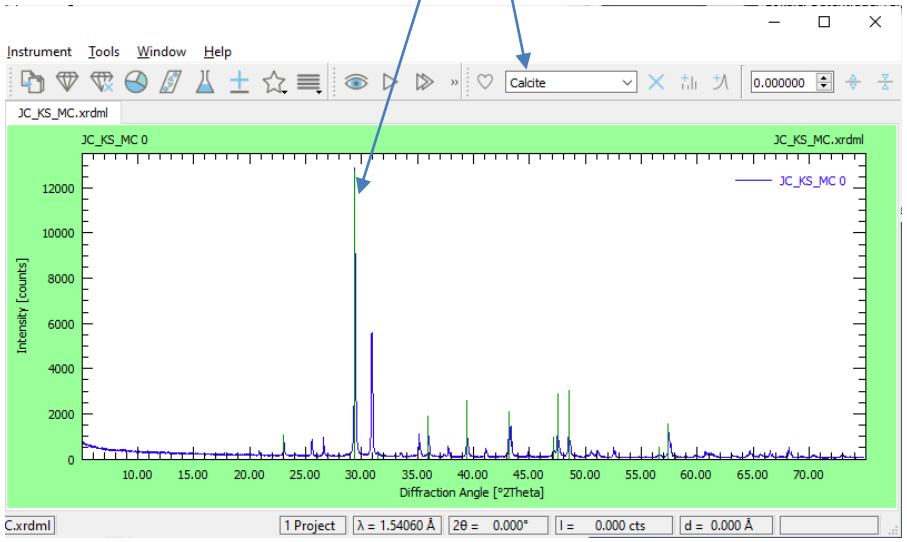


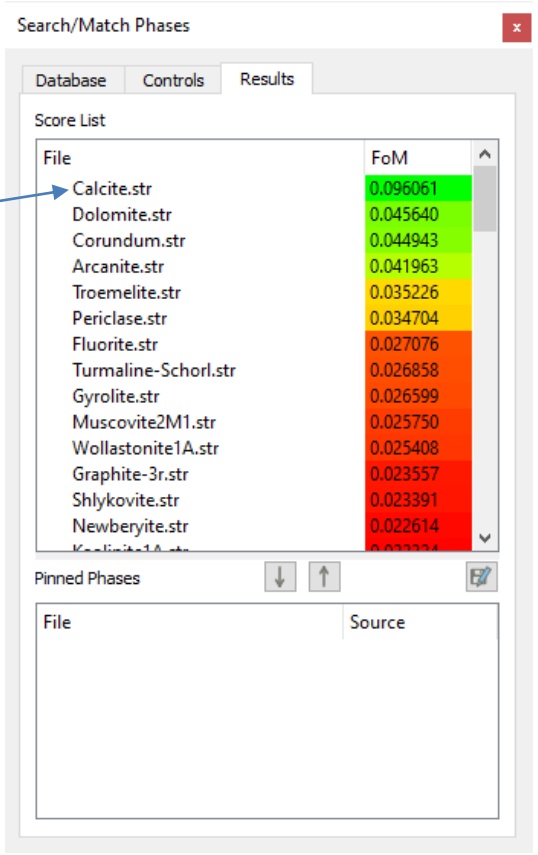


Full-Profile Search-Match

Page «Results»:

- Best matches appear on top of the list
- Absolute FoM has no meaning,
  varies strongly from dataset to dataset
- Click on phase to show hkl lines

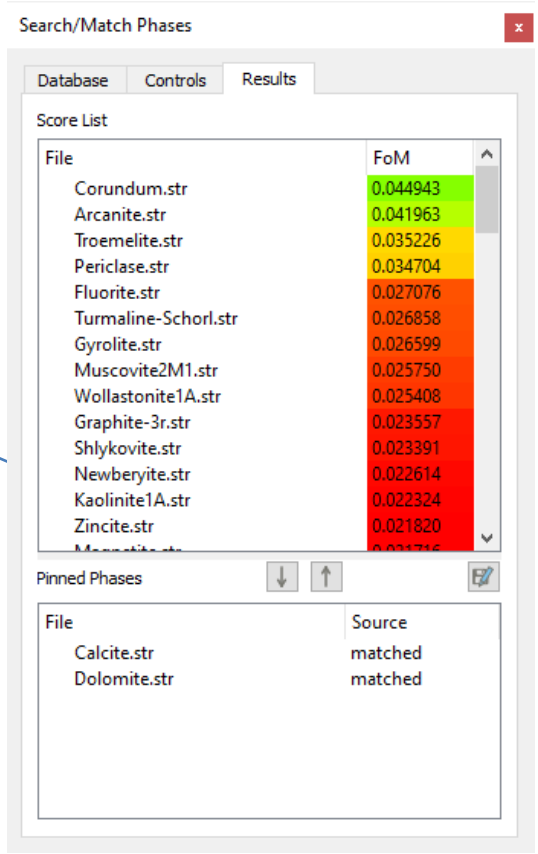




Full-Profile Search-Match

Page «Results»:

- To accept matches, select the file and pin it

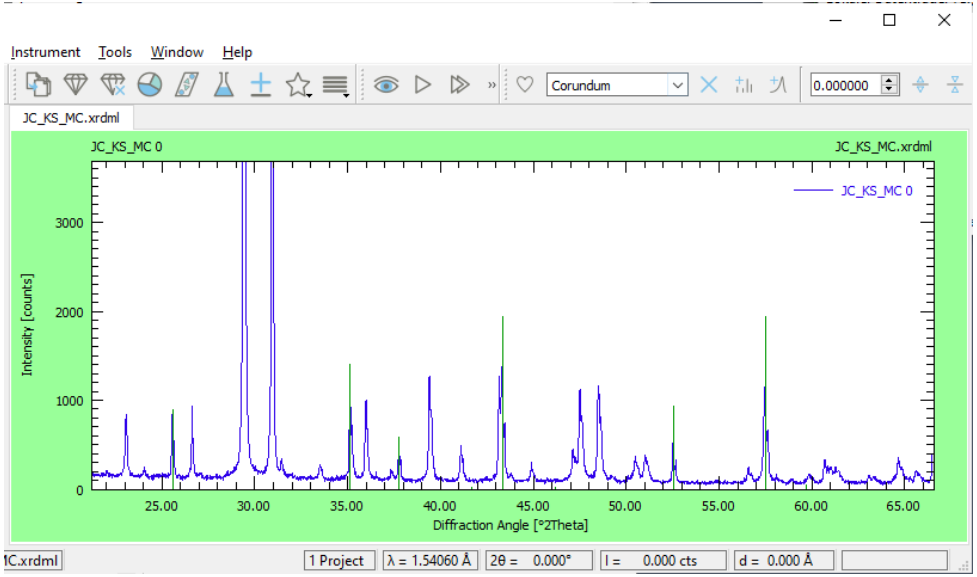


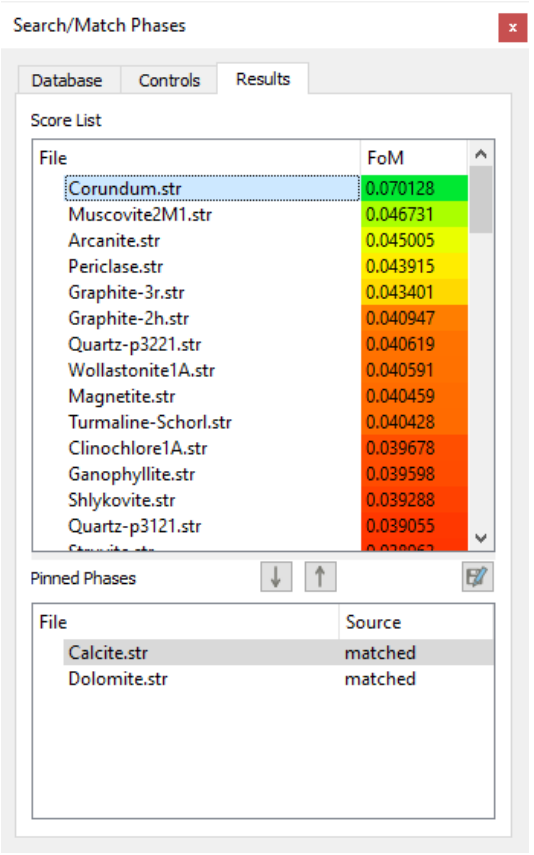
Run Search/Match again to perform a residual search

Full-Profile Search-Match

Page «Results»:

- Residual search results
- Pin Corundum.str
- Run another residual search…

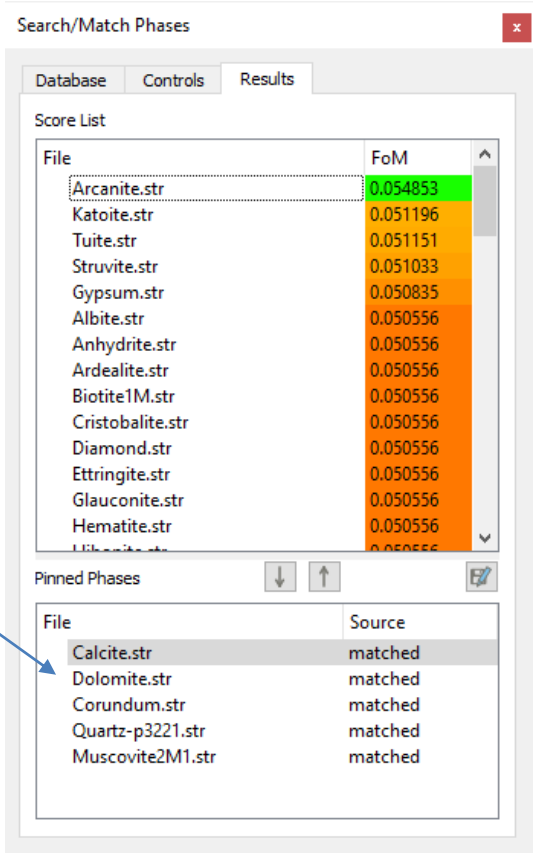




Full-Profile Search-Match

Page «Results»:

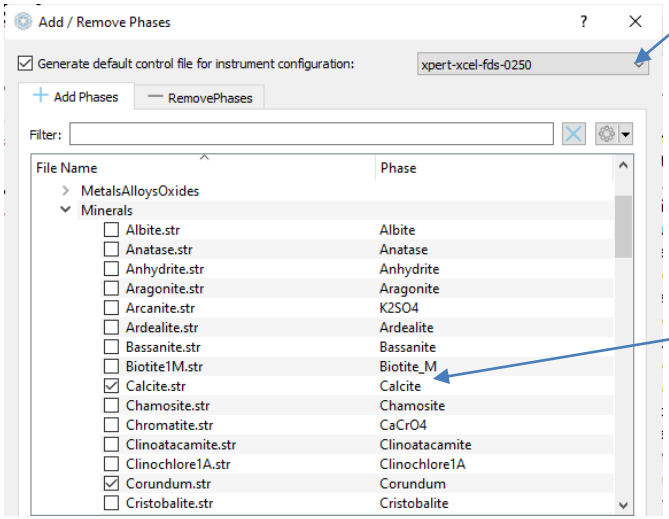
- Arcanite is a false positive
  due to proximity of dolomite peaks.
- Final match list:

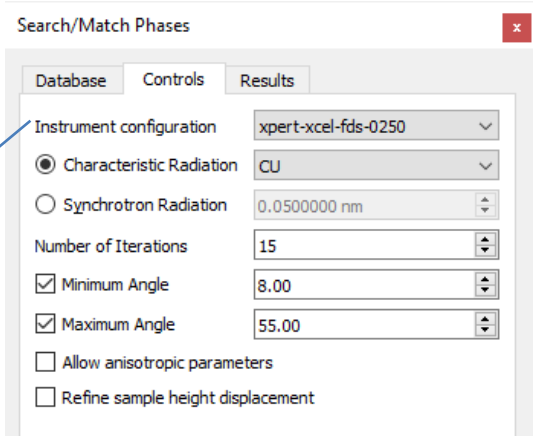


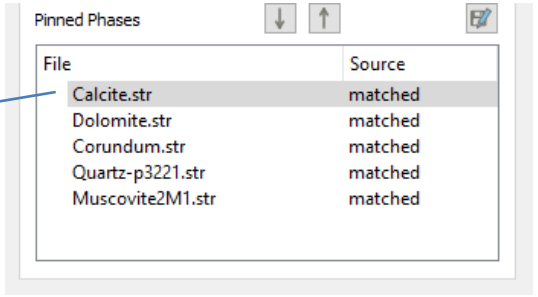
Full-Profile Search-Match

Page «Results»:

- Create a refinement project from the pinned phases: 
- Instrument settings are applied and pinned phases are pre-selected

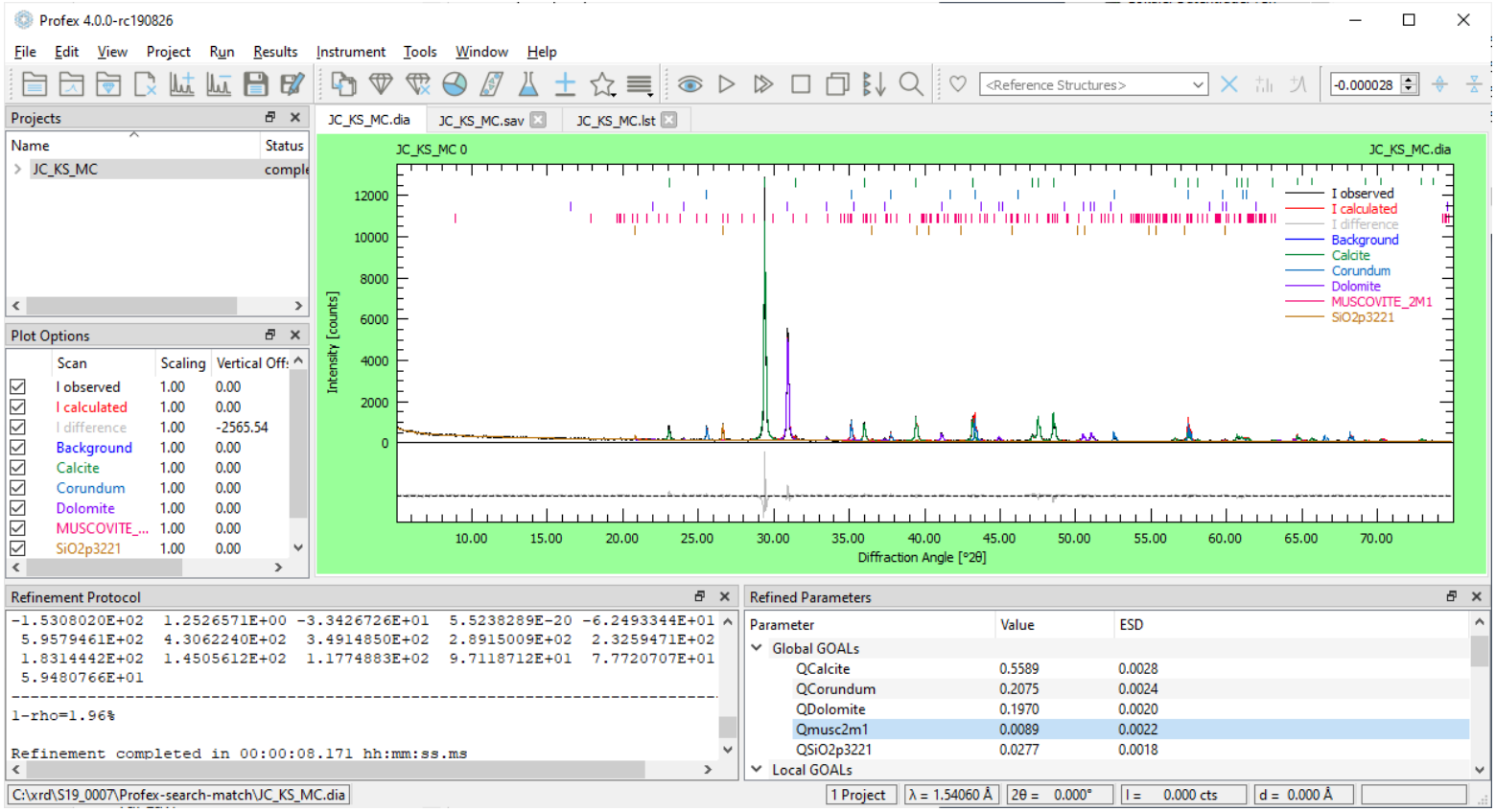






Full-Profile Search-Match

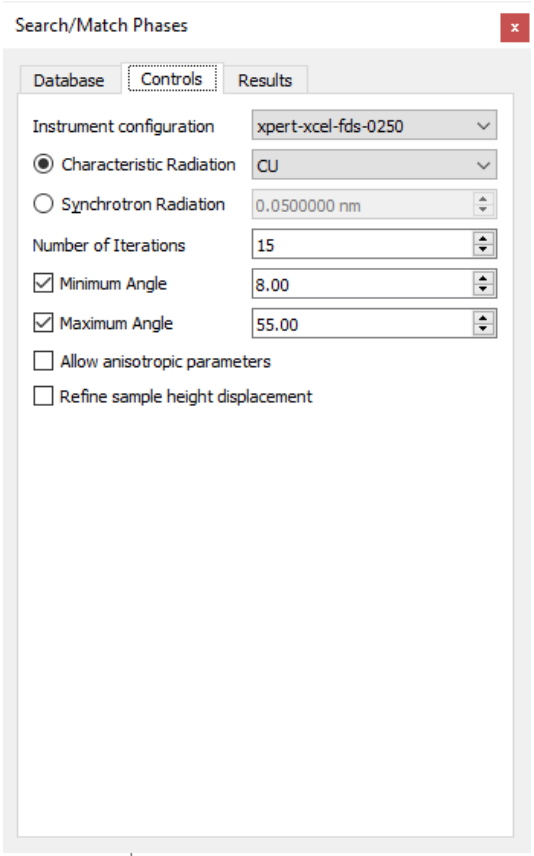
Refinement can be started immediately…



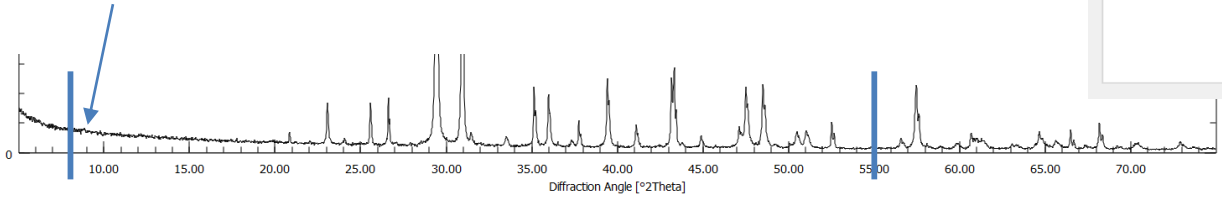
Full-Profile Search-Match

Speed up the process:

- Reduce number of iterations (typically 10 – 15)
- Increase minimum angle to just below the first observed peak
- Reduce maximum angle



Do not clip low-angle peaks!

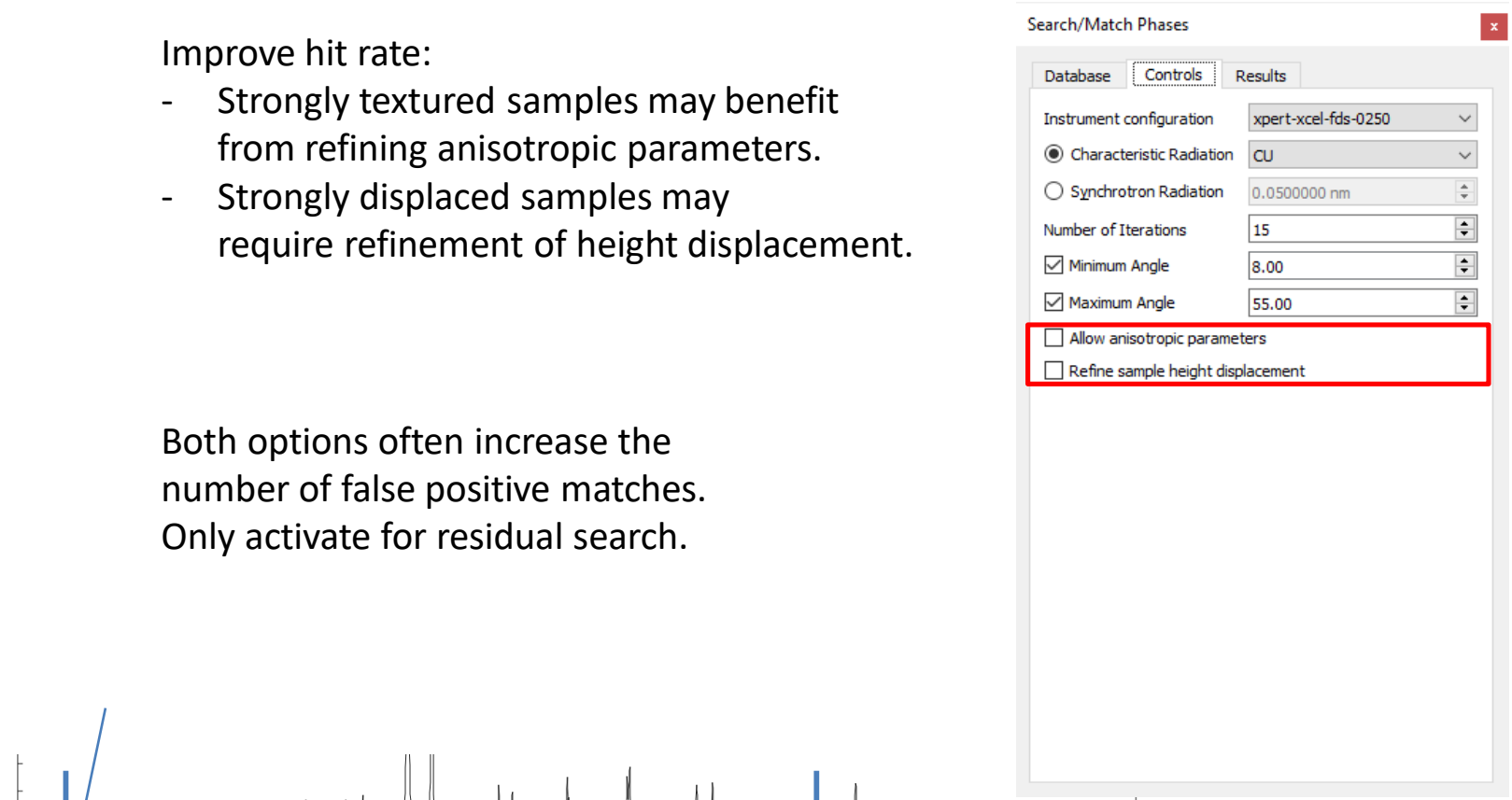


Full-Profile Search-Match

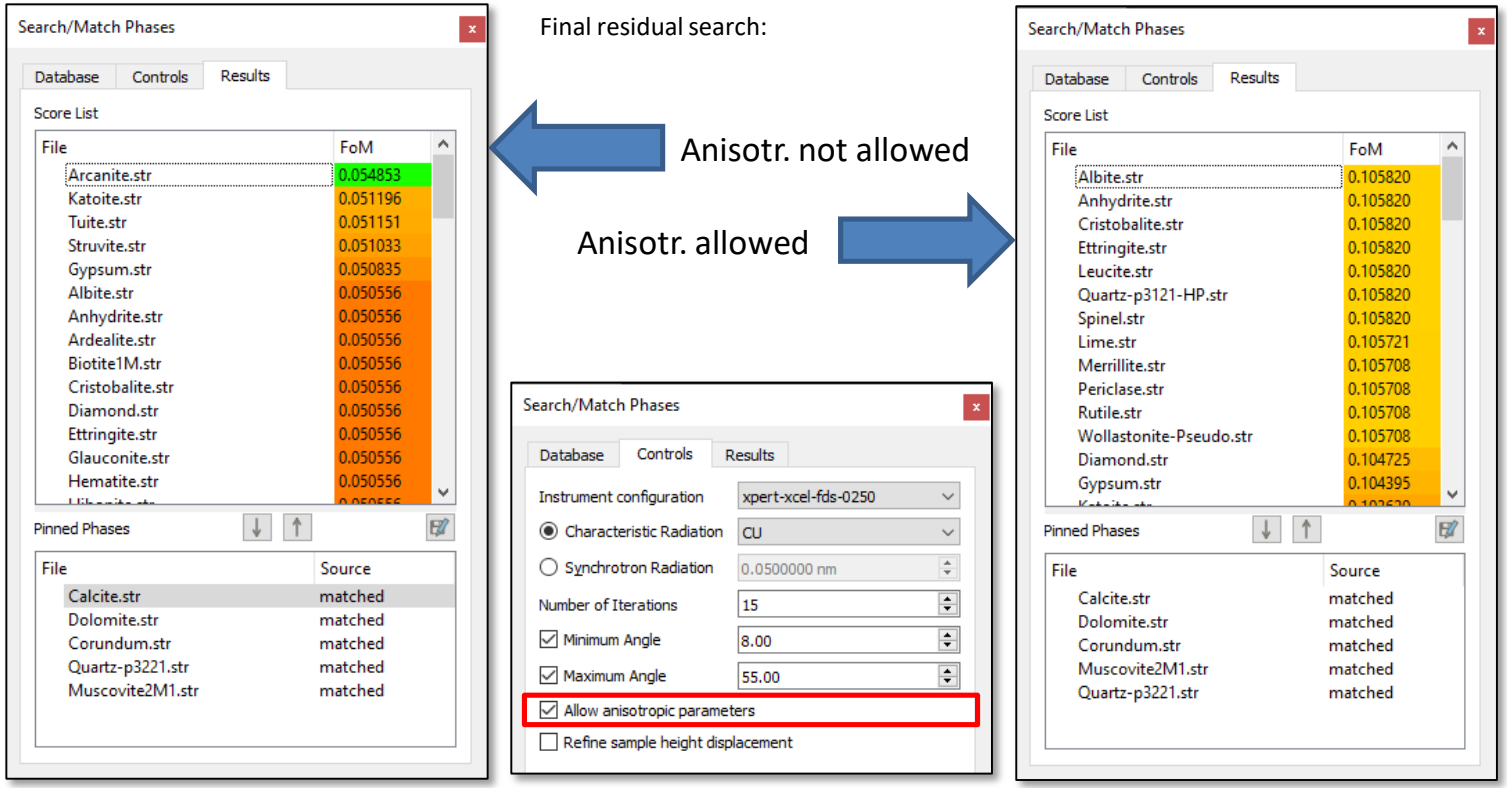
Improve hit rate:

- Strongly textured samples may benefit from refining anisotropic parameters.
- Strongly displaced samples may require refinement of height displacement.

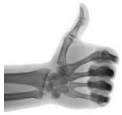
Both options often increase the number of false positive matches.
Only activate for residual search.



Full-Profile Search-Match

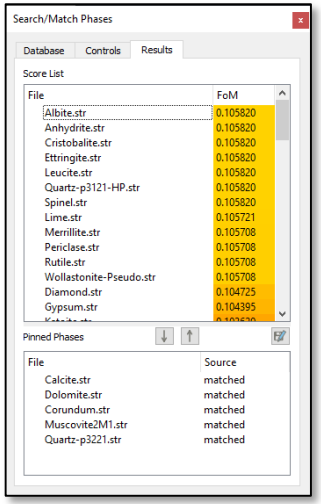


Full-Profile Search-Match

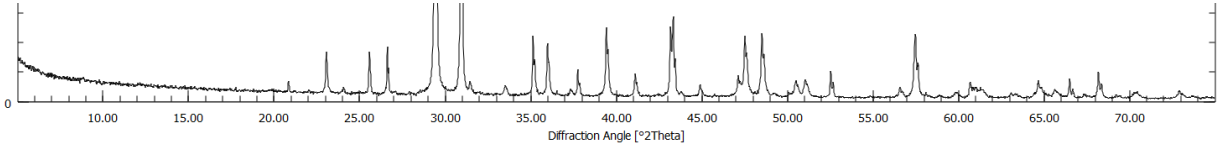


Summary:

- Full-profile search-match with good hit rate
- Excellent residual search hit rate
- Easily create refinement projects from matched phases

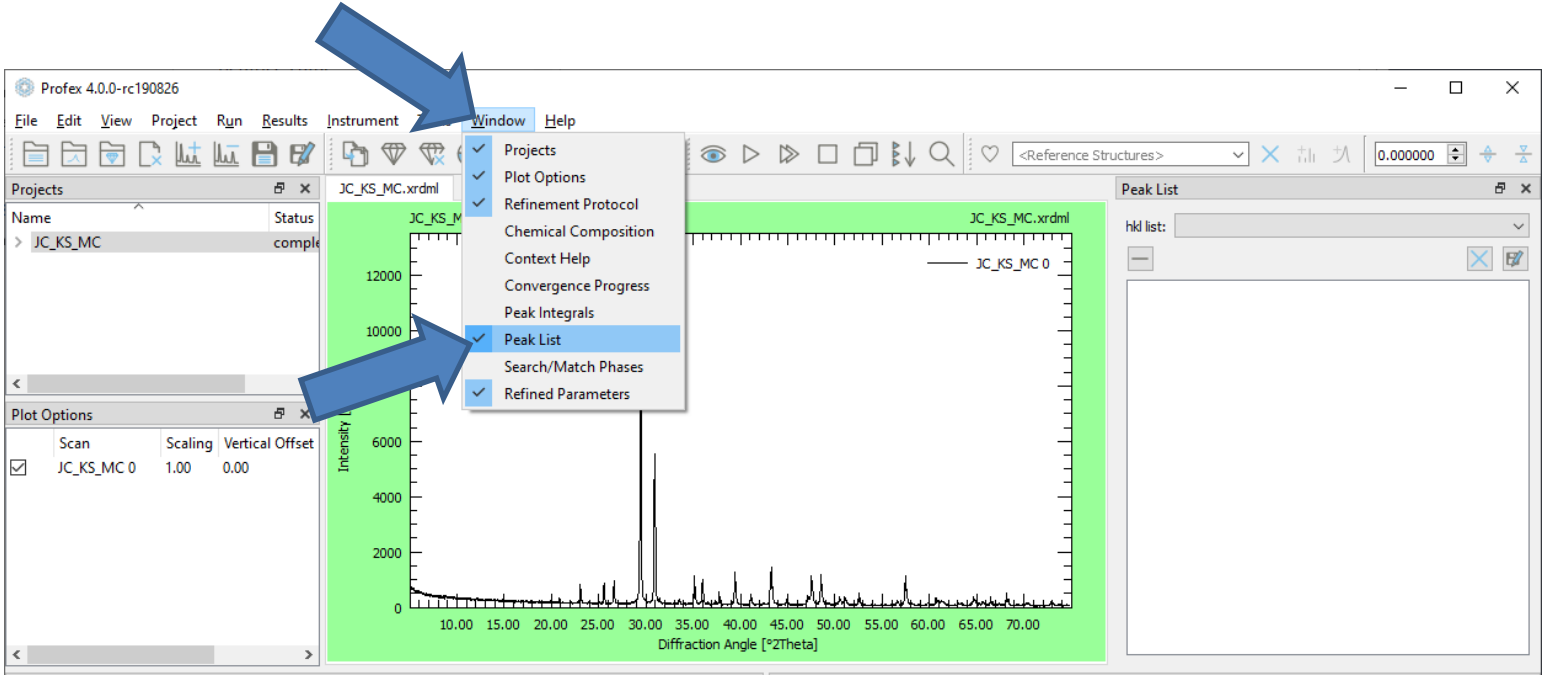


- Slow
- Only searches internal structure repository

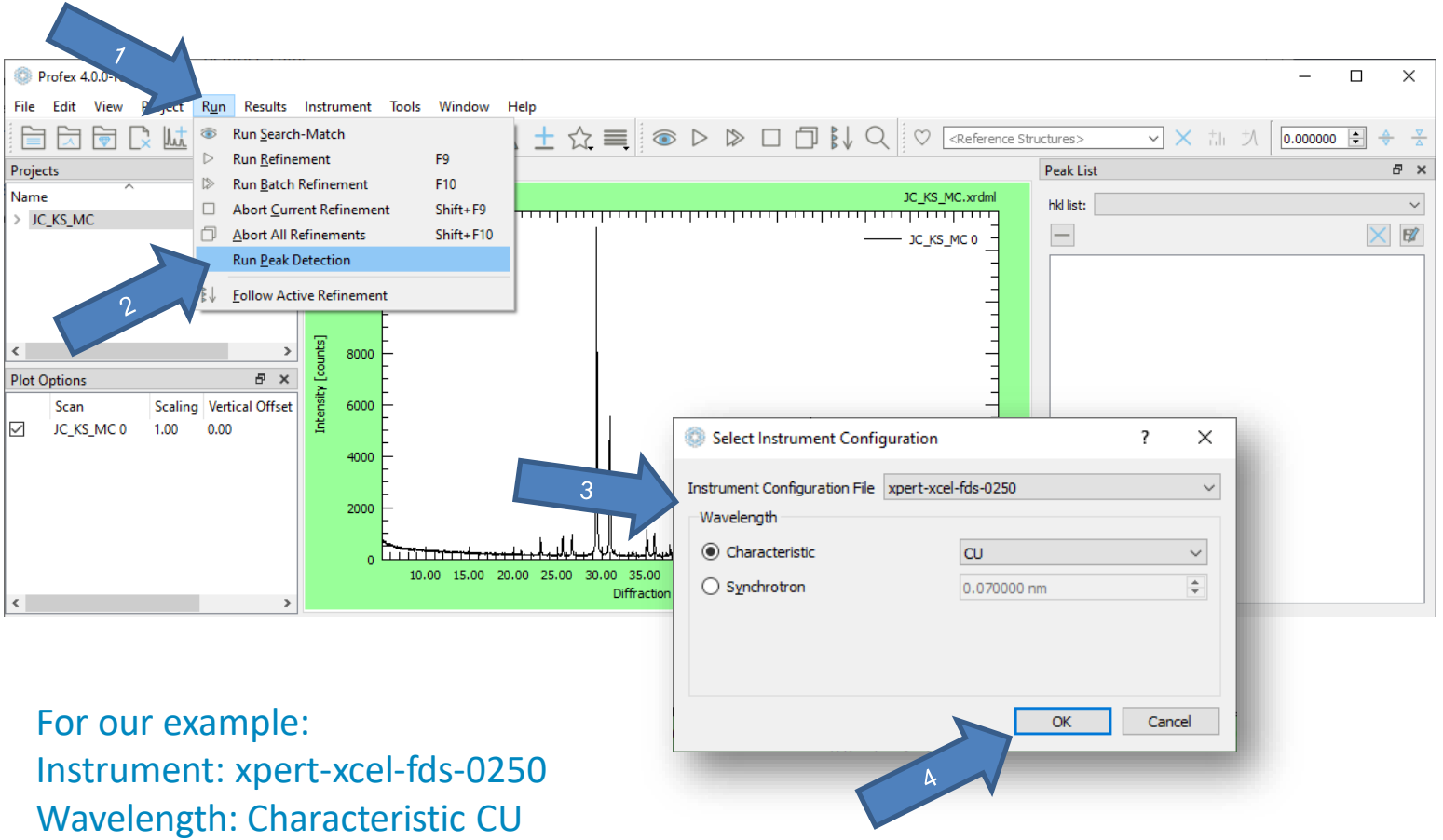


Peak Detection

Peak detection with EFLECH (part of BGMN)
Export peak list to 3rd party Search/Match software

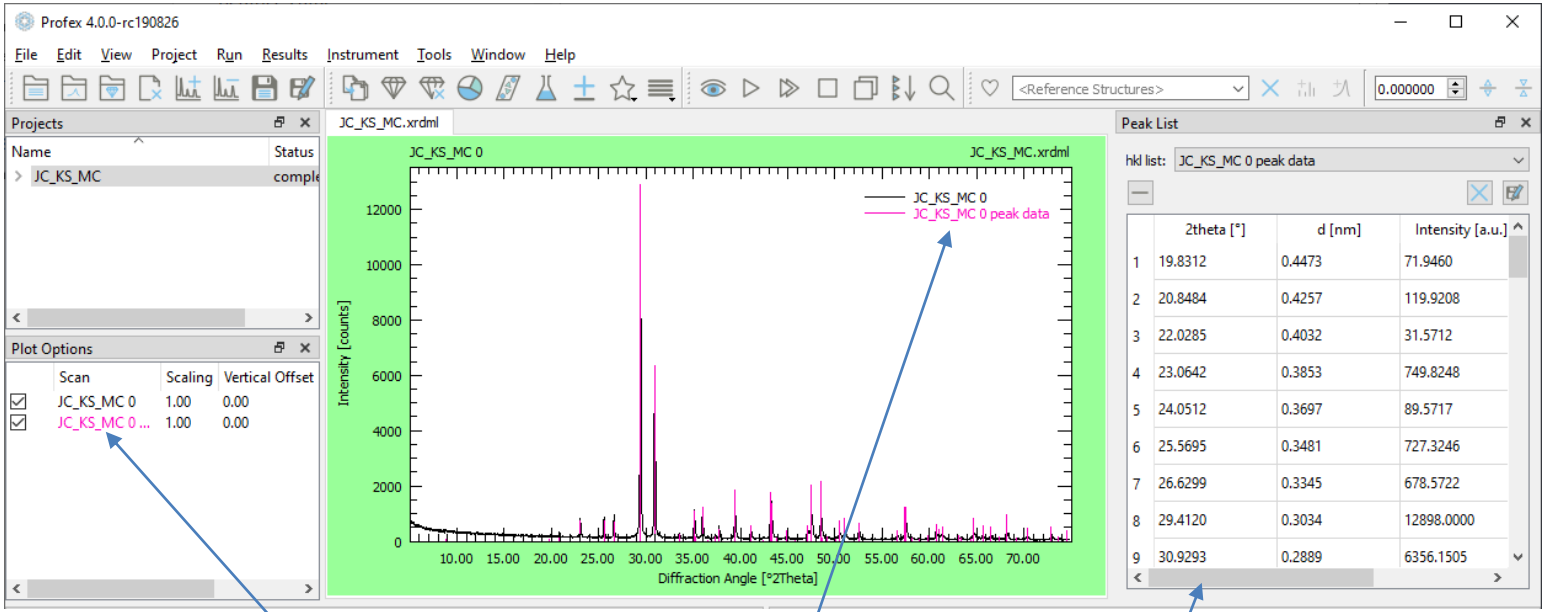


Peak Detection



For our example:
Instrument: xpert-xcel-fds-0250
Wavelength: Characteristic CU

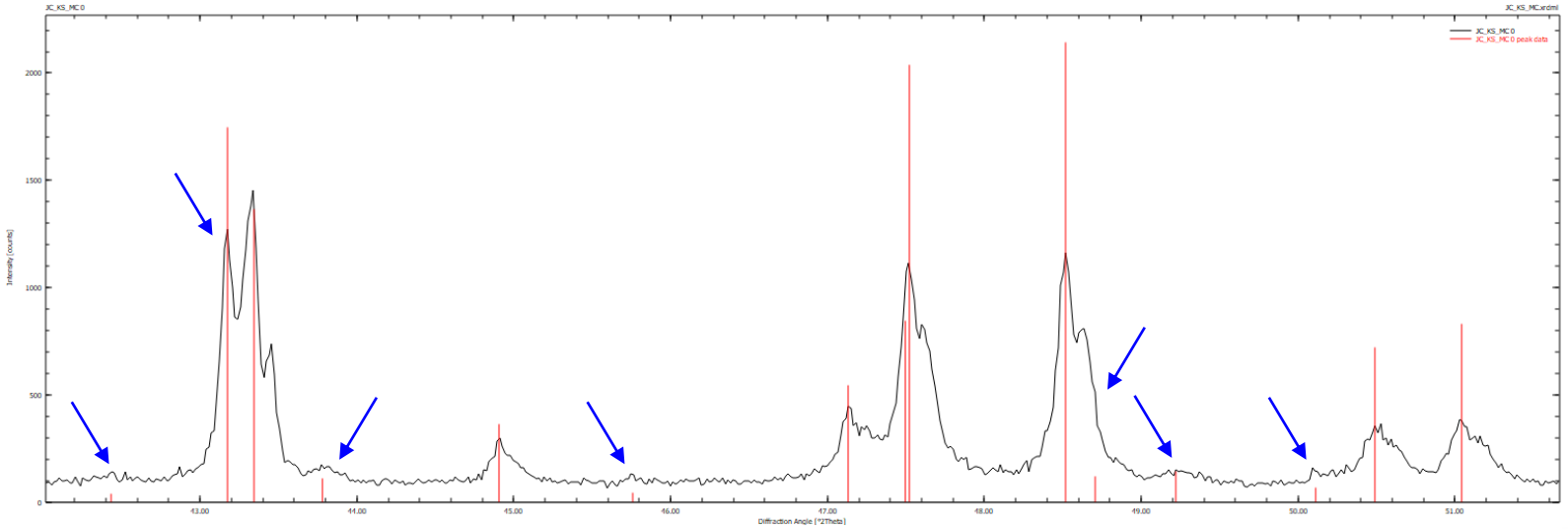
Peak Detection



List of detected peaks is appended as a new hkl scan

Peak Detection

- Correct identification of $K\alpha_2$ and $K\beta$ peaks
- Outstanding deconvolution of overlapping peaks
- Very sensitive to low-intensity peaks



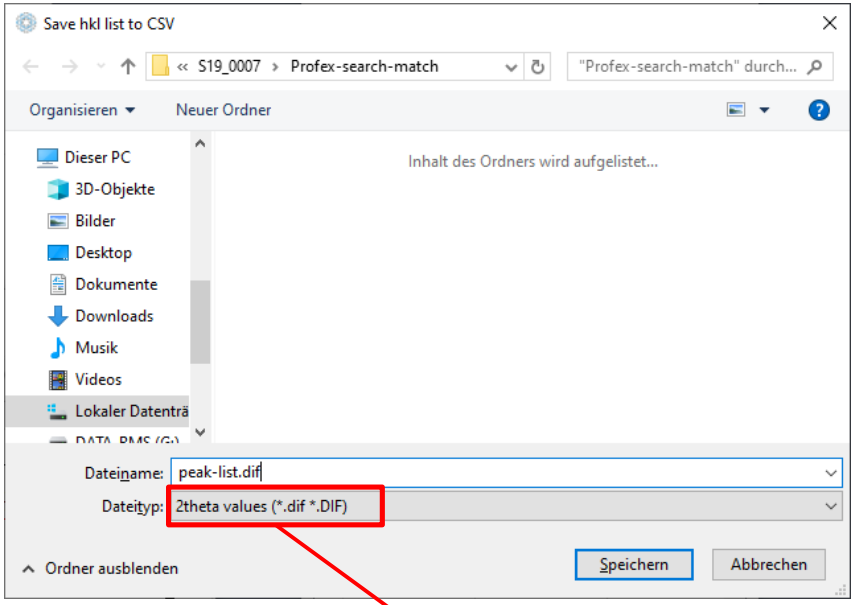
Peak Detection

Peak List

hkl list: JC_KS_MC 0 peak data

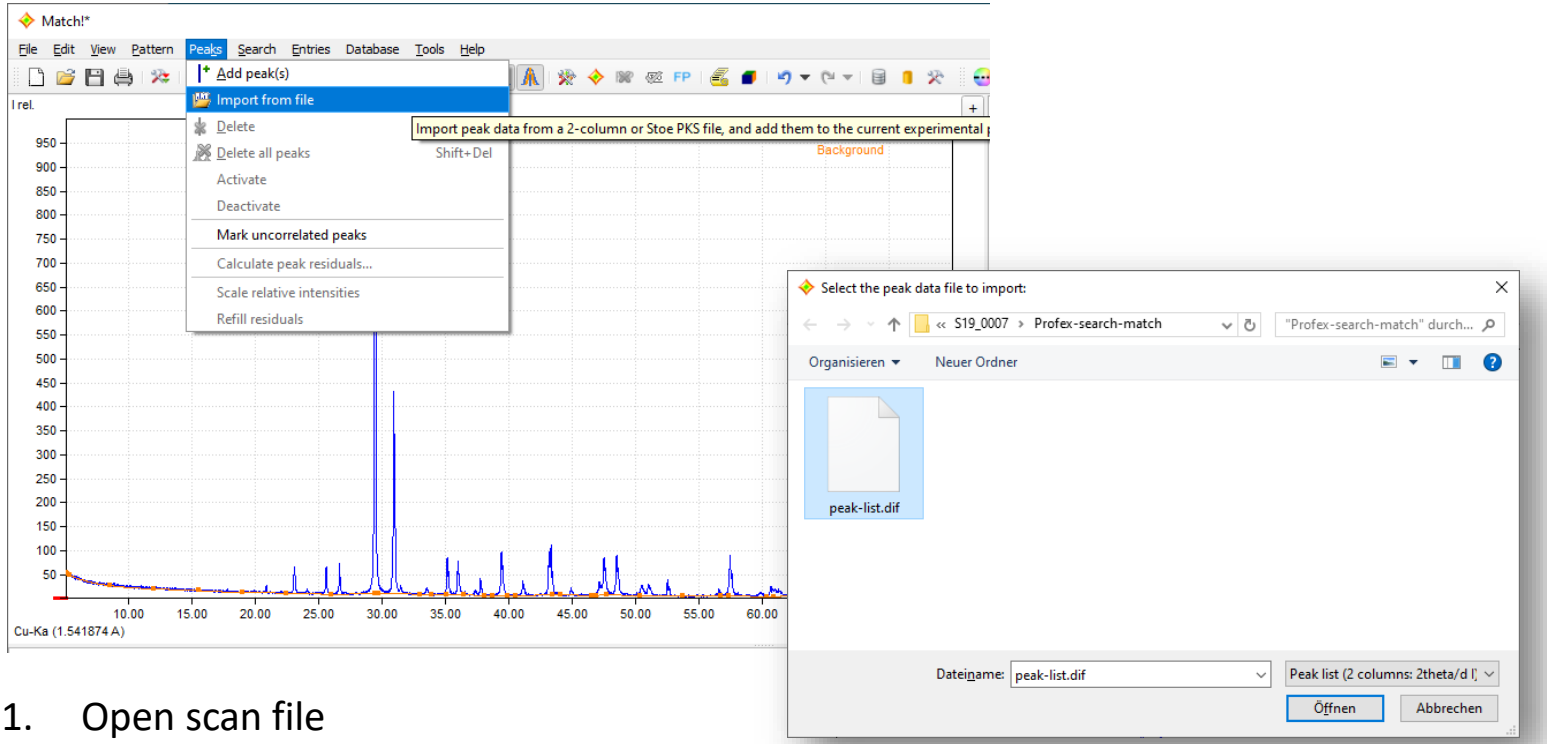
	2theta [°]	d [nm]	Intensity [a.u.]
1	19.8312	0.4473	71.9460
2	20.8484	0.4257	119.9208
3	22.0285	0.4032	31.5712
4	23.0642	0.3853	749.8248
5	24.0512	0.3697	89.5717
6	25.5695	0.3481	727.3246
7	26.6299	0.3345	678.5722
8	29.4120	0.3034	12898.0000
9	30.9293	0.2889	6356.1505
10	33.5049	0.2672	231.2011
11	35.1395	0.2552	1093.2758
12	35.2735	0.2542	141.1452
13	35.9862	0.2494	1211.9070
14	36.5304	0.2458	35.0244

Save peak list to file



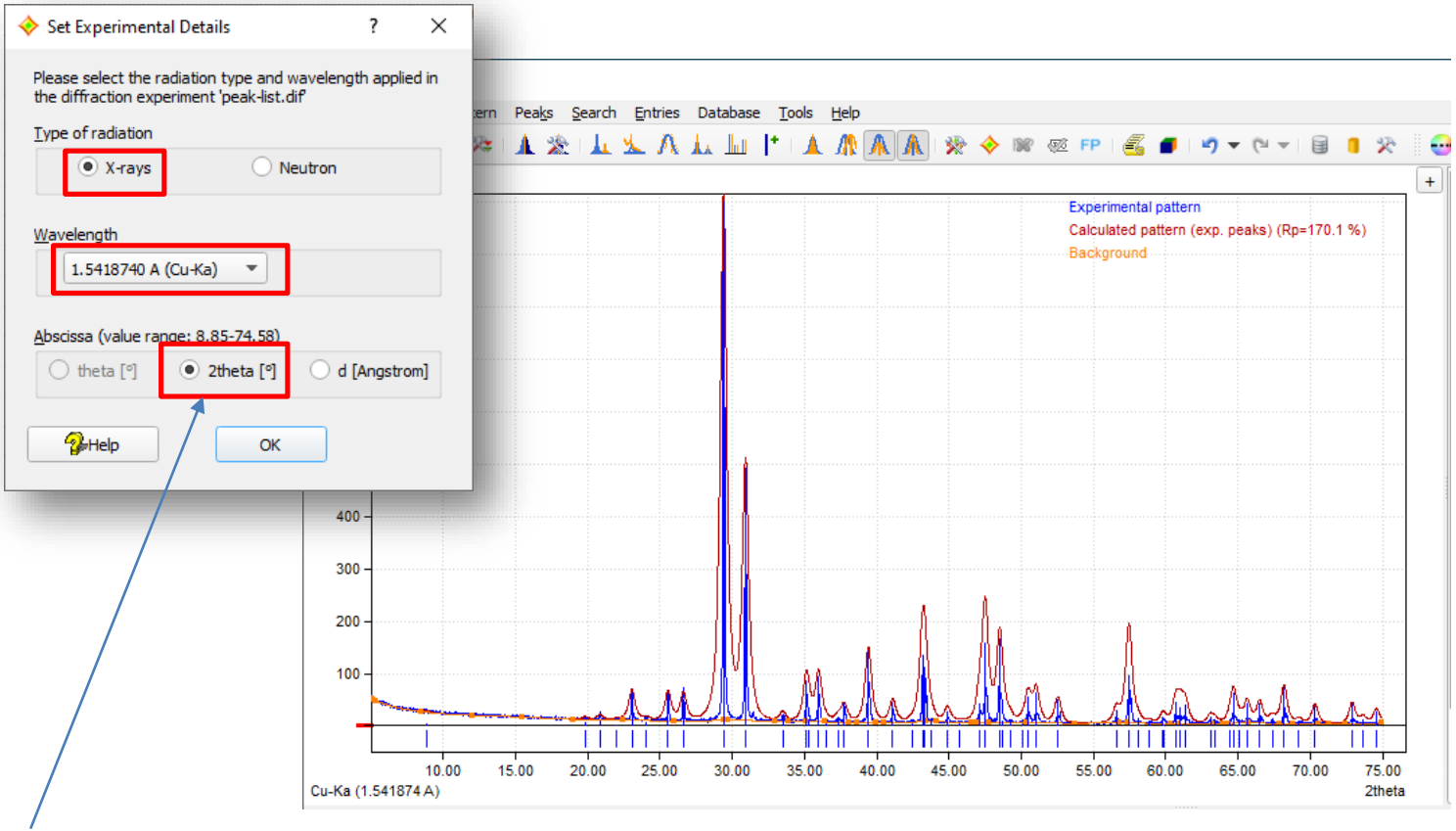
Set format to «2theta values (*.dif)»

Import Peak List in Match!



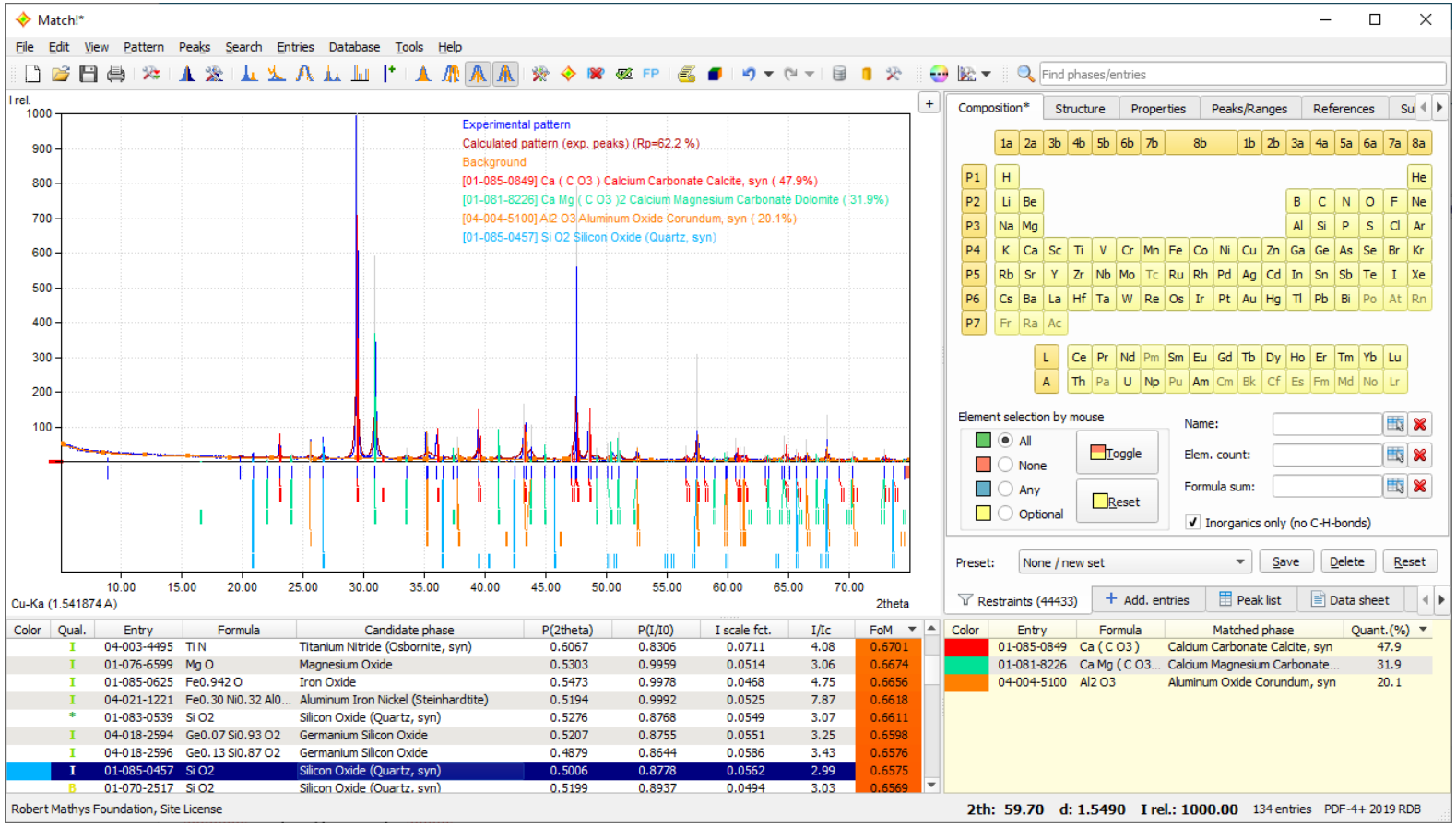
1. Open scan file
2. Select «Peaks → Import from file»
3. Format «Peak list (2 columns: 2theta/d I)»

Import Peak List in Match!



When exported as «d values (*.dif)» from Profex,
select «d [Angstrom]» here.

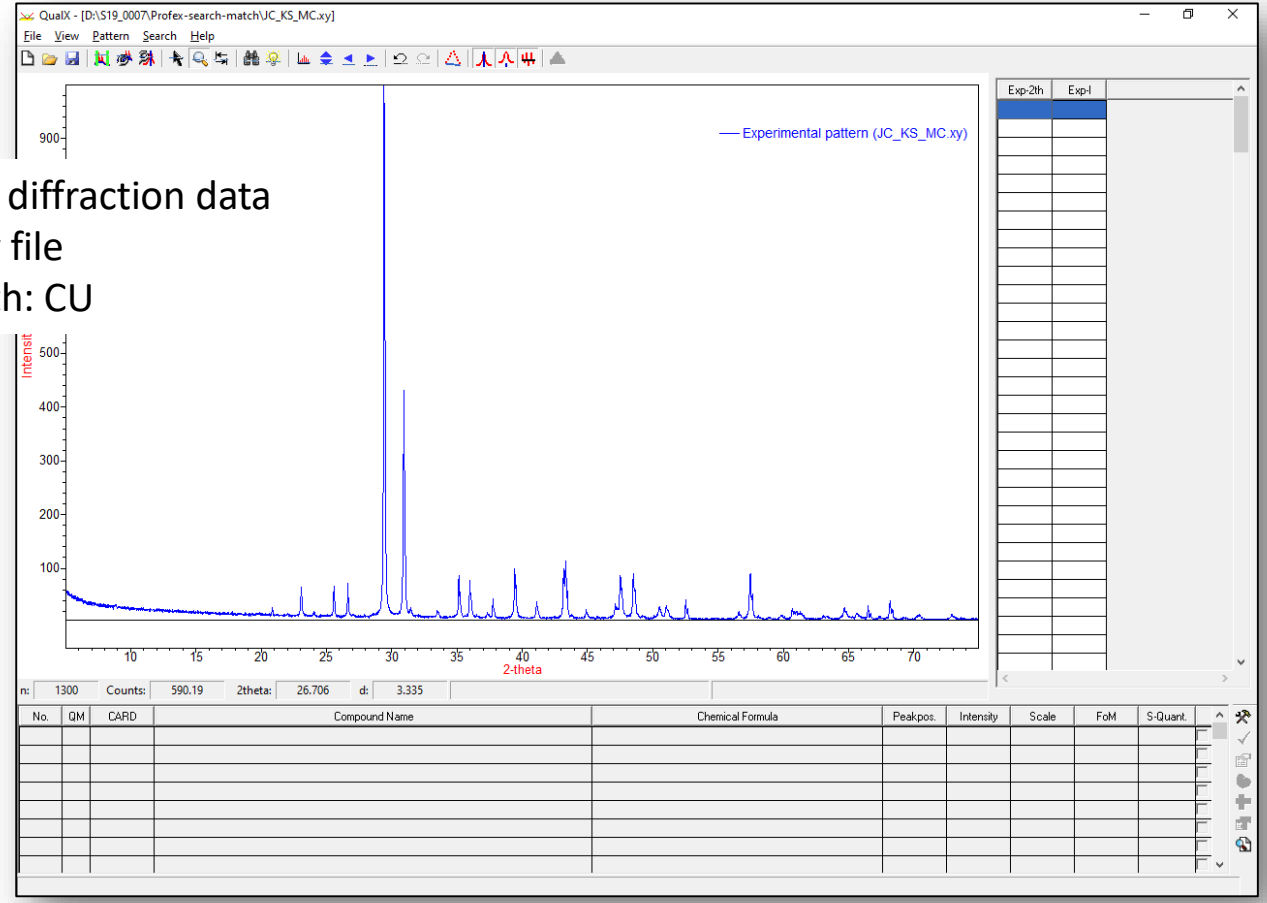
Import Peak List in Match!



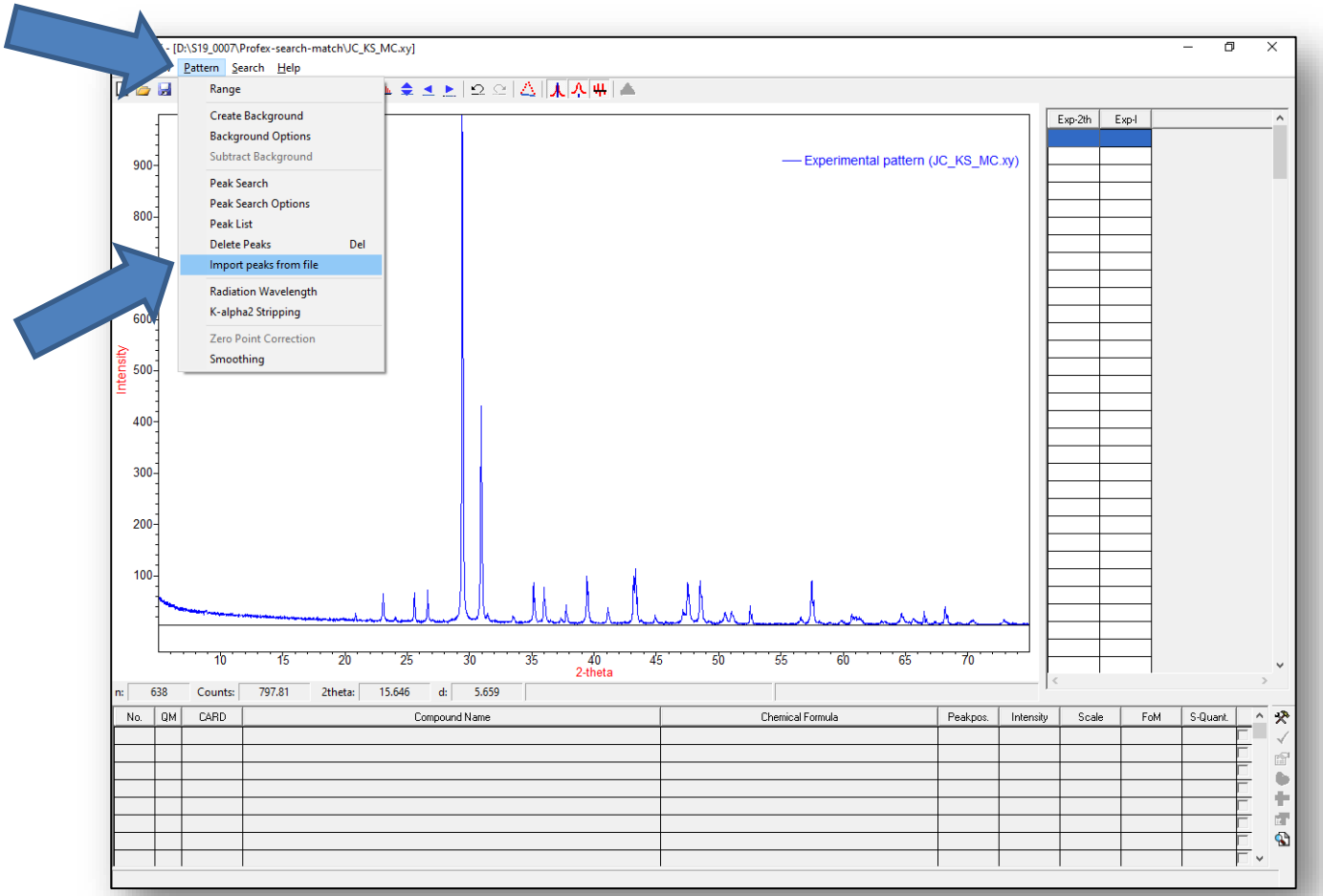
Import Peak List in QualX2

File → Import diffraction data

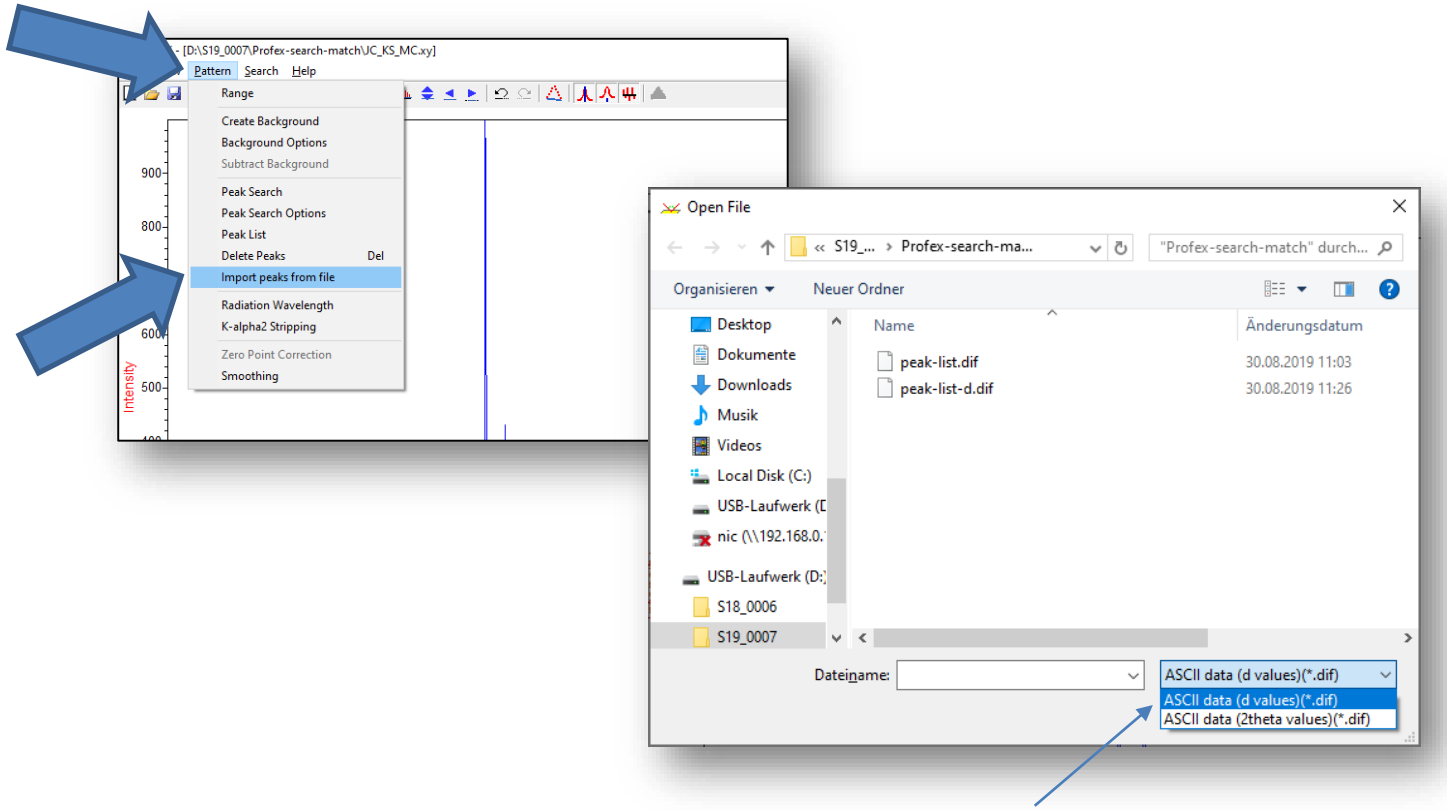
- Select *.xy file
- Wavelength: CU



Import Peak List in QualX2

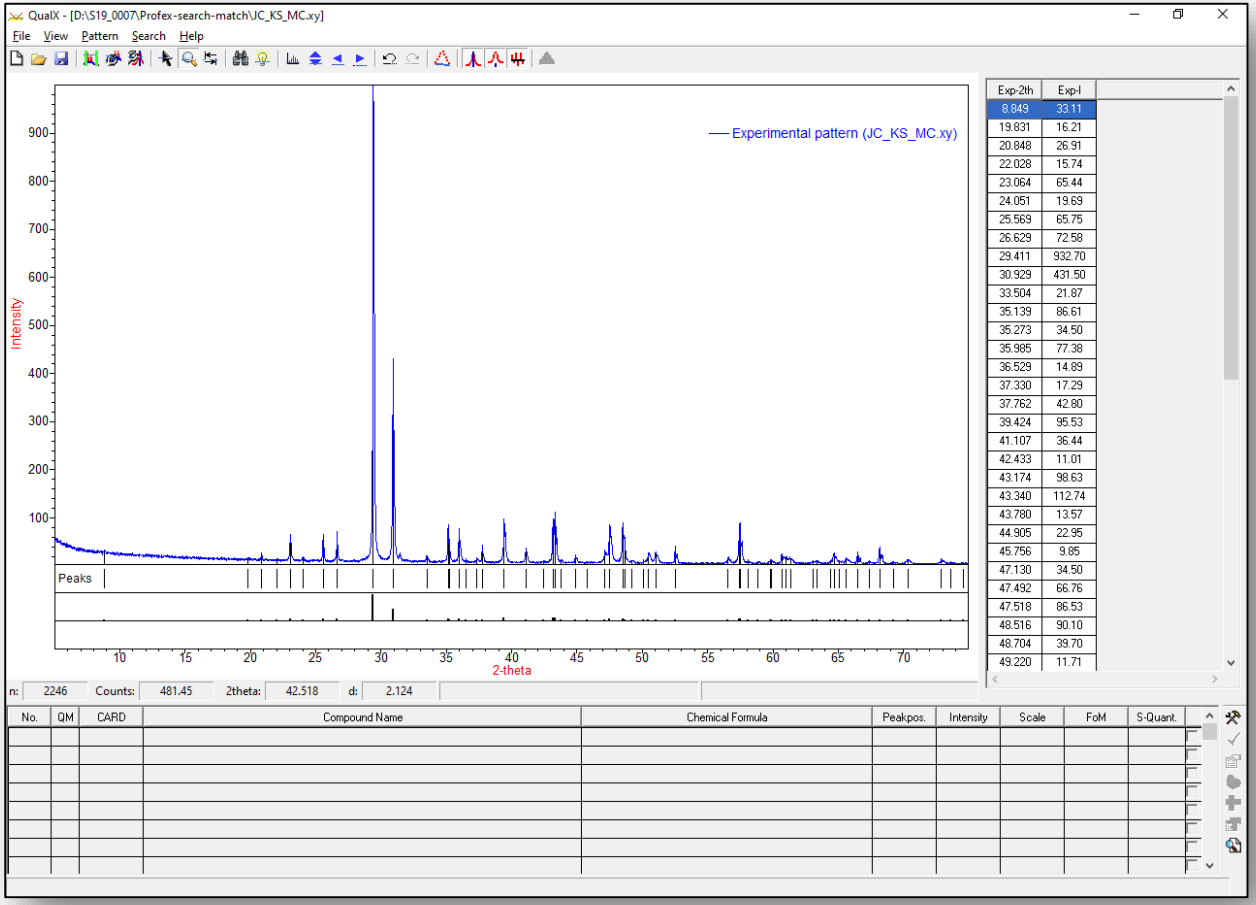


Import Peak List in QualX2

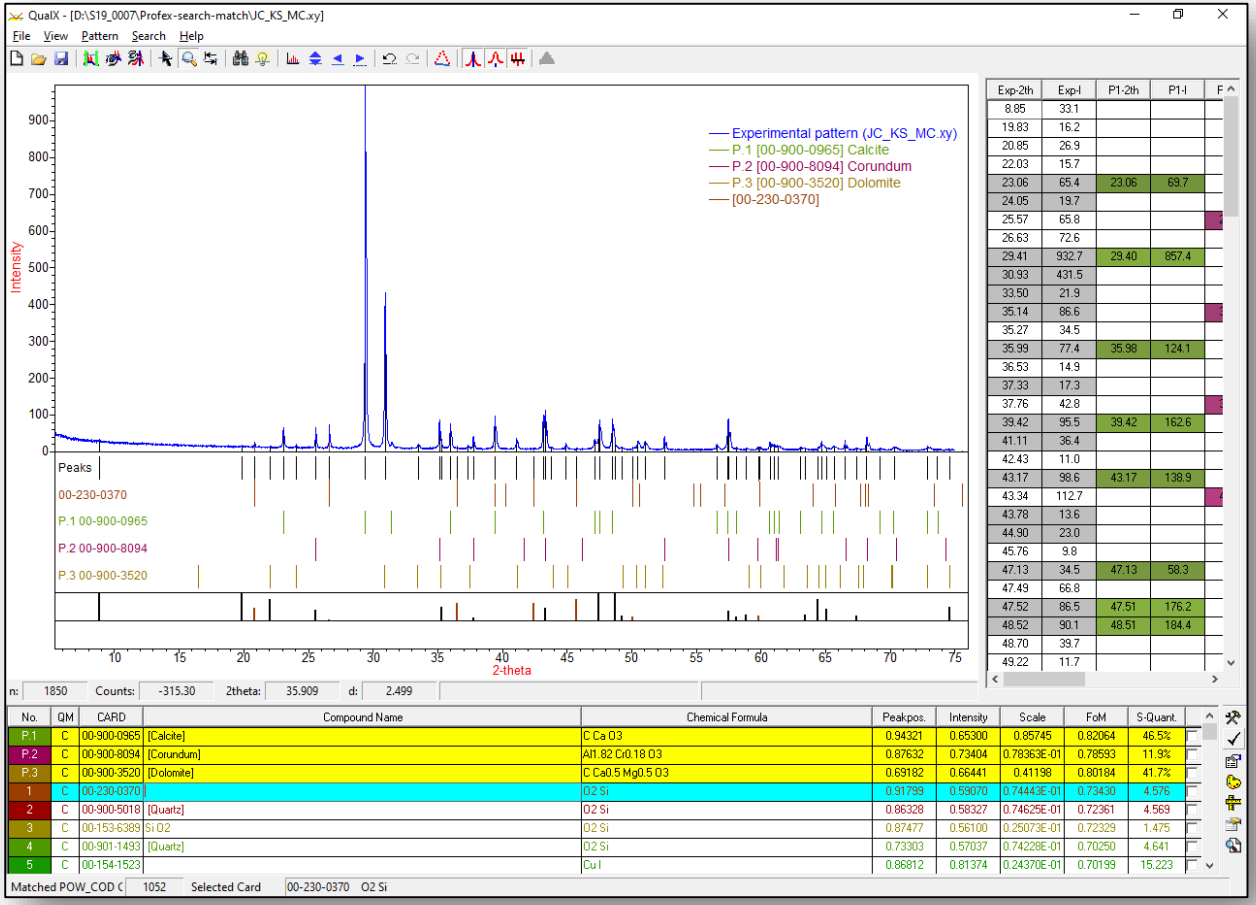


Select format depending on which type of data was exported from Profex.

Import Peak List in QualX2



Import Peak List in QualX2

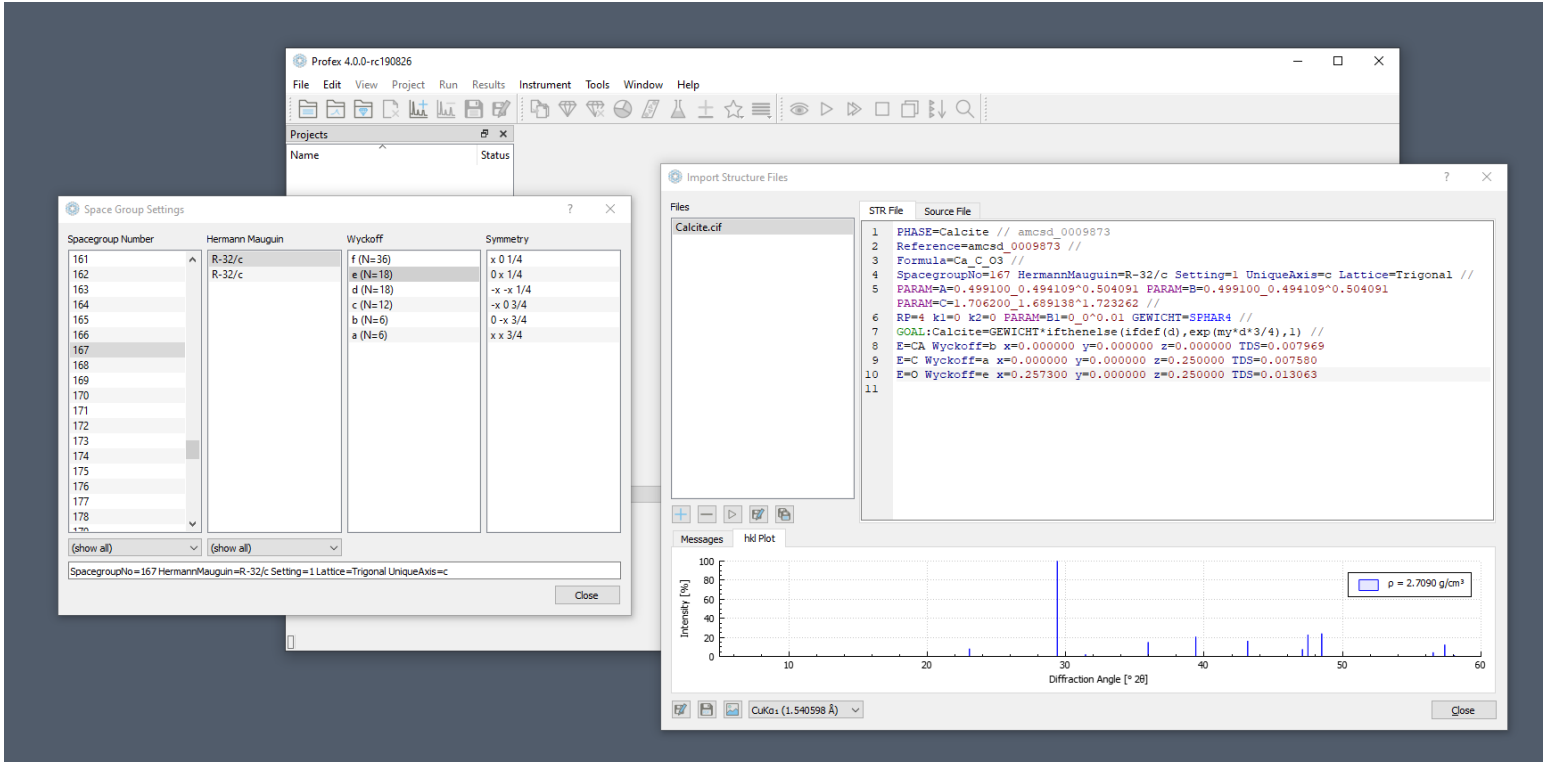


Exp-2th	Exp-I	P1-2th	P1-I
8.85	33.1		
19.83	16.2		
20.85	26.9		
22.03	15.7		
23.06	65.4	23.06	69.7
24.05	19.7		
25.57	65.8		
26.63	72.6		
29.41	932.7	29.40	857.4
30.93	431.5		
33.50	21.9		
35.14	86.6		
35.27	34.5		
35.99	77.4	35.98	124.1
36.53	14.9		
37.33	17.3		
37.76	42.8		
39.42	95.5	39.42	162.6
41.11	36.4		
42.43	11.0		
43.17	98.6	43.17	138.9
43.34	112.7		
43.78	13.6		
44.90	23.0		
45.76	9.8		
47.13	34.5	47.13	58.3
47.49	66.8		
47.52	86.5	47.51	176.2
48.52	90.1	48.51	184.4
48.70	39.7		
49.22	11.7		

No.	QM	CARD	Compound Name	Chemical Formula	Peakpos.	Intensity	Scale	FoM	S-Quant.
P.1	C	00-900-0965	[Calcite]	C Ca O3	0.94321	0.65300	0.85745	0.82064	46.5%
P.2	C	00-900-8094	[Corundum]	Al1.82 Cr0.18 O3	0.87632	0.73404	0.78363E-01	0.78593	11.9%
P.3	C	00-900-3520	[Dolomite]	C Ca0.5 Mg0.5 O3	0.69182	0.66441	0.41198	0.80184	41.7%
1	C	00-230-0370		O2 Si	0.91799	0.59070	0.74443E-01	0.73430	4.576
2	C	00-900-5018	[Quartz]	O2 Si	0.86328	0.58327	0.74625E-01	0.72361	4.569
3	C	00-153-6389	Si O2	O2 Si	0.87477	0.56100	0.25073E-01	0.72329	1.475
4	C	00-901-1493	[Quartz]	O2 Si	0.73303	0.57037	0.74228E-01	0.70250	4.641
5	C	00-154-1523		Cu I	0.86812	0.81374	0.24370E-01	0.70199	15.223

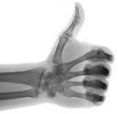
Creating STR Files

After lunch:
How to convert structure files from databases to BGMN *.str format



Peak Detection

Summary:



- Peak detection using EFLECH (part of BGMN)
- Very accurate due to correct wavelength spectrum and instrument configuration
- Export to 3rd party software (tested with Match! And QualX)

- Can be VERY slow (sometimes unable to complete)

Peak List

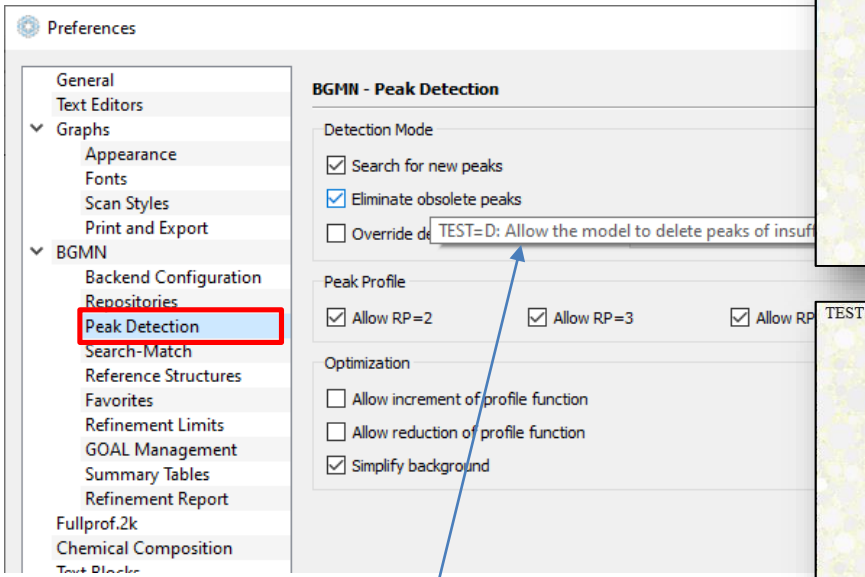
hkl list: JC_KS_MC 0 peak data

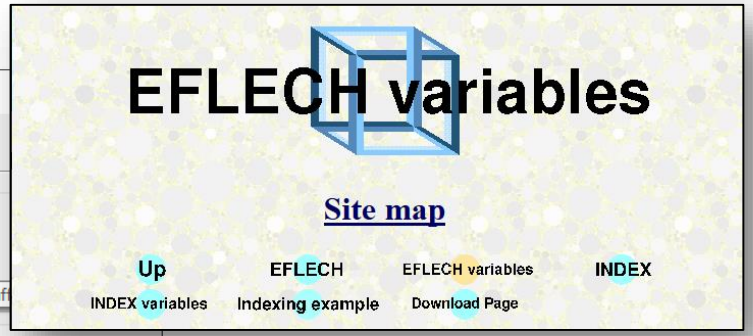
	2theta [°]	d [nm]	Intensity [a.u.]
1	19.8312	0.4473	71.9460
2	20.8484	0.4257	119.9208
3	22.0285	0.4032	31.5712
4	23.0642	0.3853	749.8248
5	24.0512	0.3697	89.5717
6	25.5695	0.3481	727.3246
7	26.6299	0.3345	678.5722
8	29.4120	0.3034	12898.0000
9	30.9293	0.2889	6356.1505
10	33.5049	0.2672	231.2011
11	35.1395	0.2552	1093.2758
12	35.2735	0.2542	141.1452
13	35.9862	0.2494	1211.9070
14	36.5304	0.2458	35.0244

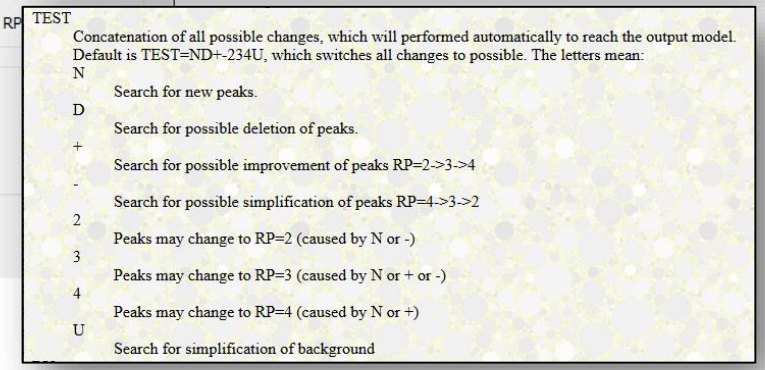
Peak Detection

Tuning peak detection parameters
Edit → Preferences…

http://www.bgmn.de/eflech_variables.html







Hover mouse to show tool tip

New Features in Profex 4

- GUI changes
- Search / Match
- Peak detection
- Interaction with QualX / Match!

Tomorrow:

- Electron-density maps
- Drawing crystal structures with Vesta / Mercury
- Customizing report layouts