RMS · · · · · · · Testing • Research • Consulting

Workshop „Rietveld Refinement with Profex“

Lesson 5: Introduction to Profex - Rietveld Refinement

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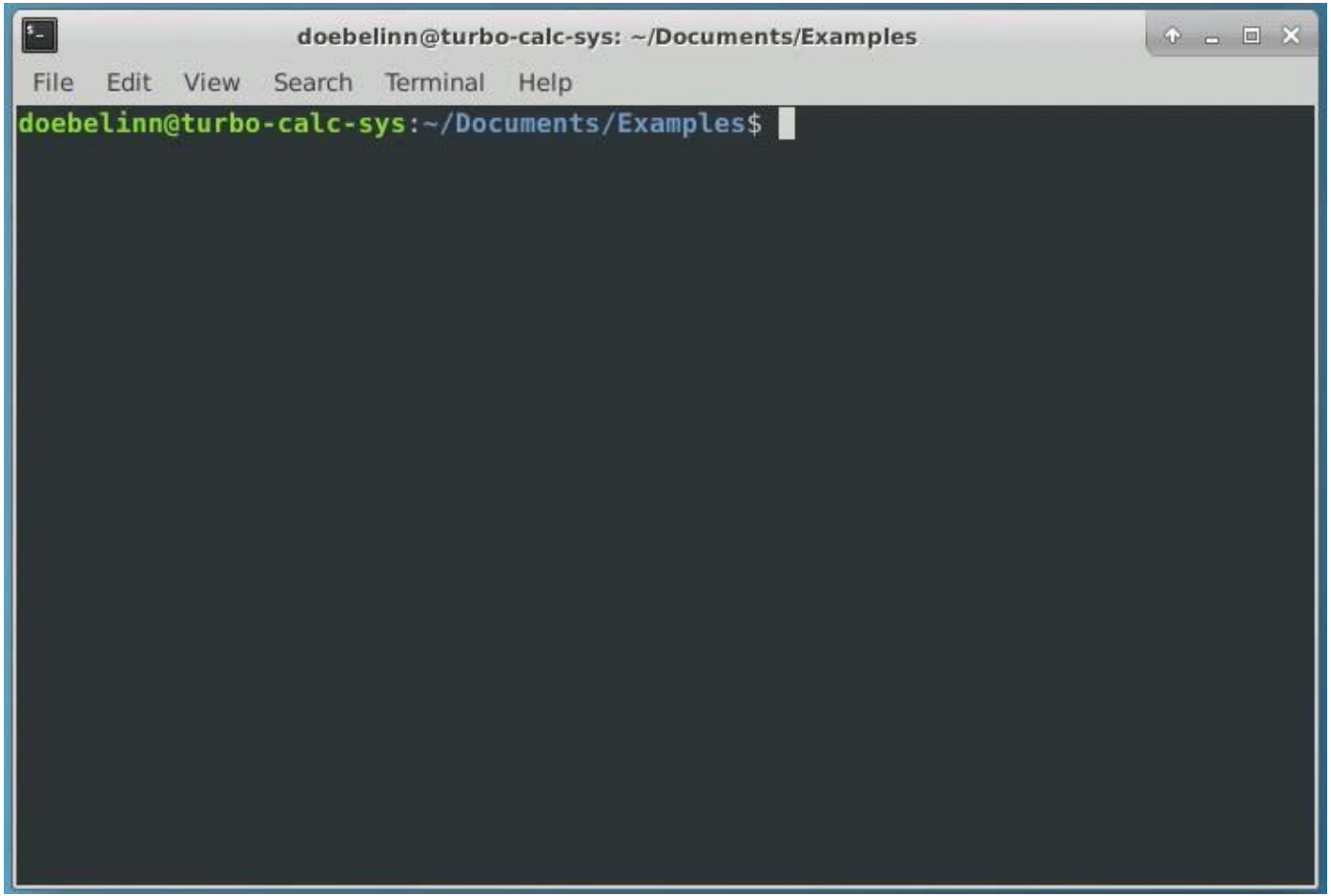
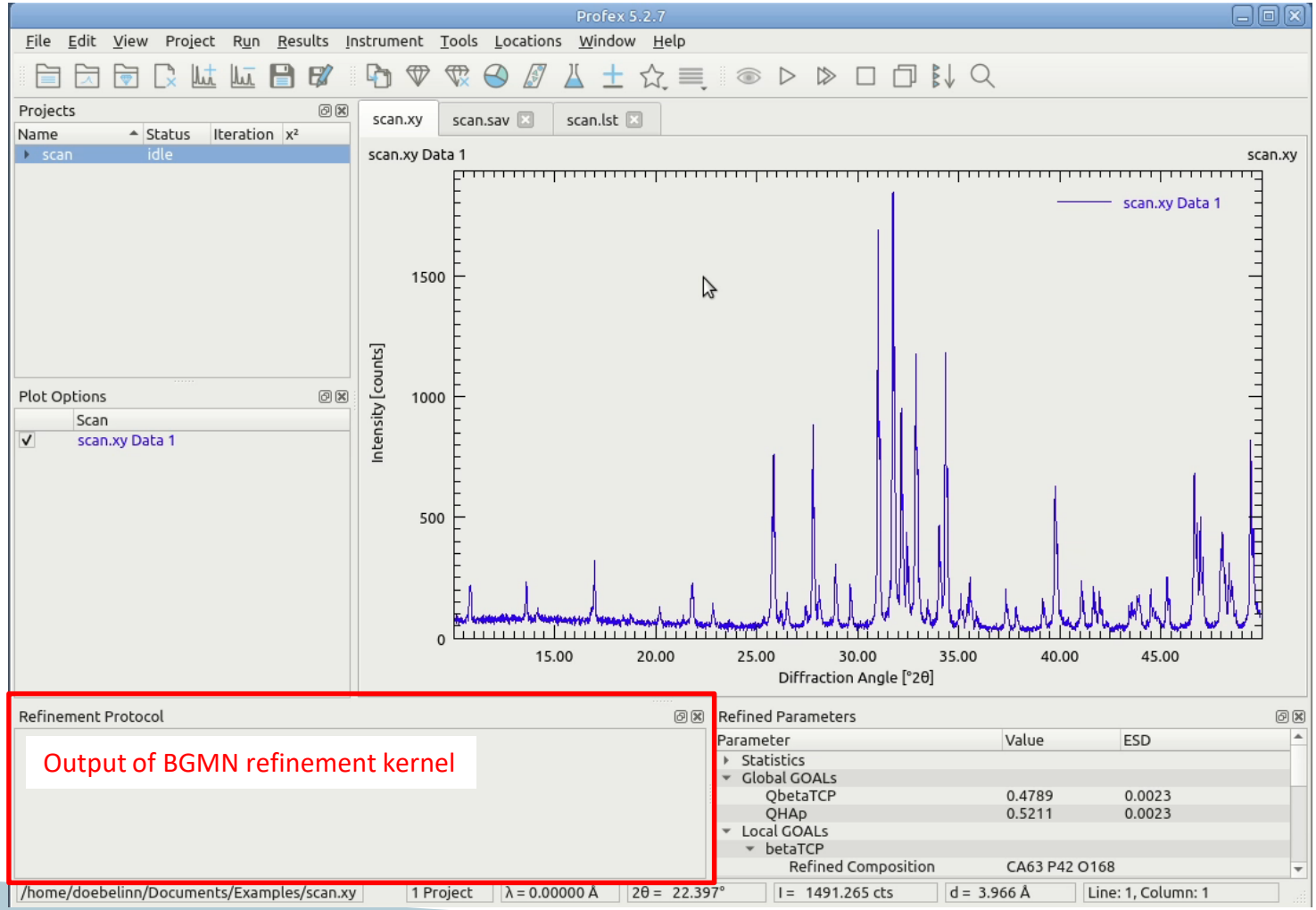
Forschungszentrum Jülich, Germany

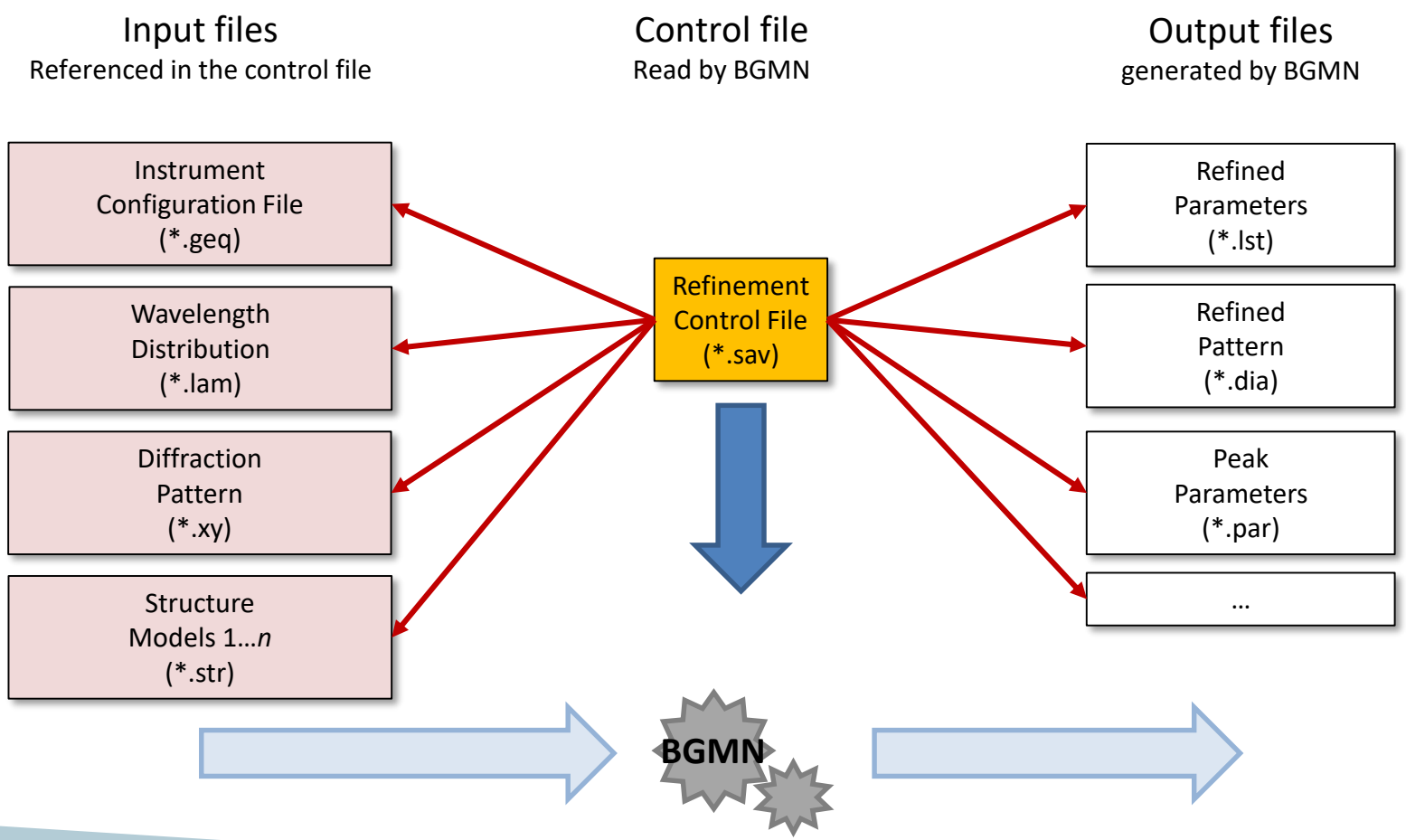
BGMN: Rietveld Refinement Kernel



Profex: Graphical User Interface for BGMN



BGMN Rietveld Refinement Kernel



BGMN Input and Output Files

Input files

File	Extension	Content
Refinement control file	*.sav	Project configuration
Instrument configuration file	*.geq	Peak profile: Instrument contribution
Wavelength distribution	*.lam	Peak profile: Wavelength contribution
Diffraction pattern	*.xy	Measured diffraction data
Structure models	*.str	Unit cell, atomic positions etc.

Output files

File	Extension	Content
Refined parameters	*.lst	Refinement results
Refined pattern	*.dia	Iobs, Icalc, background etc. for graphical representation
Peak parameters	*.par	Position, intensity, width of refined peaks

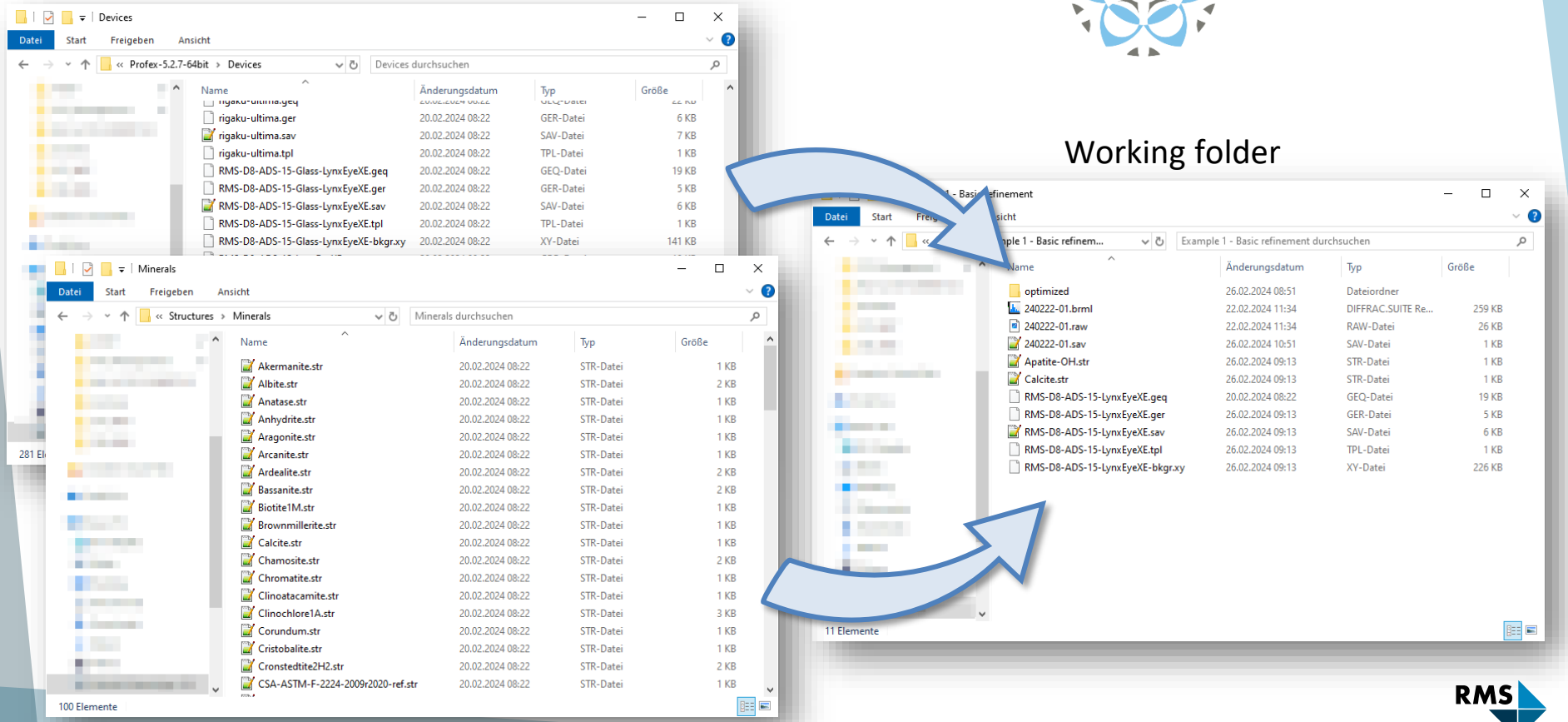
Profex Repositories

Profex repositories

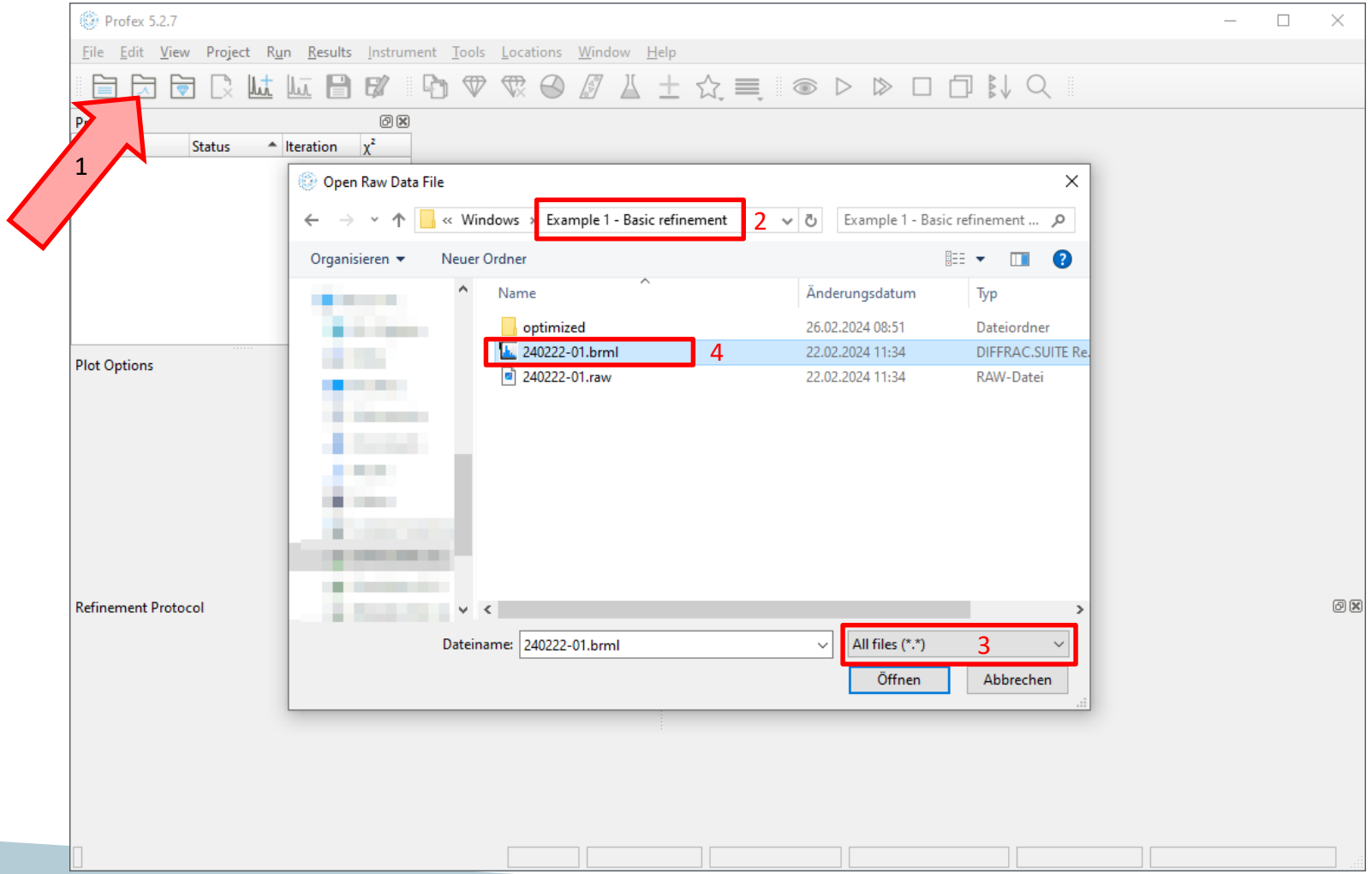
Collections of device and structure files

Profex manages file templates

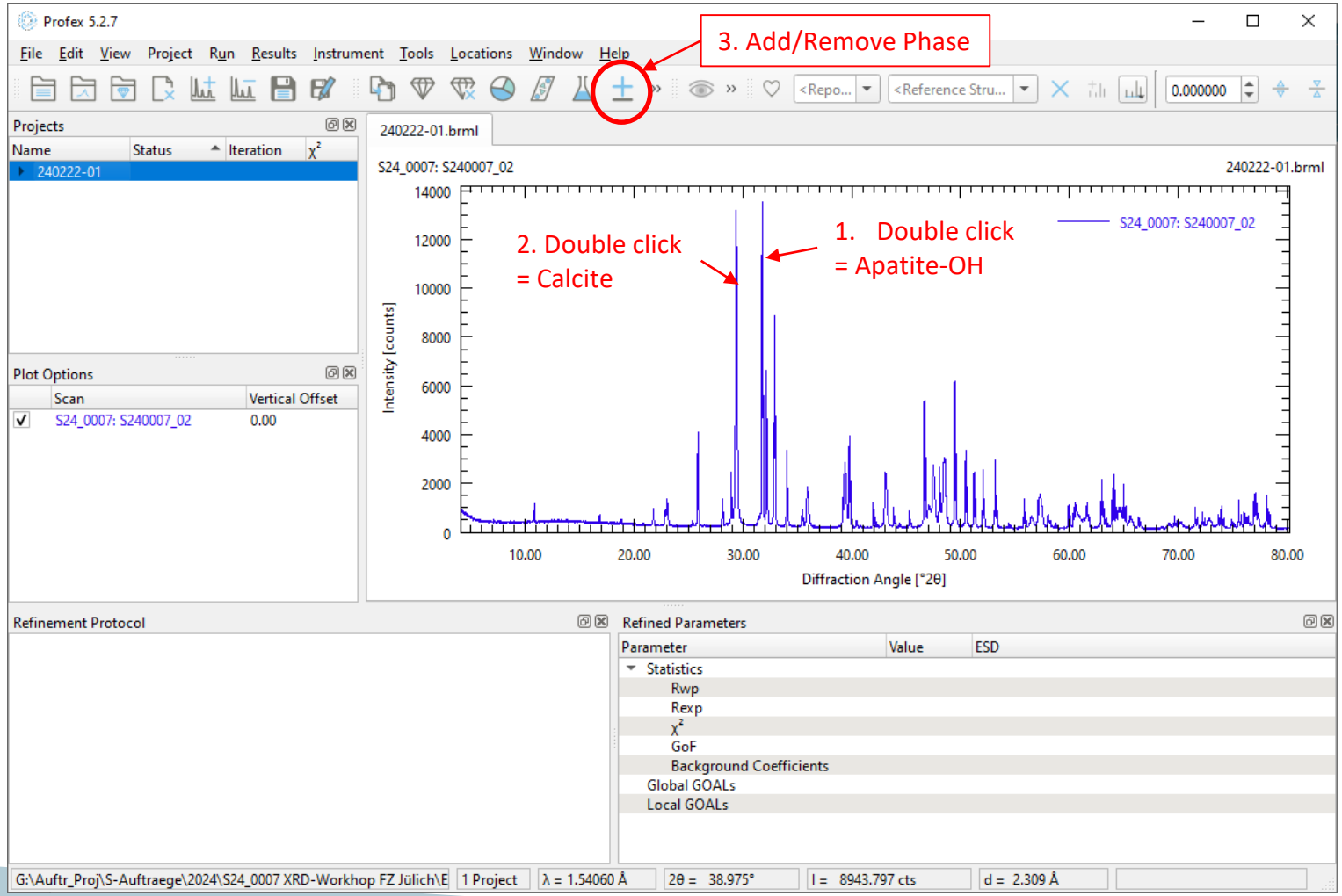
Working folder



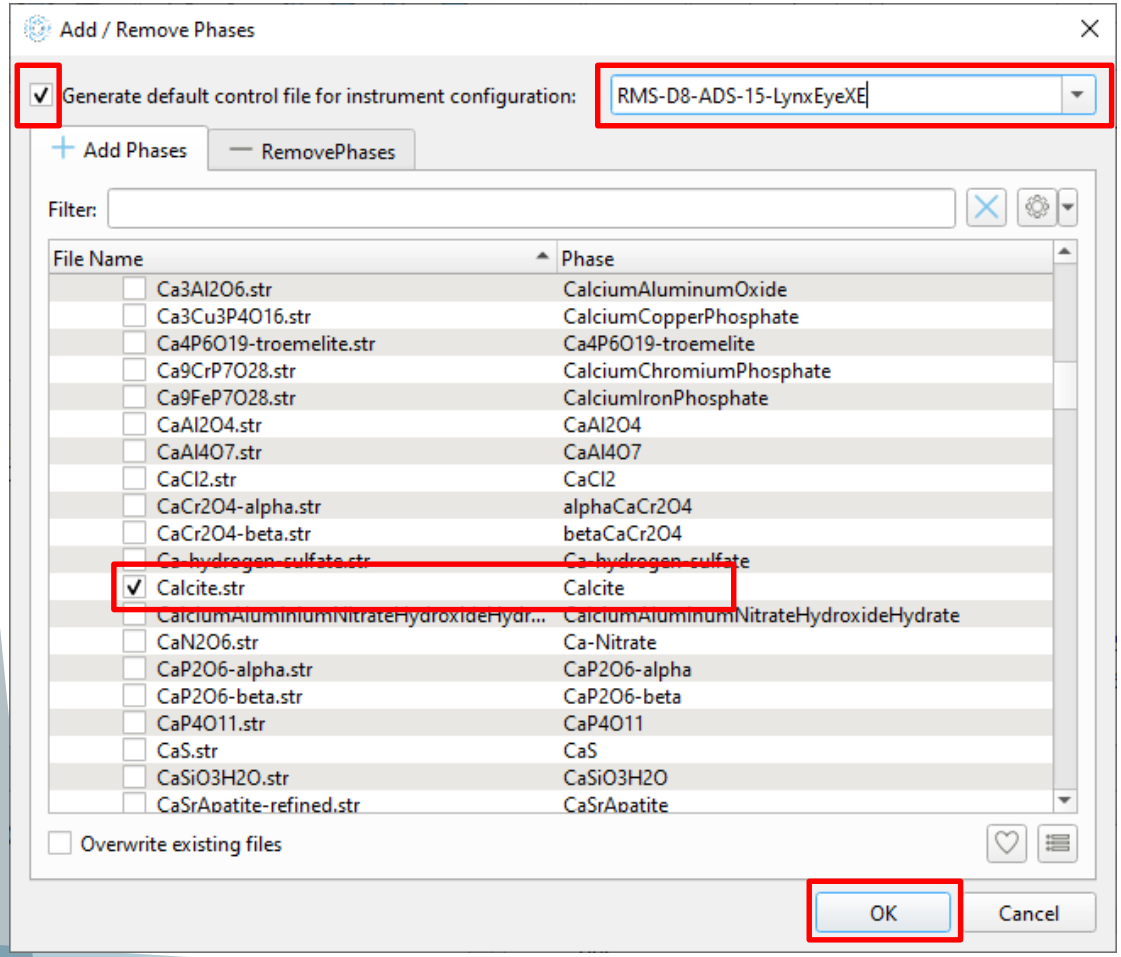
First Rietveld Refinement: Loading the Scan File



First Rietveld Refinement: Identifying Phases



First Rietveld Refinement: Creating Refinement Project



1. Generate refinement control file for the instrument used to measure the dataset.

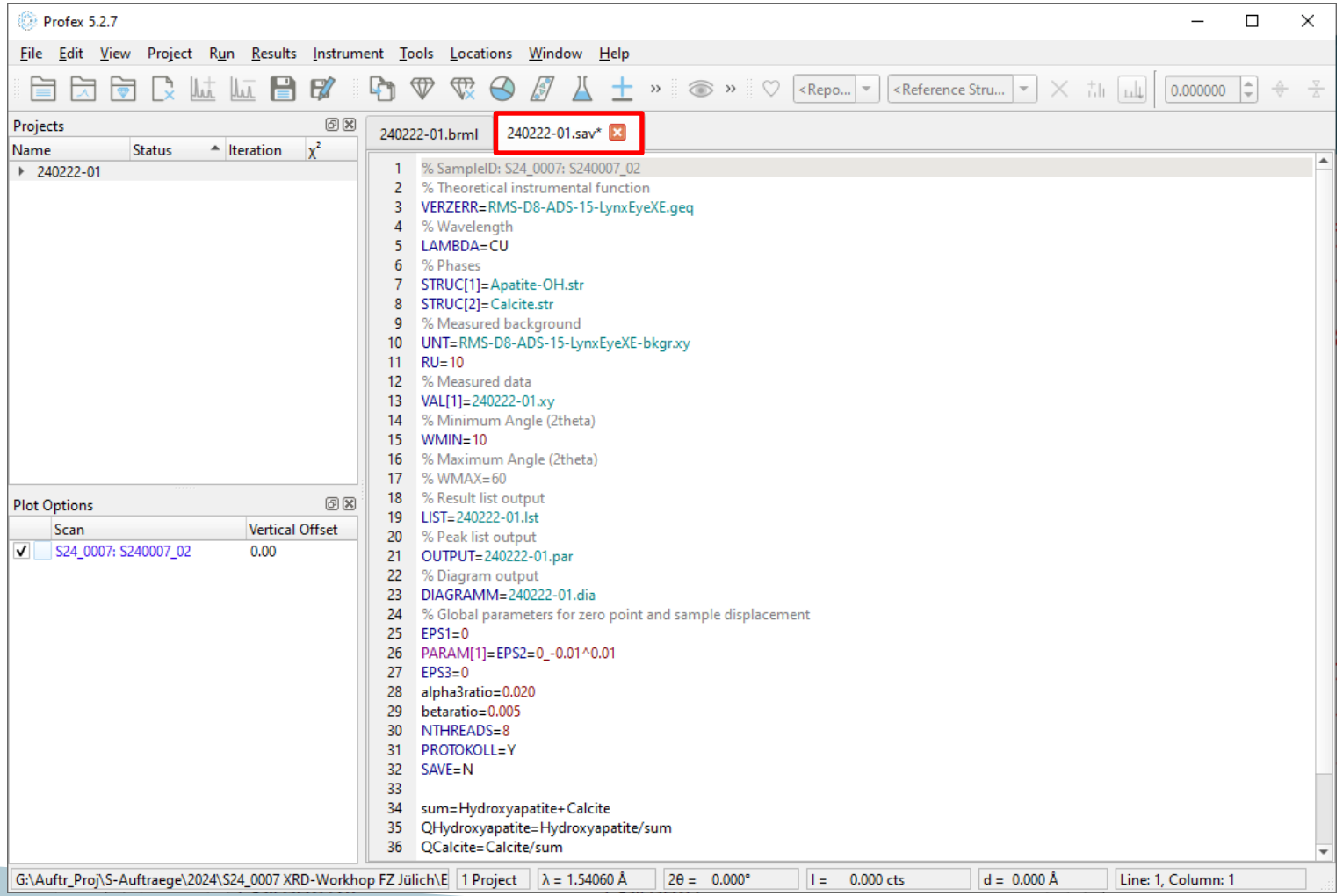
Here: „RMS-D8-ADS-15-LynxEyeXE“

2. Select the phases „Calcite.str“ and „Apatite-OH.str“.

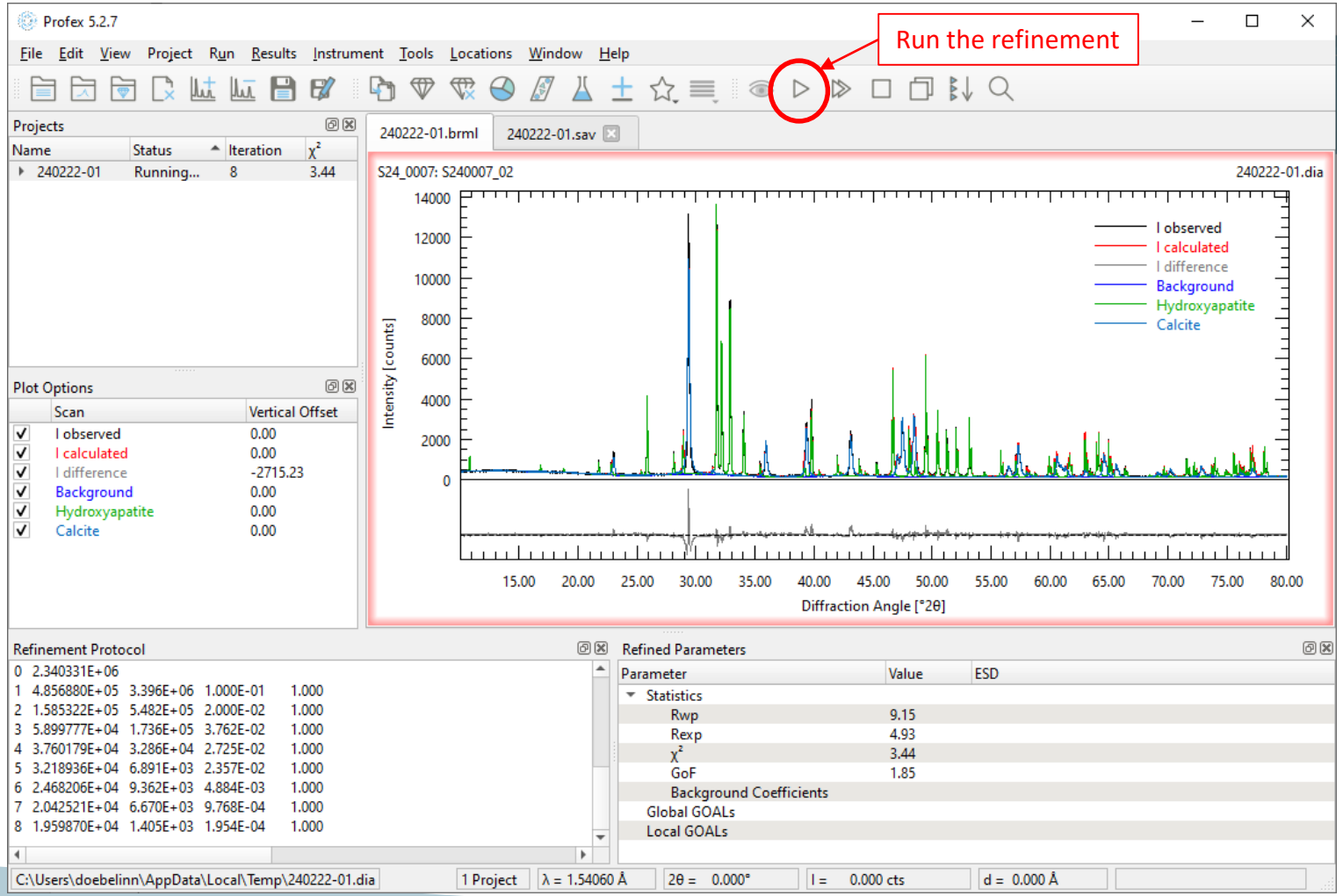
(Use filters to find the phases.)

3. Click OK

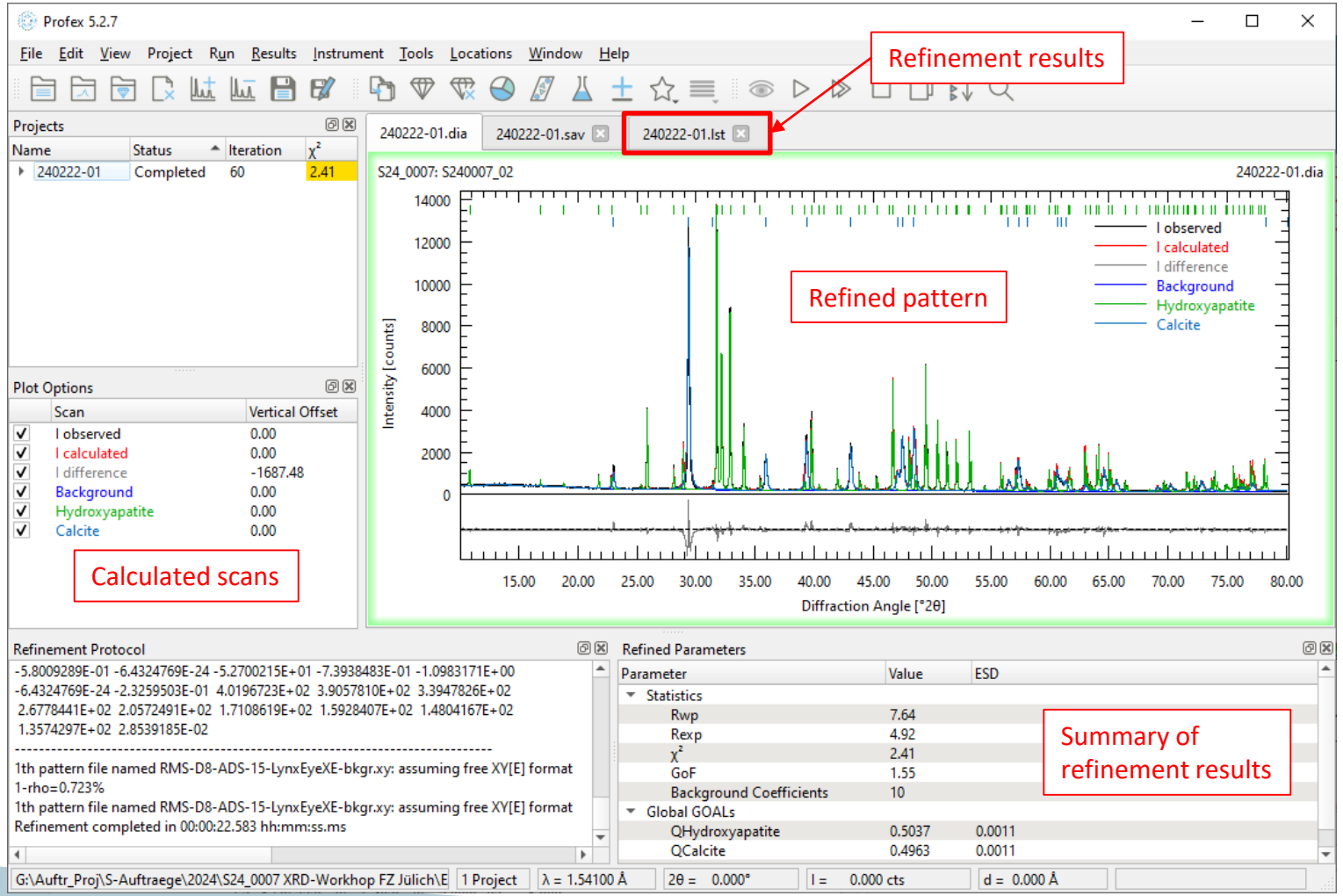
First Rietveld Refinement: Refinement Control File (*.sav)



First Rietveld Refinement: Run Refinement



First Rietveld Refinement: Completed



Refinement Control Files (*.sav)

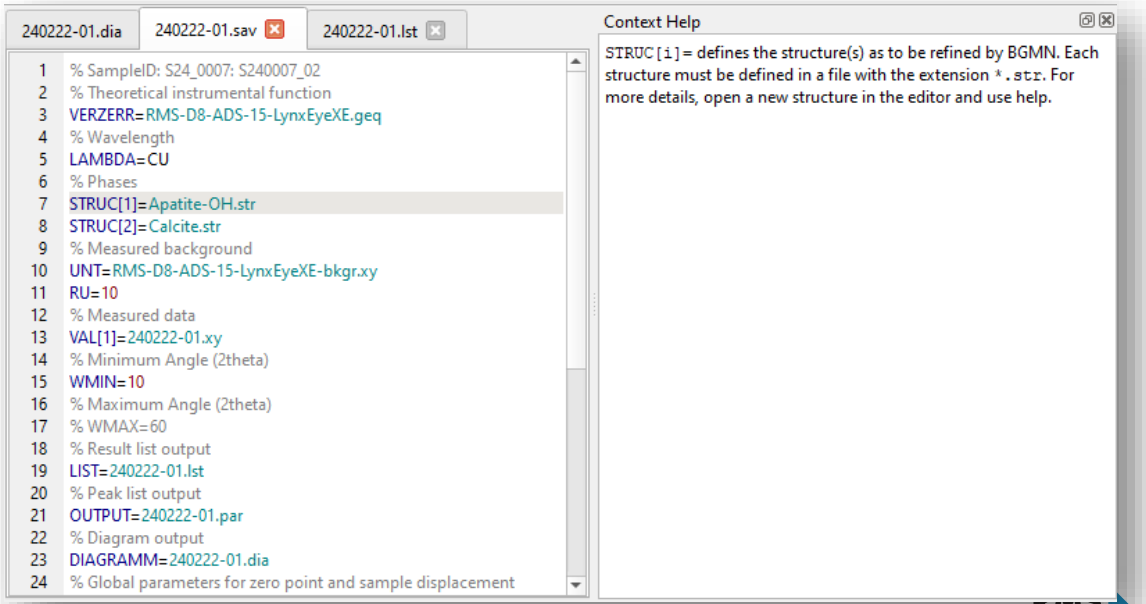
```
% SampleID: S24_0007: S240007_02
% Theoretical instrumental function
VERZERR=RMS-D8-ADS-15-LynxEyeXE.geq
% Wavelength
LAMBDA=CU
% Phases
STRUC[1]=Apatite-OH.str
STRUC[2]=Calcite.str
% Measured background
UNT=RMS-D8-ADS-15-LynxEyeXE-bkgr.xy
RU=10
% Measured data
VAL[1]=240222-01.xy
% Minimum Angle (2theta)
WMIN=10
% Maximum Angle (2theta)
% WMAX=60
% Result list output
LIST=240222-01.lst
% Peak list output
OUTPUT=240222-01.par
% Diagram output
DIAGRAMM=240222-01.dia
% Global parameters for zero point and
% sample displacement
EPS1=0
PARAM[1]=EPS2=0_-0.01^0.01
EPS3=0
alpha3ratio=0.020
betaratio=0.005
NTHREADS=8
PROTOKOLL=Y
SAVE=N

sum=Hydroxyapatite+Calcite
QHydroxyapatite=Hydroxyapatite/sum
QCalcite=Calcite/sum

GOAL[1]=QHydroxyapatite
GOAL[2]=QCalcite
```

% = comments (will be ignored by BGMN)
BLUE = variable name
Green = input / output file name
Red = numerical value

Window → Context help displays a description of variables.



Refinement Control Files (*.sav)

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sum=Hydroxyapatite+Calcite
QHydroxyapatite=Hydroxyapatite/sum
QCalcite=Calcite/sum

GOAL[1]=QHydroxyapatite
GOAL[2]=QCalcite
```

Reference to instrument configuration file

Reference to wavelength distribution file

Reference to structure files (must be numbered)

Reference to measured background scan

Reference to measured diffraction pattern
(Profex will convert all formats to xy format)

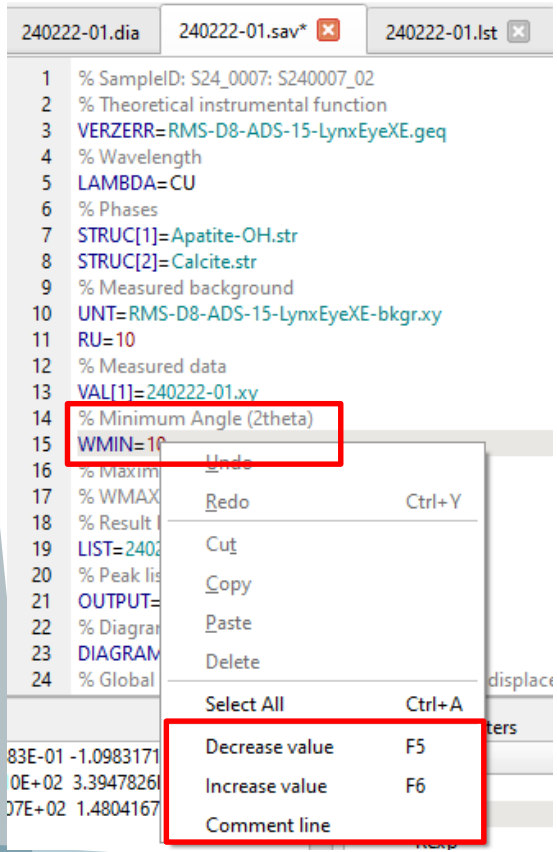
Various output file names

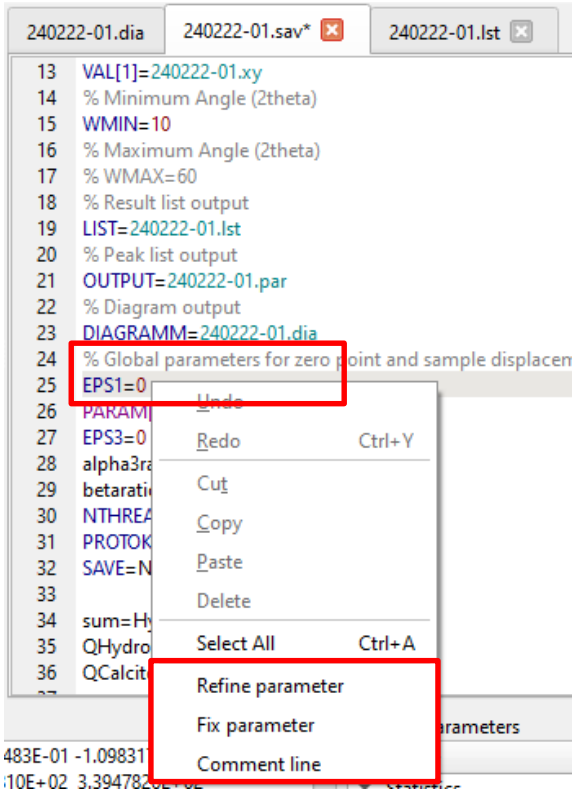
Refinement of sample height error

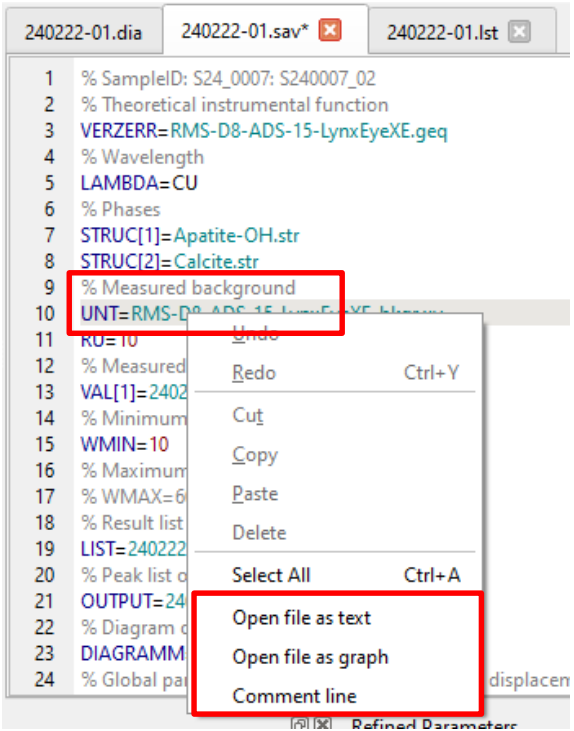
Calculation of phase quantities

Refinement Control Files (*.sav)

Context menu (right mouse button) for many parameters:

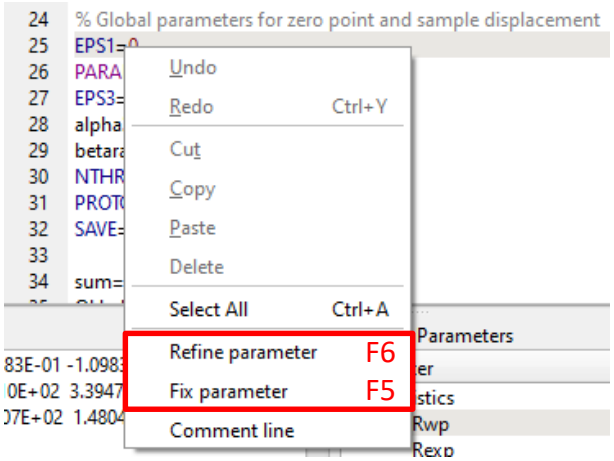






Refinement Control Files (*.sav)

```
% Global parameters for zero point and
% sample displacement
EPS1=0
PARAM[1]=EPS2=0_-0.01^0.01
EPS3=0
```



How to refine parameters

Parameter fixed at 0 (not refined):

```
EPS1=0
```

Parameter refined:

```
PARAM[n]=EPS1=0
```

Parameter refined with limits:

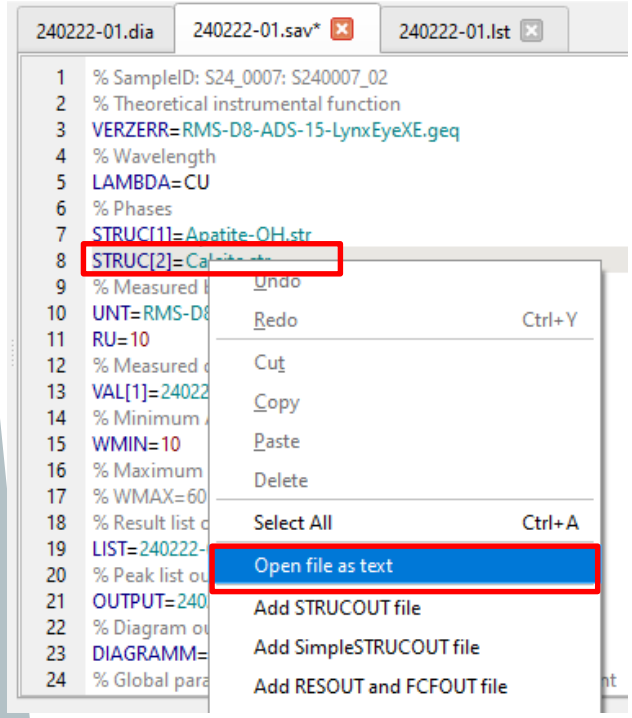
```
PARAM[n]=EPS1=0_-0.01^0.01
```

`PARAM[n]= name= initial value _lower limit ^upper limit`

`n` = consecutive number

Structure Files (*.str)

Opening structure files



or Ctrl+T on STRUC[n] line

```
PHASE=Calcite // 04-008-0788
MineralName=Calcite //
Formula=Ca_(CO3) //
SpacegroupNo=167 HermannMauguin=R-32/c //
PARAM=A=0.4991_0.4941^0.5041 PARAM=C=1.7062_1.6891^1.7233 //
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHAR4 //
GOAL=GrainSize(1,1,1) //
GOAL:Calcite=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1)
E=CA Wyckoff=b x=0.0000 y=0.0000 z=0.0000 TDS=0.00796938
E=C Wyckoff=a x=0.0000 y=0.0000 z=0.2500 TDS=0.00757986
E=O Wyckoff=e x=0.2573 y=0.0000 z=0.2500 TDS=0.01400168
```

//	= comments (will be ignored by BGMN) (trailing // are optional / used for historic reasons)
BLUE	= variable name
Light blue	= input / output file name
Red	= numerical value
GREEN	= GOAL declaration

Refined parameters in STR files are **not numbered**:

```
PARAM=name=value_lowerLimit^upperLimit
```

Structure Files (*.str)

```
PHASE=Calcite // 04-008-0788

MineralName=Calcite
Formula=Ca_(CO3)

SpacegroupNo=167 HermannMauguin=R-32/c

PARAM=A=0.4991_0.4941^0.5041 PARAM=C=1.7062_1.6891^1.7233

RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01

GEWICHT=SPHAR4

GOAL=GrainSize(1,1,1)

GOAL:Calcite=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1)

E=CA Wyckoff=b x=0.0000 y=0.0000 z=0.0000 TDS=0.00796938
E=C Wyckoff=a x=0.0000 y=0.0000 z=0.2500 TDS=0.00757986
E=O Wyckoff=e x=0.2573 y=0.0000 z=0.2500 TDS=0.01400168
```

Phase name (don‘t use spaces or special characters)
Database record (used by Profex)

Informative (not used by Profex or BGMN)

Space group number and HM symbol

Cell parameters (A, B, C, ALPHA, BETA, GAMMA)

Profile parameters (peak broadening)

Scale factor and texture refinement

Calculate crystallite size

Brindley correction for micro-absorption and assign corrected scale factor GEWICHT to variable „Calcite“

Atomic sites (TDS = thermal displacement parameter)

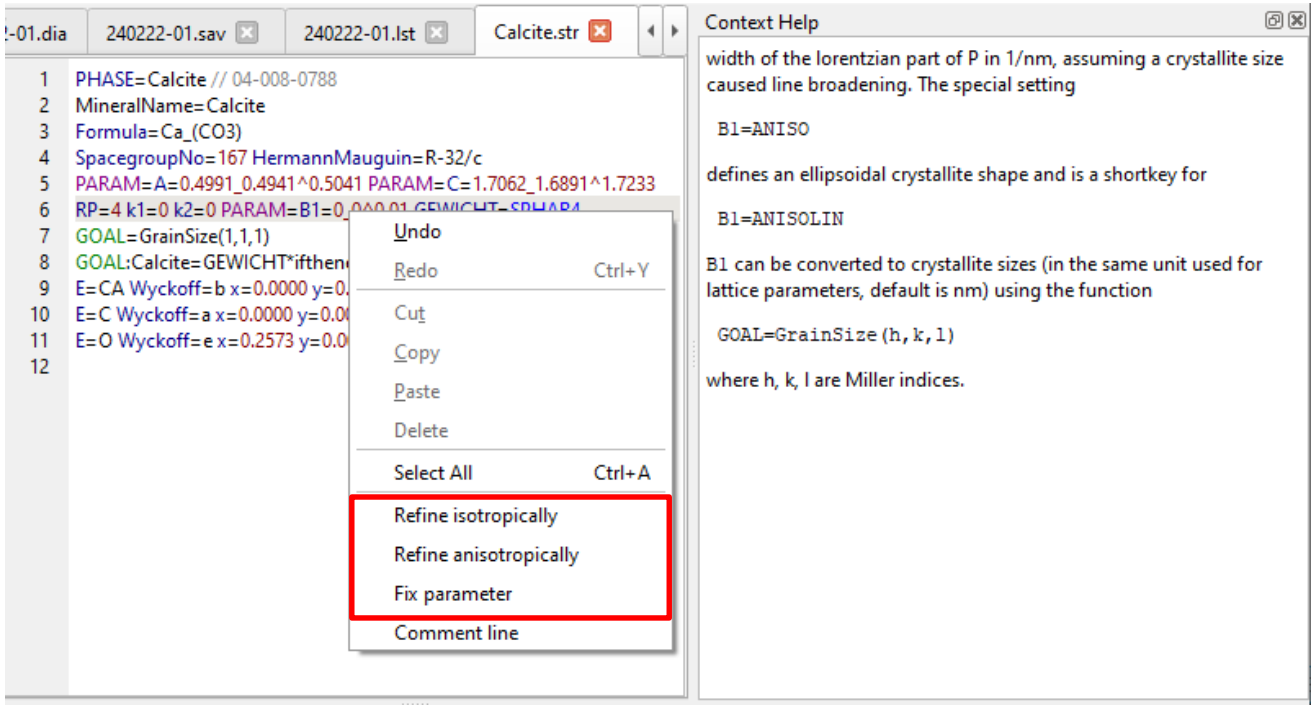
Structure Files (*.str)

```
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHAR4
```

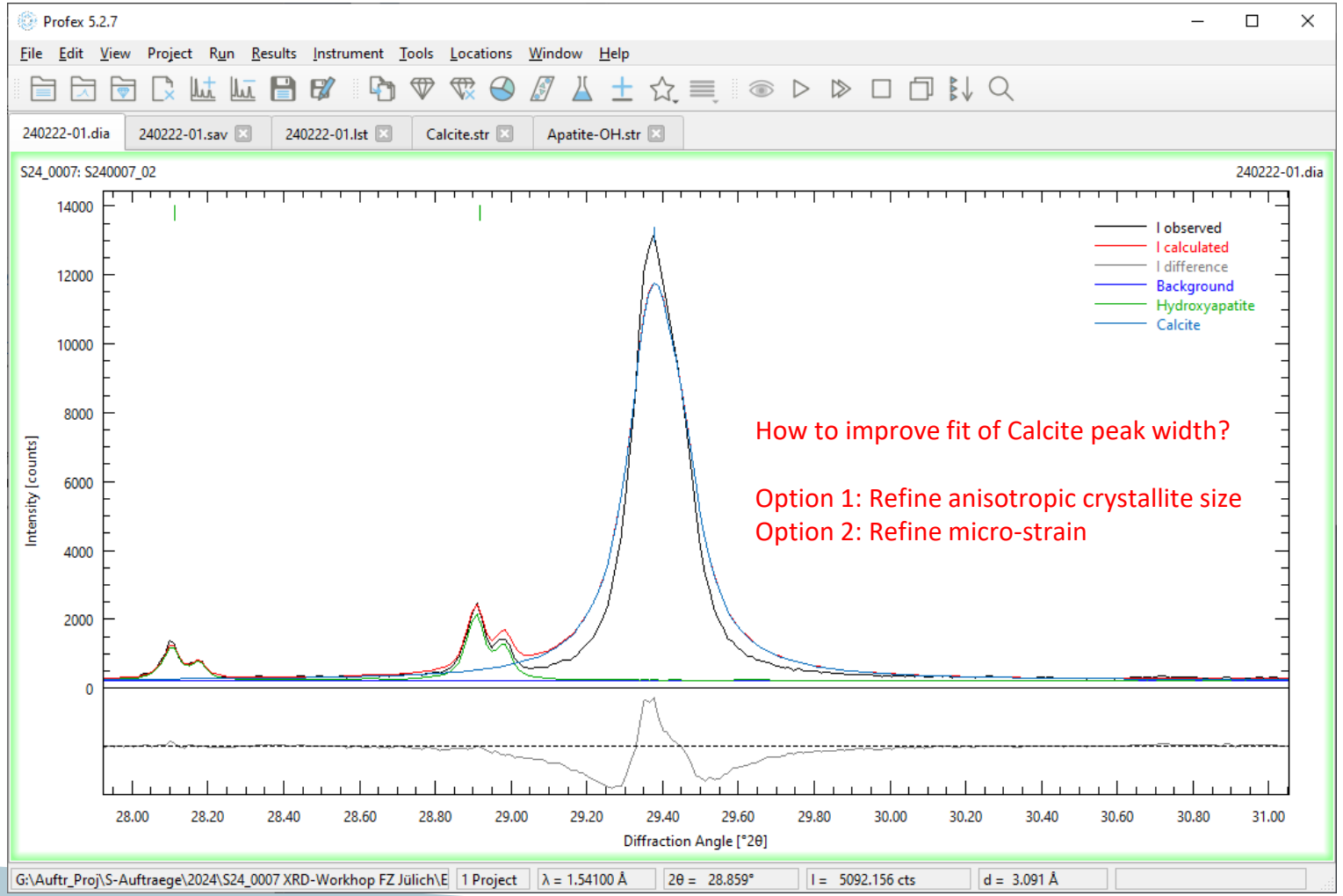
RP=4	Profile function: RP=2: no peak broadening RP=3: only crystallite size peak broadening RP=4: crystallite size + micro-strain peak broadening
k1=0	Crystallite size distribution
k2=0	Micro-strain related peak broadening (can be anisotropic)
PARAM=B1=0_0^0.01	Crystallite size related peak broadening (can be anisotropic)
GEWICHT=SPHAR4	Scale factor corrected for unit cell density (GEWICHT = S · (Z · M · V)) SPHAR0: no texture SPHAR>0: texture model activated

Structure Files (*.str)

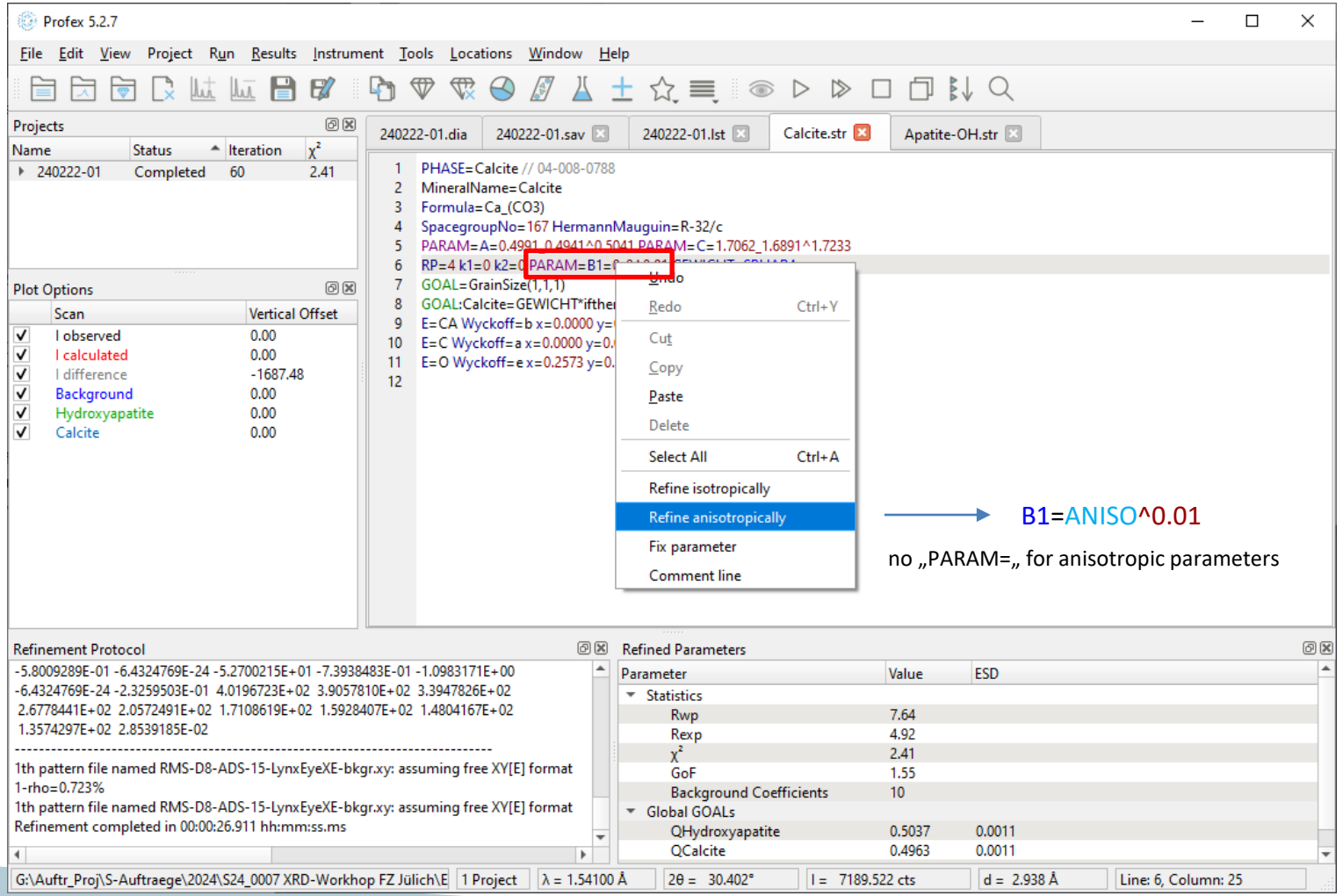
Use the context help (Window → Context Help) for a description of the parameters, and the context menu (right mouse button) to toggle the refinement state:



First Rietveld Refinement: Improving the Refinement



First Rietveld Refinement: Improving the Refinement



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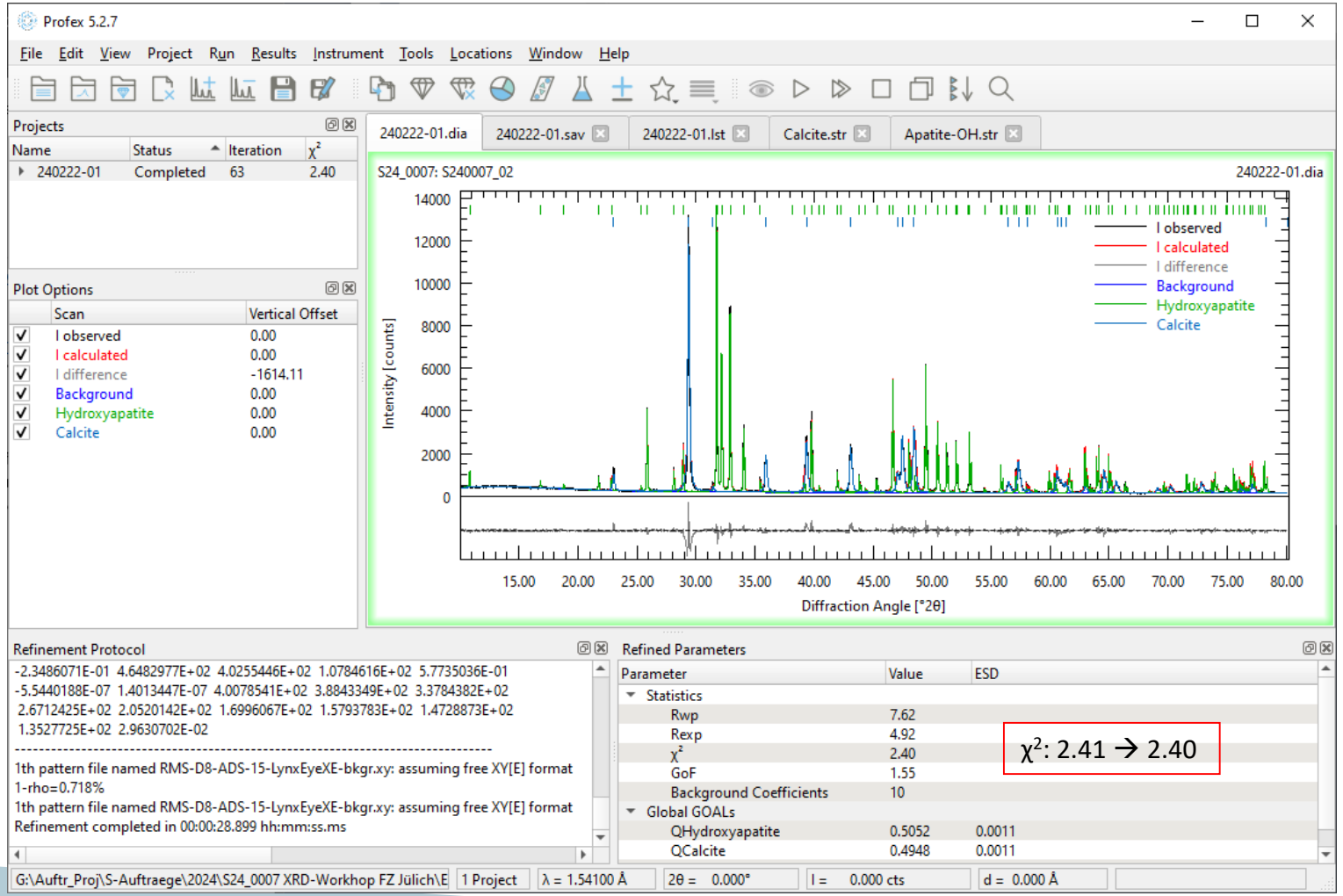
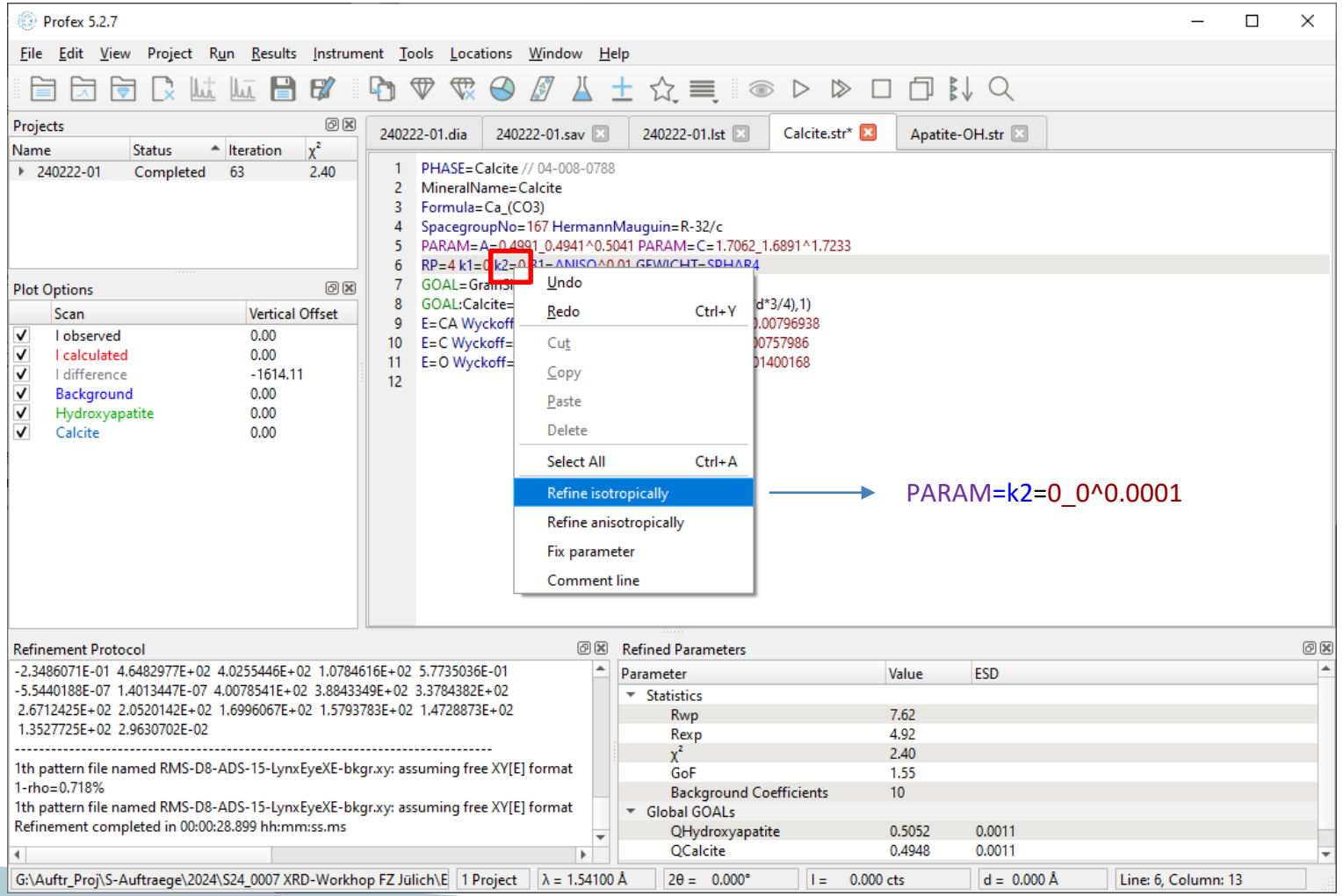
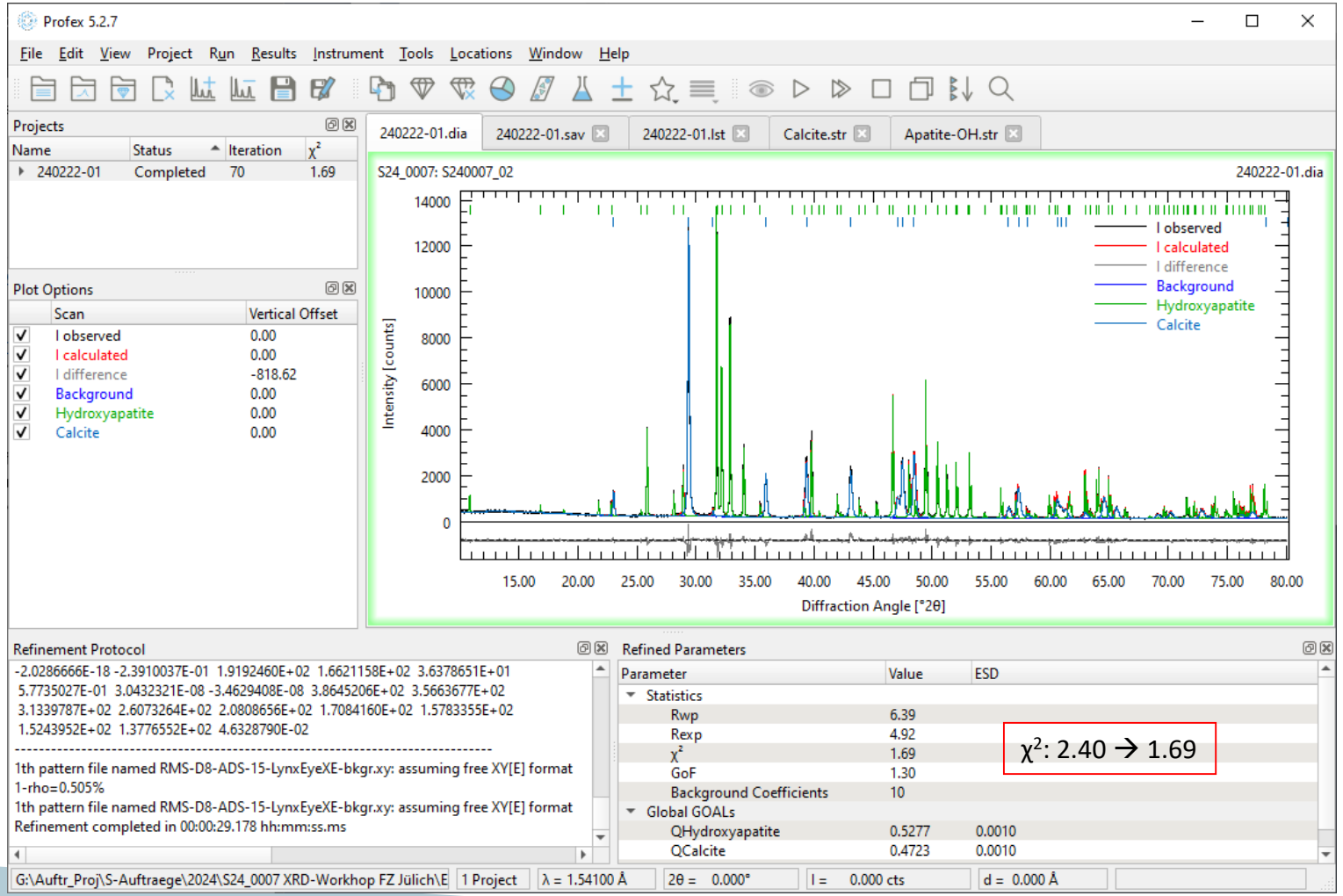


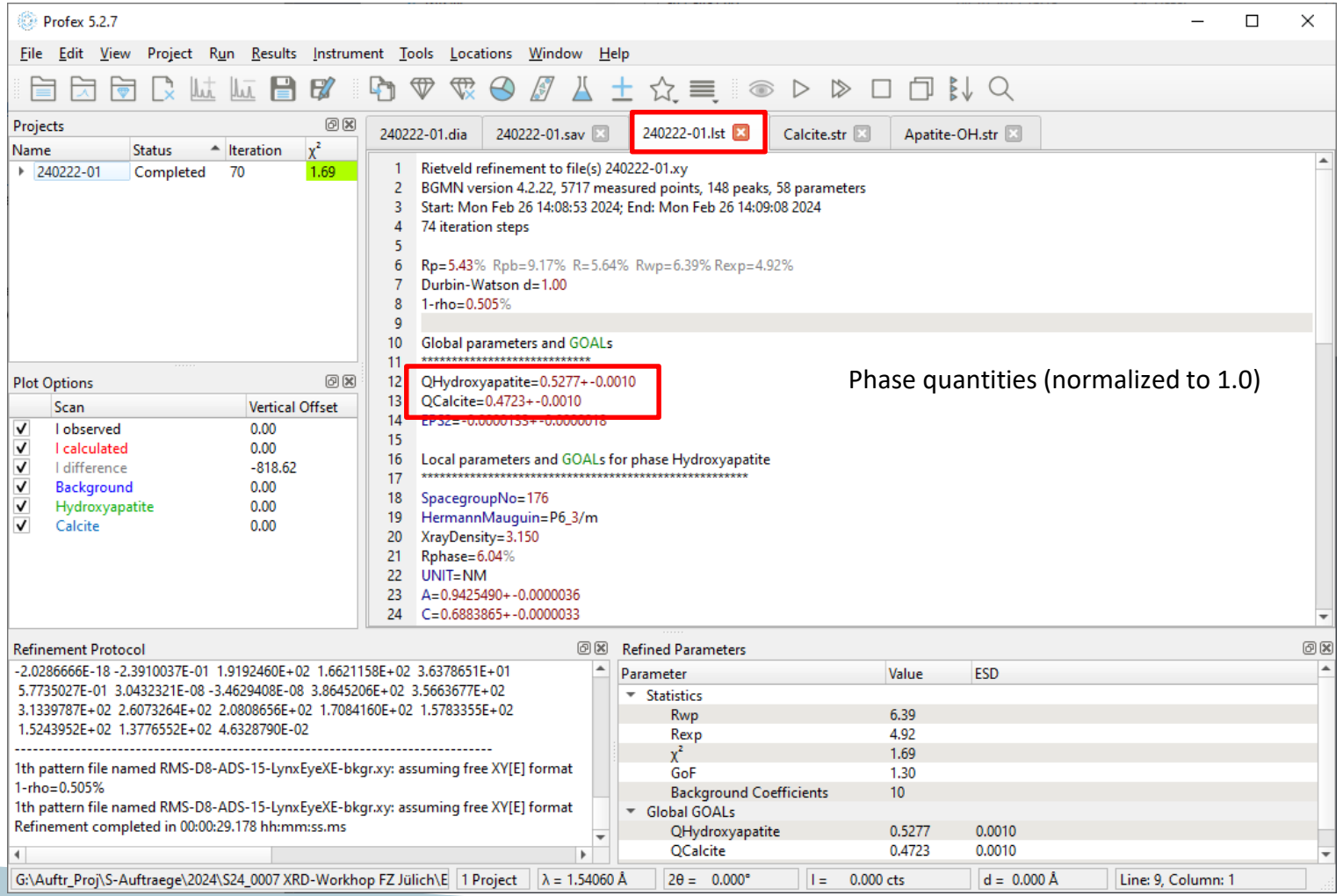
First Rietveld Refinement: Improving the Refinement



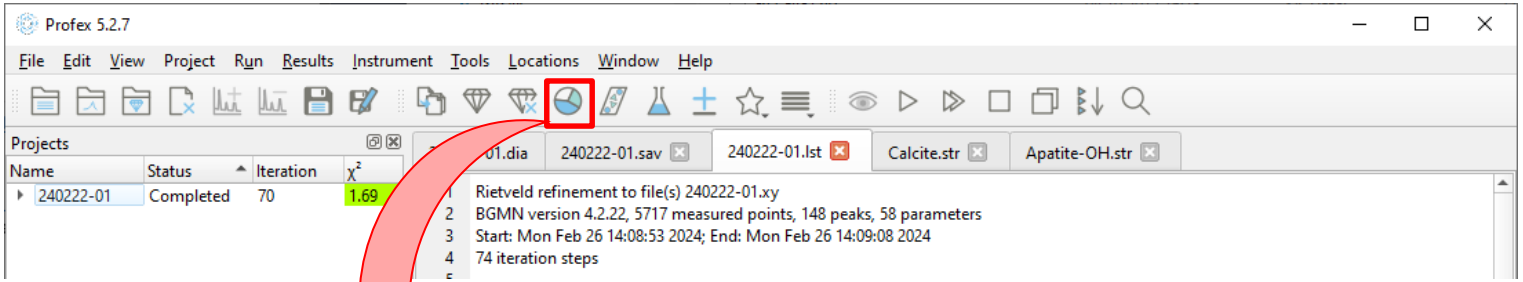
First Rietveld Refinement: Improving the Refinement

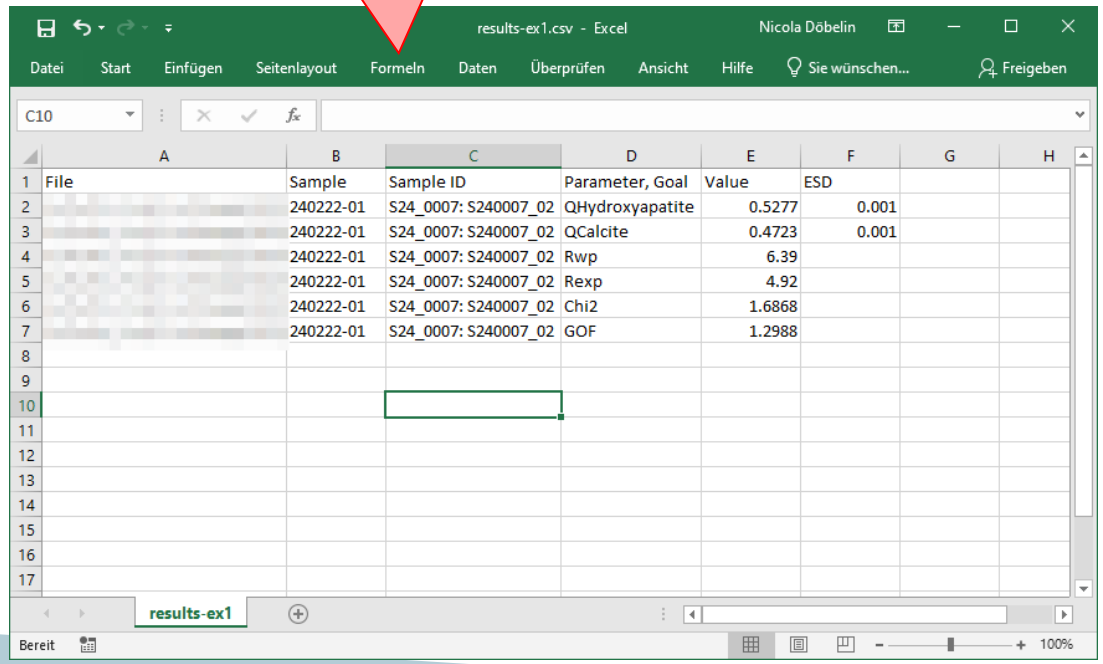


First Rietveld Refinement: Results

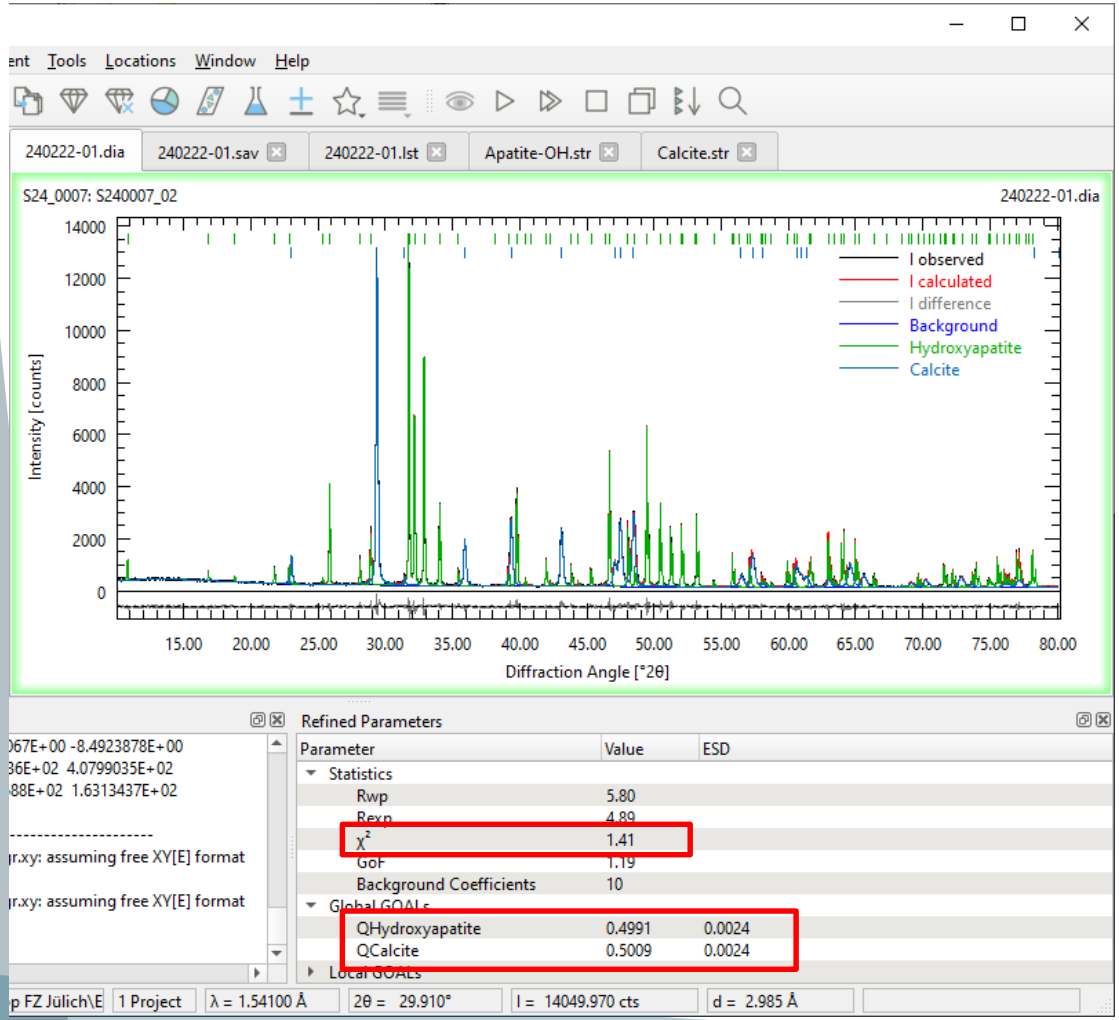


First Rietveld Refinement: Results





First Rietveld Refinement: Outlook



My best fit (with additional tweaks):

- Anisotropic k2 and B1 for both phases
- Texture SPHAR6 for both phases
- Bimodal crystallite size for Calcite
- Substitution Ca → Mg for Calcite
- Substitution Ca → Na for Apatite-OH

The sample is a synthetic mixture of
50.0 wt-% Calcite + 50.0 wt-% Apatite-OH