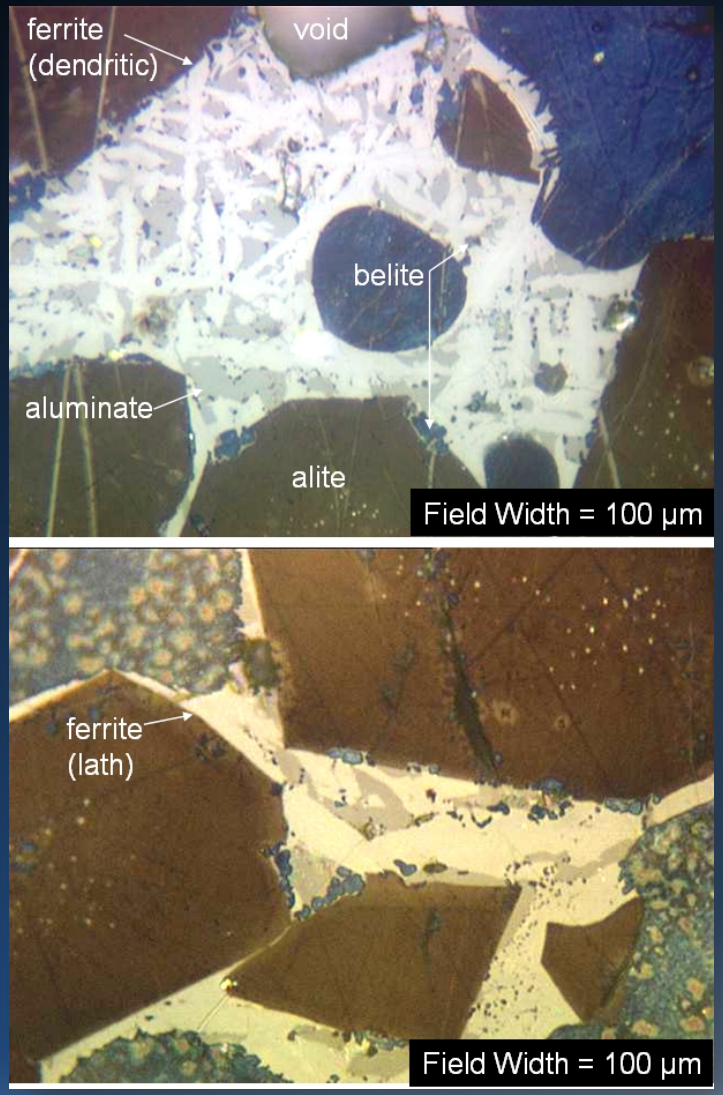
Single Phase Example

Ferrite Phase from SRM2686a

Nitric acid/methanol extraction



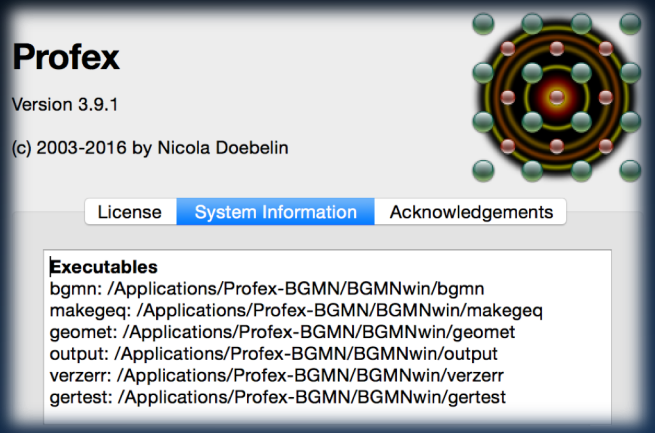
We will be using only a subset of the features provided by Profex/BGMN to run a basic refinement for quantitative analysis; we leave it to you to explore the more advanced refinement features after you have had some experience.

Introduction to the Rietveld Method, R.A. Young

- Provides background on the method, its historical development and subsequent application to laboratory X-ray diffraction
- Refining parameters in models for the structure, specimen and instrument effects on the diffraction pattern
- Least-squares refinements are carried out until the best fit is obtained between the entire observed powder diffraction pattern and calculated pattern based upon simultaneously refined models for crystal structures and instrument effects
- Potential systematic errors in include preferred orientation, background, anisotropic peak broadening, profile shapes, absorption, specimen displacement, specimen transparency, extinction, 2-theta-Zero error, graininess, beam instability

Introduction to the Rietveld Method, R.A. Young *in* The Rietveld Method, IUCR Monograph on Crystallography #5

Profex and BGMN*



BGMN Author: Dr. Joerg Bergmann
http://www.bgmn.de

Profex Author: Nicola Dobelin
http://profex.doebelin.org
GPL Open Source, Windows, OS X, Linux; front end for BGMN and for Fullprof 2k
Fundamental Parameters, adaptable to different instruments, multi-platform, robust

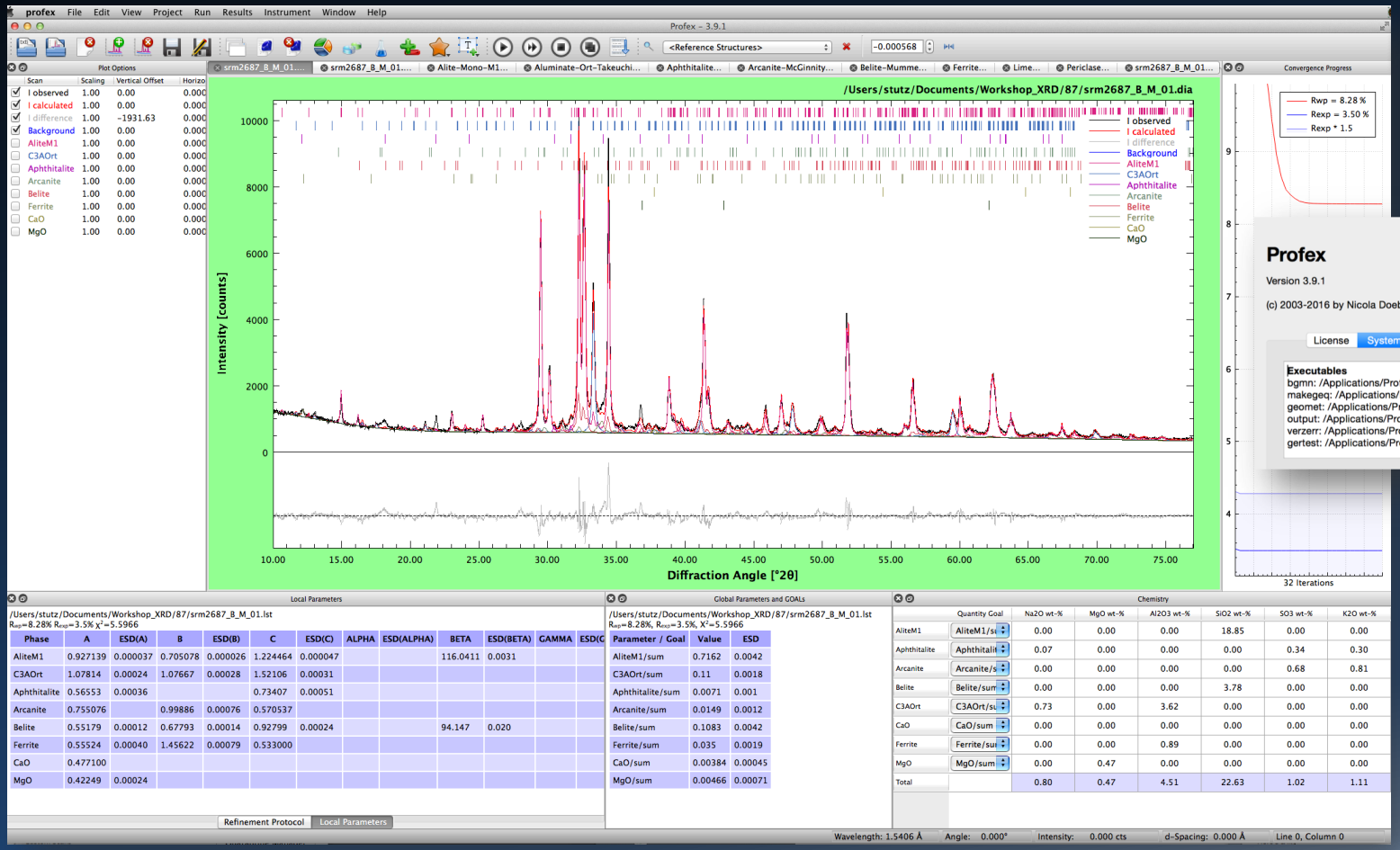
Profex

- Creates a control (input) file
- Copies structure models to working folder
- Copies the instrument configuration files
  (they allow calculation of the instrument contribution to the diffraction peak shapes based upon the X-ray optics)
- Allows editing of the working versions of the structure files and saves changes/refined values

Döbelin, N., Kleeberg, R., "Profex: a graphical user interface for the Rietveld refinement program *BGMN*", Journal of Applied Crystallography 48 (2015), 1573-1580.
DOI: doi:10.1107/S1600576715014685

** Adapted from Profex tutorials with some modification*

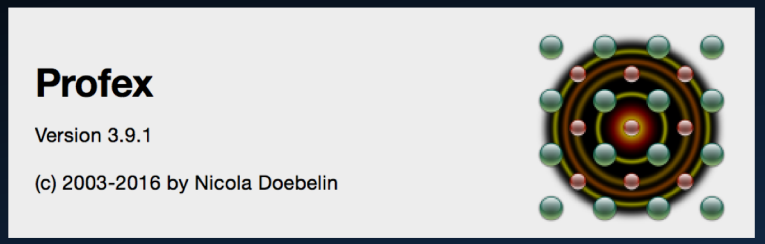
Profex/BGMN



In place of actual powder reference specimens for calibration, crystal structure models allow calculation of phase diffraction patterns. These models include space group, lattice parameters, atomic positions, atomic site occupancies, atomic vibrational parameters.

The code fits a multivariable model of structure, peak profile, background to the observed data

Initialize Profex



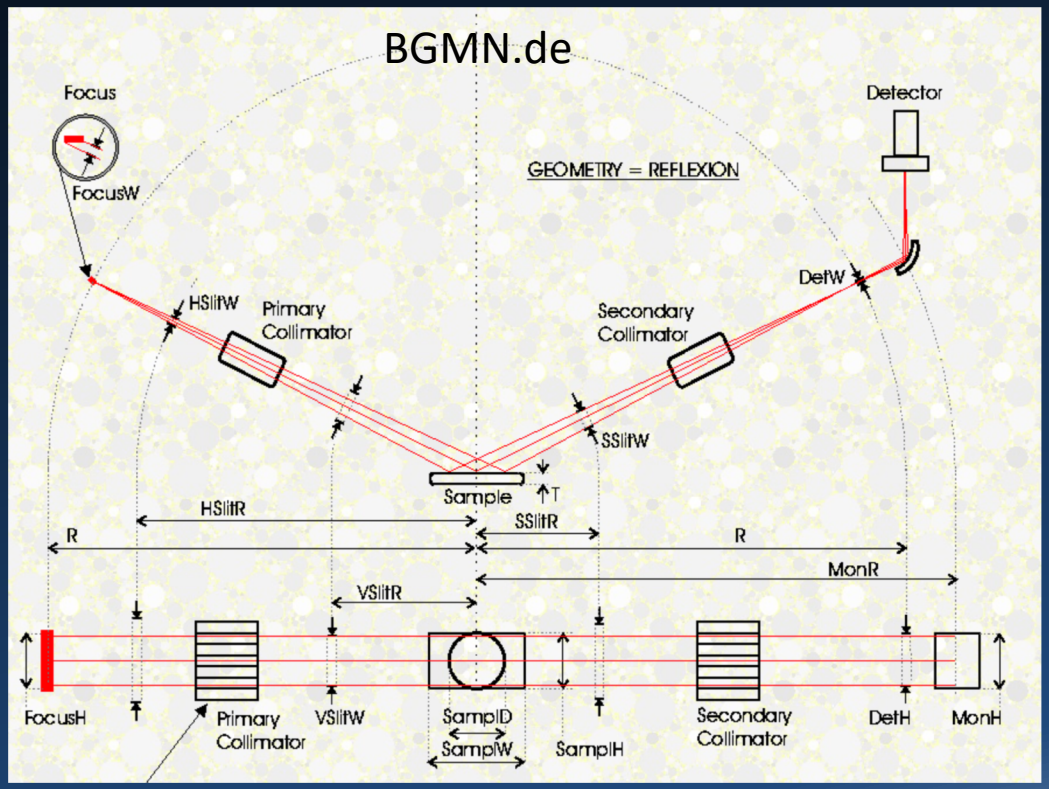
Plot Options
Select/deselect

Diffraction pattern display region

Refinement
Progress plot

Display global
parameters and goals

Profex/BGMN Instrument Configuration




```
%  BGMN Device Configuration File for Bruker D8
%  ----------------------------------------
%  Created by Nicola Doebelin, RMS Foundation,
%  Switzerland
%  November 12, 2012
%
%  Device Configuration:
%  - Detector: LynxEye
%  - Radiation: CuKa, Ni-filtered
%  - Soller Slits: 2.5 degrees
%  - Divergence Slit: fixed, 0.6 mm
%  - Anti-Scatter Slit: fixed, 6.76 mm
%  -Goniometer Radius: 217.5 mm
```

We will use File=D8_6div_4SS.geq or D8-06mm.geq, modifications of the D8 file provided.

Device functions are available for a wide variety of instruments and configurations, though some may require editing and re-saving. Different configurations will require a new device file!

Profex Configuration

Control file (.SAV)

- References model structures from the Cement_Structures Folder
- A copy of the structure is stored in the same folder as the data

```
% SampleID: SRM2688, 1b, micronized 0.6 divergence, 4 deg SS, 11-80 30 min, rep. 2
% Theoretical instrumental function
VERZERR=D8-06mm.geq
% Wavelength
LAMBDA=cu
% Phases
STRUC[1]=Alite-Mono_M1.str
STRUC[2]=Aluminate-Ort-Takeuchi.str
STRUC[3]=Belite_Beta_Mumme.str
STRUC[4]=Ferrite.str
STRUC[5]=Periclase.str
% Measured data
VAL[1]=88_1b_2.xy
% Minimum Angle (2theta)
% WMIN=10
% Maximum Angle (2theta)
% WMAX=60
% Result list output
LIST=88_1b_2.lst
% Peak list output
OUTPUT=88_1b_2.par
% Diagram output
DIAGRAMM=88_1b_2.dia
% Global parameters for zero point and sample displacement
EPS1=0
PARAM[1]=EPS2=0_-0.01^0.01
NTHREADS=8
PROTOKOLL=Y

sum=AliteM1+C3AOrt+Belite+Ferrite+MgO
GOAL[1]=AliteM1/sum
GOAL[2]=C3AOrt/sum
GOAL[3]=Belite/sum
GOAL[4]=Ferrite/sum
GOAL[5]=MgO/sum
```

Instrument profile

Wavelength

Refined Phases

Data File

Set two-theta angles for refinement (remove the leading "%")

Zero-Error (really *should* be zero)

Sample Displacement Parameter

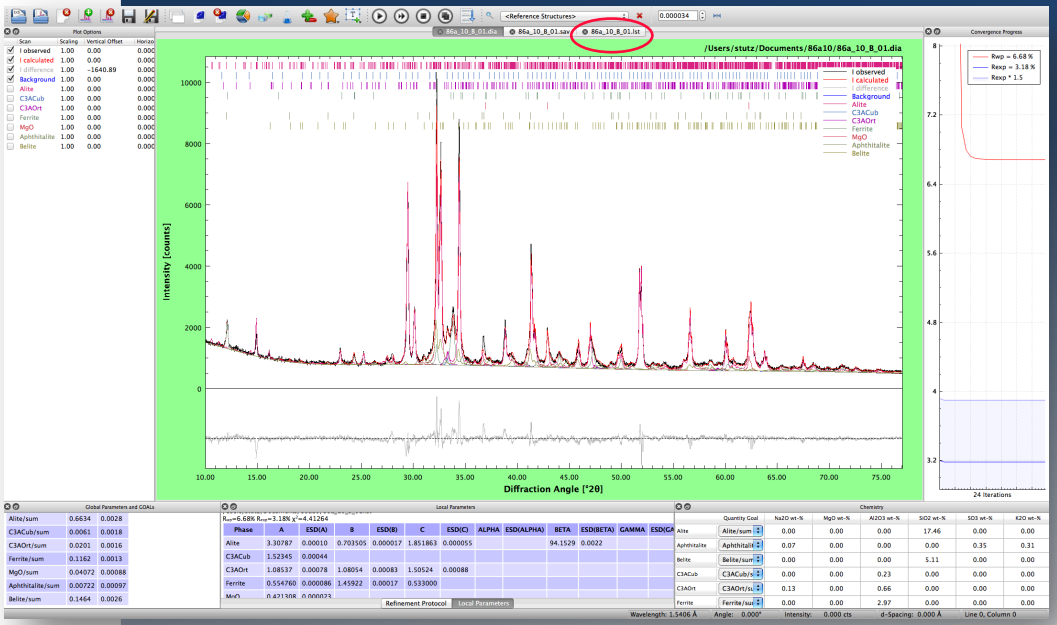
Phase Quantity Calculation

Profex Configuration

.LST file: Refinement Summary

```
Rietveld refinement to file(s) 88_1b_2.xy
BGMN version 4.2.22, 4420 measured points, 1295 peaks, 35 parameters
Start: Wed Jun 29 17:24:42 2016; End: Wed Jun 29 17:25:18 2016
63 iteration steps

Rp=5.00%  Rpb=17.21%  R=6.29%  Rwp=6.24% Rexp=3.78%
Durbin-Watson d=0.74
1-rho=1.88%

Global parameters and GOALs
***************************
AliteM1/sum=0.6680+-0.0034
C3AOrt/sum=0.0322+-0.0019
Belite/sum=0.1407+-0.0032
Ferrite/sum=0.1576+-0.0027
MgO/sum=0.00147+-0.00057
EPS2=0.0002031+-0.0000098

Local parameters and GOALs for phase AliteM1
*********************************************************
SpacegroupNo=7
HermannMauguin=P1c1
XrayDensity=3.144
Rphase=6.83%
UNIT=NM
A=0.929734+-0.000036
B=0.708015+-0.000025
C=1.223895+-0.000045
BETA=116.0856+-0.0032
B1=0.003124+-0.000037
GrainSize(1,1,1)=135.9+-1.6
GEWICHT=0.16634+-0.00078
Atomic positions for phase AliteM1
-----------------------------------------------
 2   0.0067  0.9987  0.0210   E=(CA+2(1.0000))
 2   0.0046  0.5002  0.0088   E=(CA+2(1.0000))
 2   0.3316  0.4694  0.7907   E=(CA+2(1.0000))
 2   0.3447  0.9982  0.7810   E=(CA+2(1.0000))
 2   0.6881  0.7566  0.4851   E=(CA+2(1.0000))
 2   0.0043  0.7268  0.2516   E=(CA+2(1.0000))
 2   0.3333  0.7524  0.0482   E=(CA+2(1.0000))
 2   0.6736  0.4852  0.7255   E=(CA+2(1.0000))
 2   0.6766  0.0348  0.7350   E=(CA+2(1.0000))
 2   0.8921  0.7611  0.3827   E=(O-2(1.0000))
 2   0.1109  0.7281  0.1056   E=(O-2(1.0000))
```

- File, refinement details
- refinement statistic measures
- refinement global parameter (shift) and phase fraction goals
- refinement data, by phase

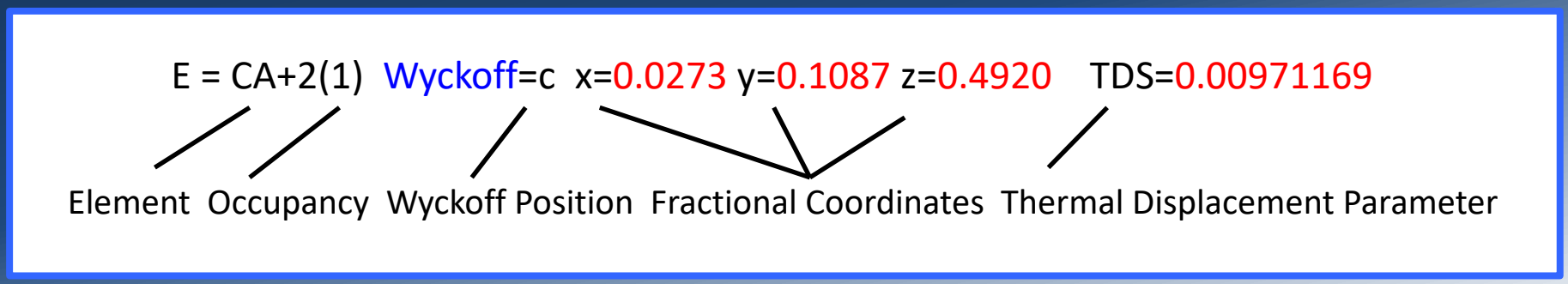


Profex Structure Files (.str)

Provided with the installation but may also be transcribed from databases

```
PHASE=Ferrite //
Formula=Ca2_Fe_Al2_O5 //
SpacegroupNo=46 Setting=2 HermannMauguin=Ibm2 Lattice=Orthorhombic //
PARAM=A=0.5557_0.551^0.563 PARAM=B=1.4543_1.43^1.465 PARAM=C=0.53616_0.533^0.542 //
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHAR0 //
GOAL=GrainSize(1,1,1) //
GOAL:Ferrite=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1) //
E=CA+2 Wyckoff=c x=0.02730000 y=0.10870000 z=0.49200000 TDS=0.00971169
E=FE+3(0.6400) Wyckoff=a x=0.00000000 y=0.00000000 z=0.00000000 TDS=0.00971169
E=AL+3(0.8300) Wyckoff=b x=0.92830000 y=0.25000000 z=0.95330000 TDS=0.00971169
E=O-2 Wyckoff=c x=0.25230000 y=0.98610000 z=0.24910000 TDS=0.00971169
E=O-2 Wyckoff=c x=0.06800000 y=0.14390000 z=0.02460000 TDS=0.00971169
E=O-2 Wyckoff=b x=0.86070000 y=0.25000000 z=0.61930000 TDS=0.00971169
E=AL+3(0.3600) Wyckoff=a x=0.00000000 y=0.00000000 z=0.00000000 TDS=0.00971169
E=FE+3(0.1700) Wyckoff=b x=0.92830000 y=0.25000000 z=0.95330000 TDS=0.00971169
```

GEWICHT (mass) = S* (Z*M*V)



Profex Structure Files (.str)

Provided with the installation but may also be transcribed from databases

```
PHASE=Ferrite //
Formula=Ca2_Fe_Al2_O5 //
SpacegroupNo=46 Setting=2 HermannMauguin=Ibm2 Lattice=Orthorhombic //
PARAM=A=0.5557_0.551^0.563 PARAM=B=1.4543_1.43^1.465 PARAM=C=0.53616_0.533^0.542 //
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHAR0 //
GOAL=GrainSize(1,1,1) //
GOAL:Ferrite=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1) //
E=CA+2 Wyckoff=c x=0.02730000 y=0.10870000 z=0.49200000 TDS=0.00971169
E=FE+3(0.6400) Wyckoff=a x=0.00000000 y=0.00000000 z=0.00000000 TDS=0.00971169
E=AL+3(0.8300) Wyckoff=b x=0.92830000 y=0.25000000 z=0.95330000 TDS=0.00971169
E=O-2 Wyckoff=c x=0.25230000 y=0.98610000 z=0.24910000 TDS=0.00971169
E=O-2 Wyckoff=c x=0.06800000 y=0.14390000 z=0.02460000 TDS=0.00971169
E=O-2 Wyckoff=b x=0.86070000 y=0.25000000 z=0.61930000 TDS=0.00971169
E=AL+3(0.3600) Wyckoff=a x=0.00000000 y=0.00000000 z=0.00000000 TDS=0.00971169
E=FE+3(0.1700) Wyckoff=b x=0.92830000 y=0.25000000 z=0.95330000 TDS=0.00971169
```

Fixed Parameter	Refined Parameter	Refined Parameter With Limits
A=0.5557	PARAM=A=0.5557	PARAM=A=0.5557_0.551^0.563
Name Value	Refine Name Value	Refine Name Value Lower Upper

B1: peak broadening from crystallite size (diffracting domains)
GEWICHT = Scale, Weight
Microabsorption correction, (ifthenelse(ifdef(d),exp(my*d*3/4),1), is not being used

Sources of Crystal Structure Data

American Mineralogist Crystal Structure Database
http://rruff.geo.arizona.edu/AMS/amcsd.php

Mincryst
http://database.iem.ac.ru/mincryst/

Crystallography Open Database
http://www.crystallography.net/cod/

Cements Crystal Structure Database
http://www.nist.gov/el/building_materials/inorganic/gsas1.cfm

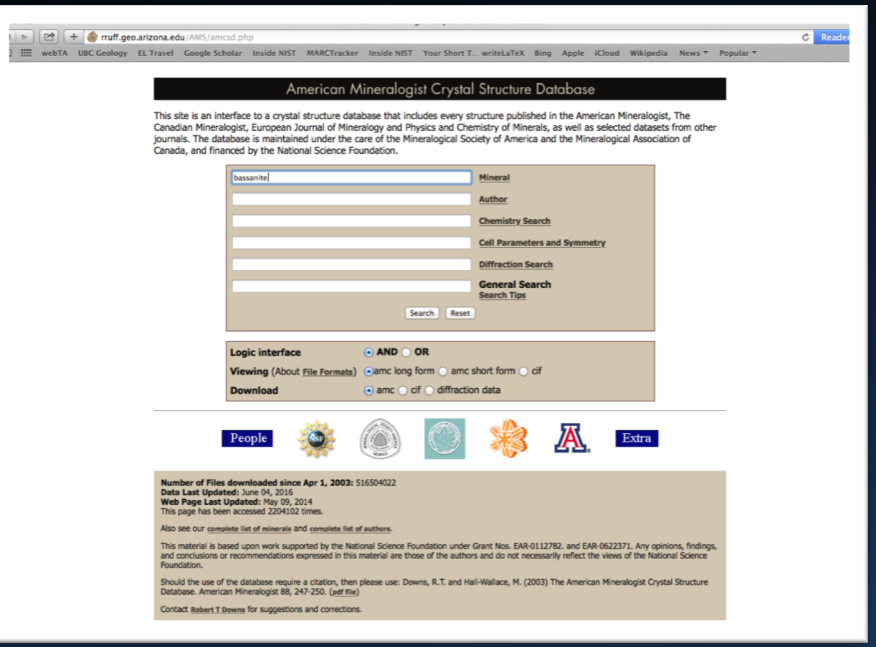
Commercial:
ICDD
https://icdd.com

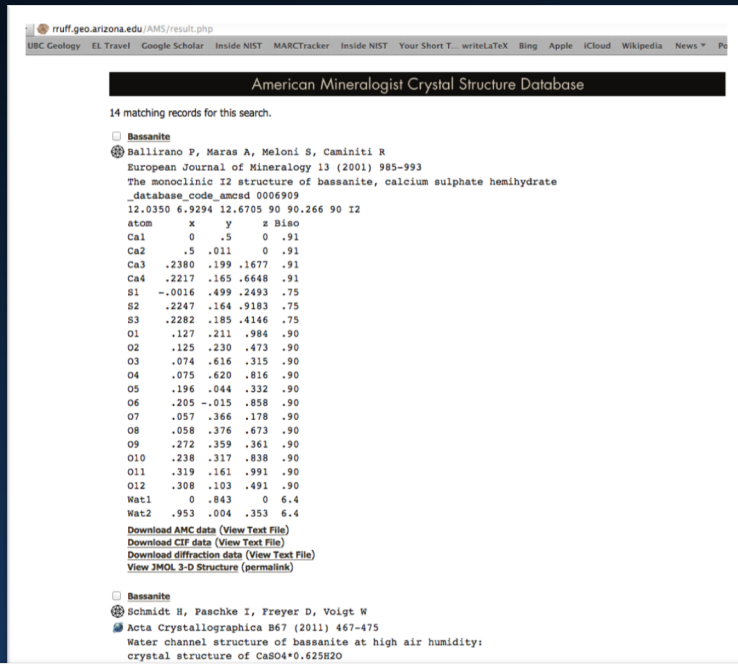
ICSD
https://icsd.fiz-karlsruhe.de/

.CIF File: Ferrite

```
data_cif
_audit_creation_date              'Monday, August 13, 2007 2:46 PM'
_audit_creation_method            'MDI-jPOWD'
_chemical_name_common
Brownmillerite
_chemical_formula_sum             'Ca2Al0.72Fe1.28O5'
_publ_section_references
 A.A.Colville & S.Geller, Crystal structures of Ca2Fe1.43Al0.57O5 and
Ca2Fe1.28Al0.72O5 , Acta Cryst. B28, 3196, 1972
Ibm2 (46) [PS oI36]
_symmetry_cell_setting            Orthorhombic
_symmetry_space_group_name_H-M    Ibm2
_symmetry_Int_Tables_number       46

_cell_length_a                    5.583
_cell_length_b                    14.58
_cell_length_c                    5.374
_cell_angle_alpha                 90
_cell_angle_beta                  90
_cell_angle_gamma                 90
_cell_volume                      437.444
_cell_formula_units_Z             4


loop_
      _atom_site_type_symbol
      _atom_site_label
      _atom_site_fract_x
      _atom_site_fract_y
      _atom_site_fract_z
      _atom_site_occupancy
      _atom_site_thermal_displace_type
      _atom_site_U_iso_or_equiv
      _atom_site_symmetry_multiplicity
Ca Ca(1)    0.27          0.1084        0.4907        1.0         Uiso    0.01230        8
Fe Fe(1)    0.0           0.0           0.0           0.83        Uiso    0.01230        4
Al Al(1)    0.0           0.0           0.0           0.17        Uiso    0.01230        4
Al Al(2)    0.9291        0.25          0.952         0.55        Uiso    0.01230        4
Fe Fe(2)    0.9291        0.25          0.952         0.45        Uiso    0.01230        4
O O(1)     0.2525        0.9859        0.2503        1.0         Uiso    0.01230        8
O O(2)     0.0683        0.1429        0.0256        1.0         Uiso    0.01230        8
O O(3)     0.8653        0.25          0.6133        1.0         Uiso    0.01230        4
```

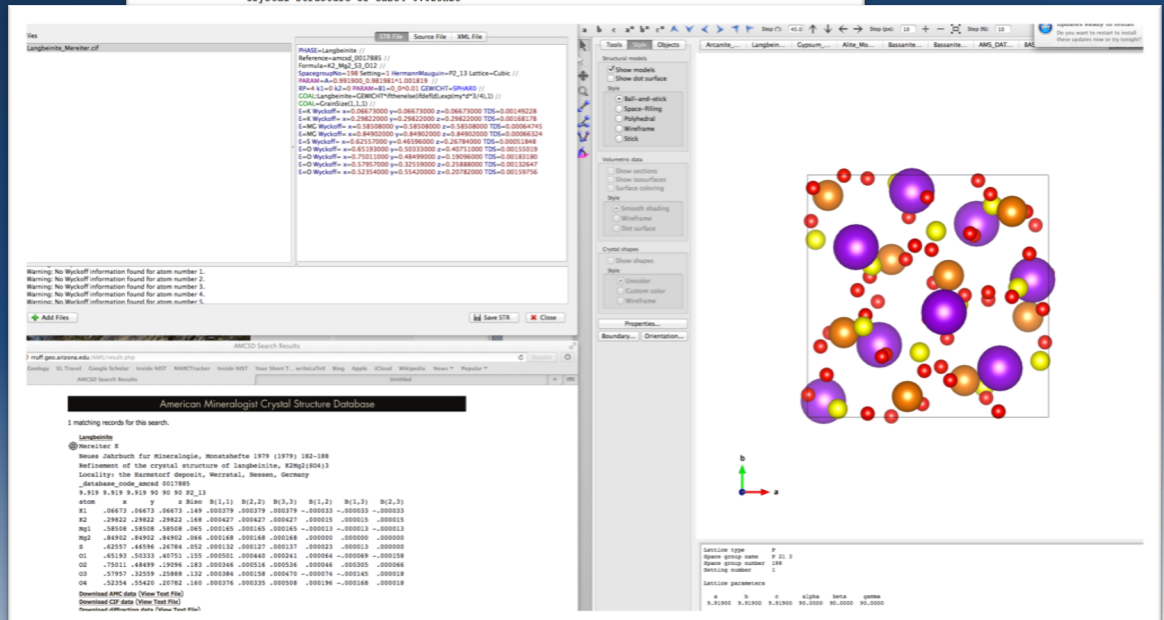
American Mineralogist Crystal Structure Database





- Search by a number of criteria
- Get a listing of structure data
- Download .CIF file
- Import into Profex *

**Use Vesta to view structure and get the Wyckoff positions*



http://rruff.geo.arizona.edu/AMS/amcsd.php

http://jp-minerals.org/vesta/en/

Import CIF Example

Profex->File->Import Structure File

- Point toward selected .CIF file
- At the same time, open that CIF file using VESTA
  - necessary to get the Wyckoff position notation
- Add PHASE name: PHASE=Ferrite //
- Add phase name to GOAL: GOAL:Ferrite=GEWICHT …
- Add atom charge (CA+2, AL+3, FE+3, O-2)
- Save to Structures Folder; include extension .str

Profex

Vesta

Import Structure Files

Files

c4af2_jp.cif

STR File | Source File | XML File

```
PHASE= //
Formula=Ca2[Fe0.76Al0.24](Fe0.24Al0.76)AlO5 //
SpacegroupNo=46 Setting=2 HermannMauguin=Ibm2 Lattice=Orthorhombic //
PARAM=A=0.558400_0.552816^0.563984 PARAM=B=1.460000_1.445400^1.474600 PARAM=C=0.537400_0.532026^0.542774 //
RP=4 k1=0 k2=0 PARAM=B1=0_0^0.01 GEWICHT=SPHAR0 //
GOAL:=GEWICHT*ifthenelse(ifdef(d),exp(my*d*3/4),1) //
E=CA Wyckoff= x=0.02730000 y=0.10870000 z=0.49200000 TDS=0.00971169
E=FE(0.7600) Wyckoff= x=0.00000000 y=0.00000000 z=0.00000000 TDS=0.00971169
E=AL(0.7600) Wyckoff= x=0.92830000 y=0.25000000 z=0.95330000 TDS=0.00971169
E=O Wyckoff= x=0.25230000 y=0.98610000 z=0.24910000 TDS=0.00971169
E=O Wyckoff= x=0.06800000 y=0.14390000 z=0.02460000 TDS=0.00971169
E=O Wyckoff= x=0.86070000 y=0.25000000 z=0.61930000 TDS=0.00971169
E=AL(0.2400) Wyckoff= x=0.00000000 y=0.00000000 z=0.00000000 TDS=0.00971169
E=FE(0.2400) Wyckoff= x=0.92830000 y=0.25000000 z=0.95330000 TDS=0.00971169
```

```
Warning: No Wyckoff information found for atom number 0.
Warning: No Wyckoff information found for atom number 1.
Warning: No Wyckoff information found for atom number 2.
Warning: No Wyckoff information found for atom number 3.
Warning: No Wyckoff information found for atom number 4.
Warning: No Wyckoff information found for atom number 5.
Warning: No Wyckoff information found for atom number 6.
Warning: No Wyckoff information found for atom number 7.
```

Add Files | Save STR | Close

Tools | Style | Objects

Structural models

Show models

Show dot surface

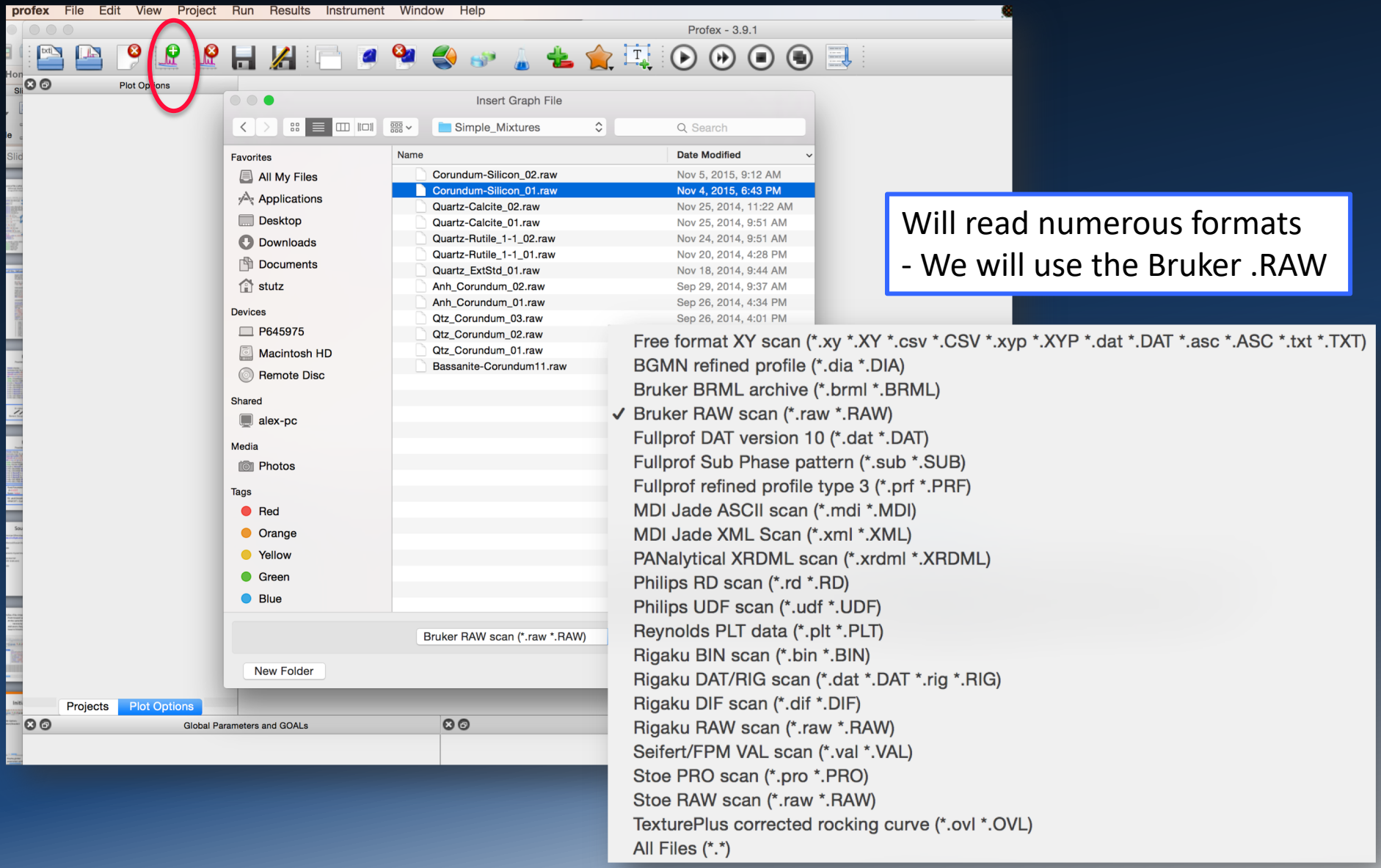
Style

Ball-and-stick

c4af2_jp.cif

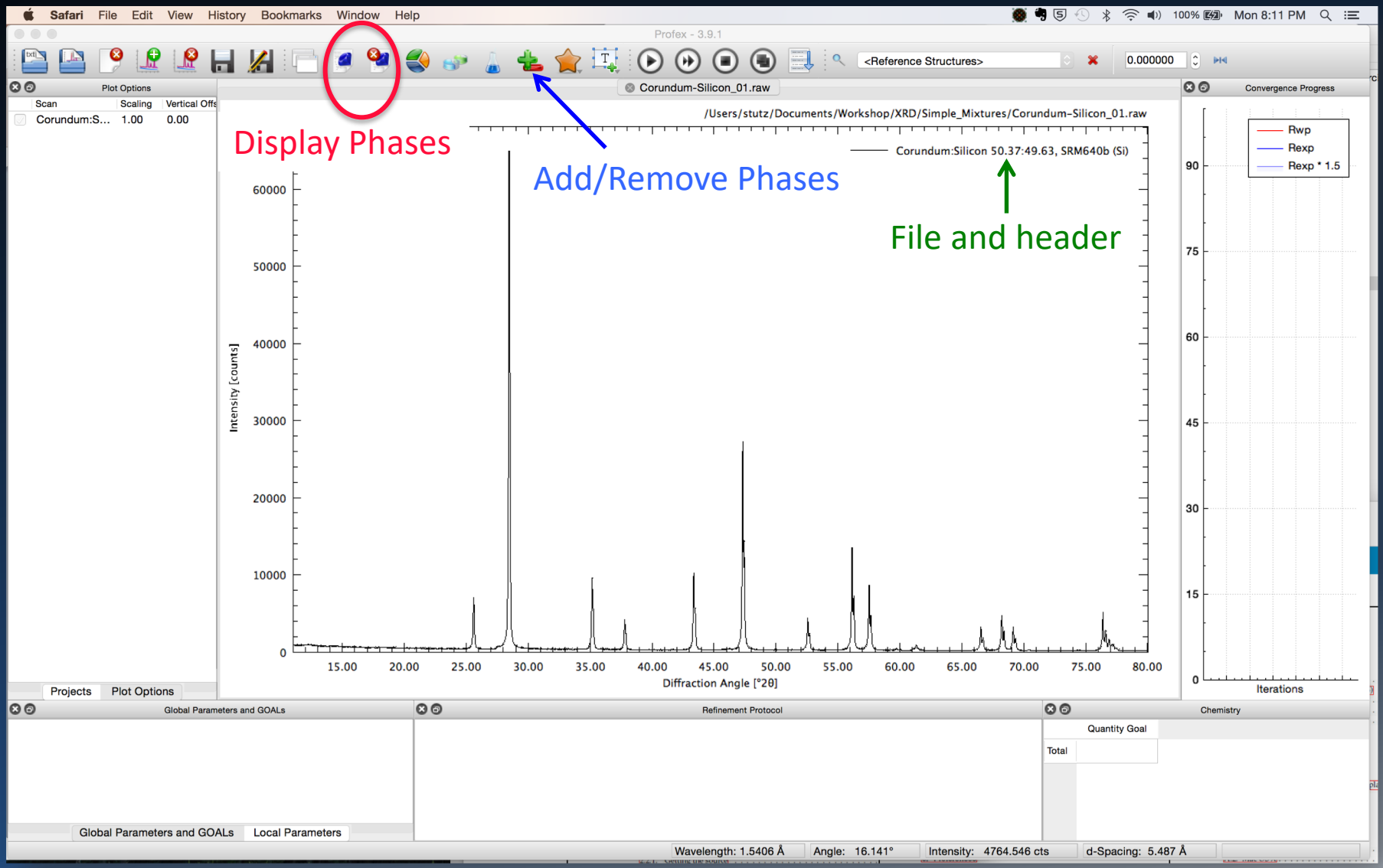
```
OpenGL version: 2.1 NVIDIA-10.4.2 310.41.35f01
Video configuration: NVIDIA GeForce GT 650M OpenGL Engine
Maximum supported width and height of the viewport: 16384 x 16384
OpenGL depth buffer bit: 16

/Users/stutz/Documents/XRD/Cements_XRD_Database/CIF_Files/c4af2_jp.cif
================================================================================
Title

Lattice type        I
Space group name    I b m 2
Space group number  46
Setting number      2

Lattice parameters

   a        b        c       alpha    beta     gamma
 5.58400 14.60000  5.37400  90.0000  90.0000  90.0000

Unit-cell volume = 438.122900 Å^3

Structure parameters

                        x        y        z        Occ.     U        Site     Sym.
   1 Ca  Ca(1)       0.02730  0.10870  0.49200  1.000    0.012    8c       1
   2 Fe  Fe(1)       0.00000  0.00000  0.00000  0.760    0.012    4a       ..2
   3 Al  Al(2)       0.92830  0.25000  0.95330  0.760    0.012    4b       m..
   4 O   O(1)        0.25230  0.98610  0.24910  1.000    0.012    8c       1
   5 O   O(2)        0.06800  0.14390  0.02460  1.000    0.012    8c       1
   6 O   O(3)        0.86070  0.25000  0.61930  1.000    0.012    4b       m..
   7 Al  Al(1)       0.00000  0.00000  0.00000  0.240    0.012    4a       ..2
   8 Fe  Fe(2)       0.92830  0.25000  0.95330  0.240    0.012    4b       m..
================================================================================

Number of polygons and unique vertices on isosurface = 0 (0)
47 atoms, 0 bonds, 0 polyhedra; CPU time = 4 ms
```

Load Data File: File->Insert Scan

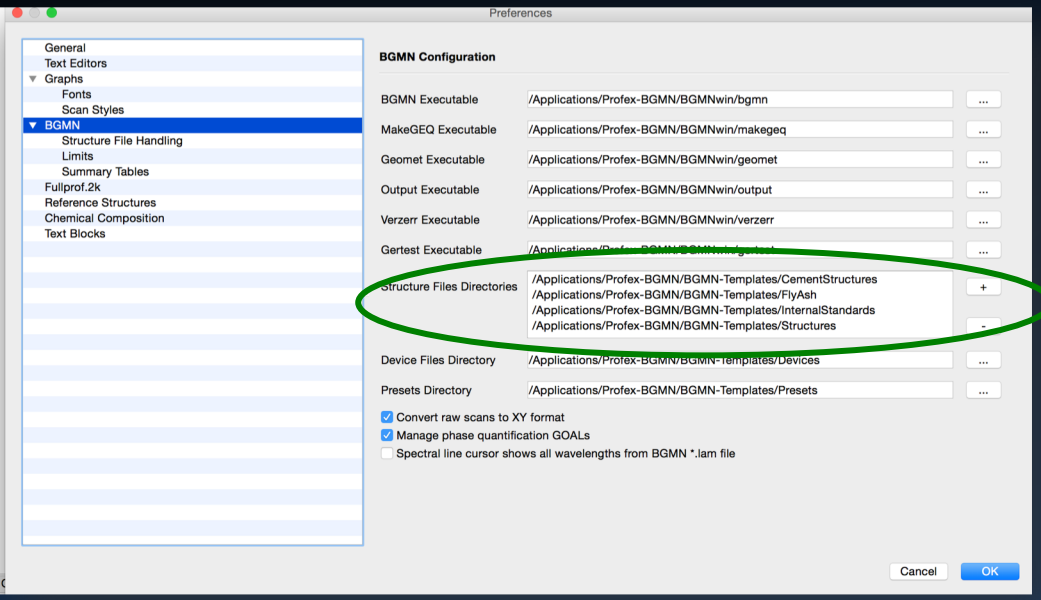


Will read numerous formats
- We will use the Bruker .RAW

Corundum-Silicon File Added



Structure Files

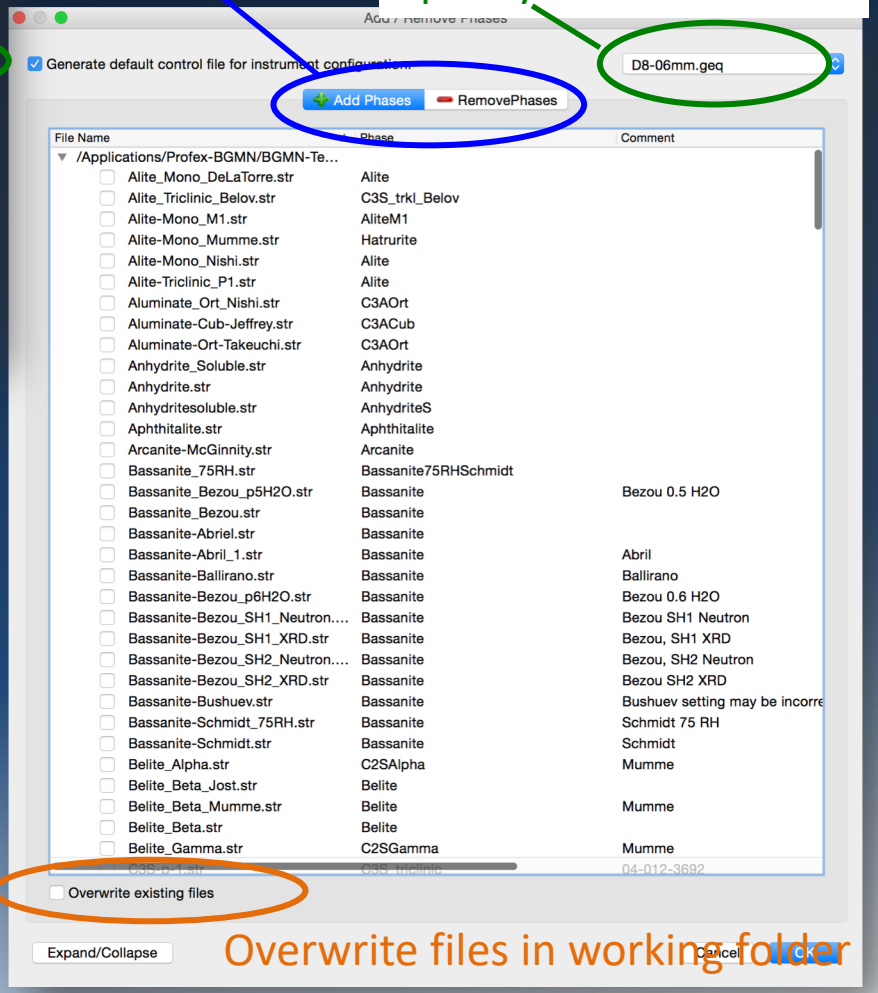


Profex->Preferences

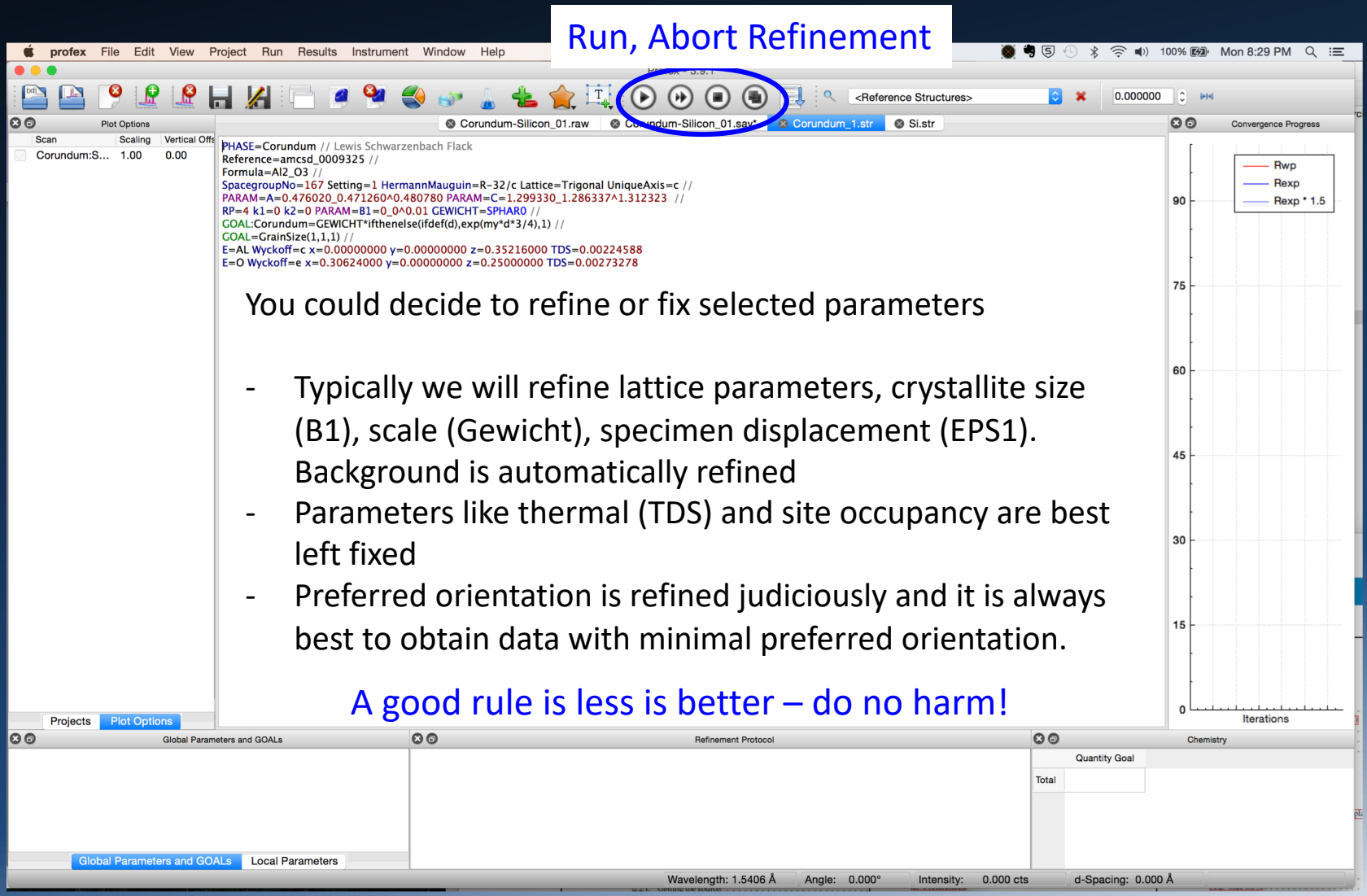
- Paths to structure files
- Will list according to list order

- CementStructures
- FlyAsh
- InternalStandards
- Structures

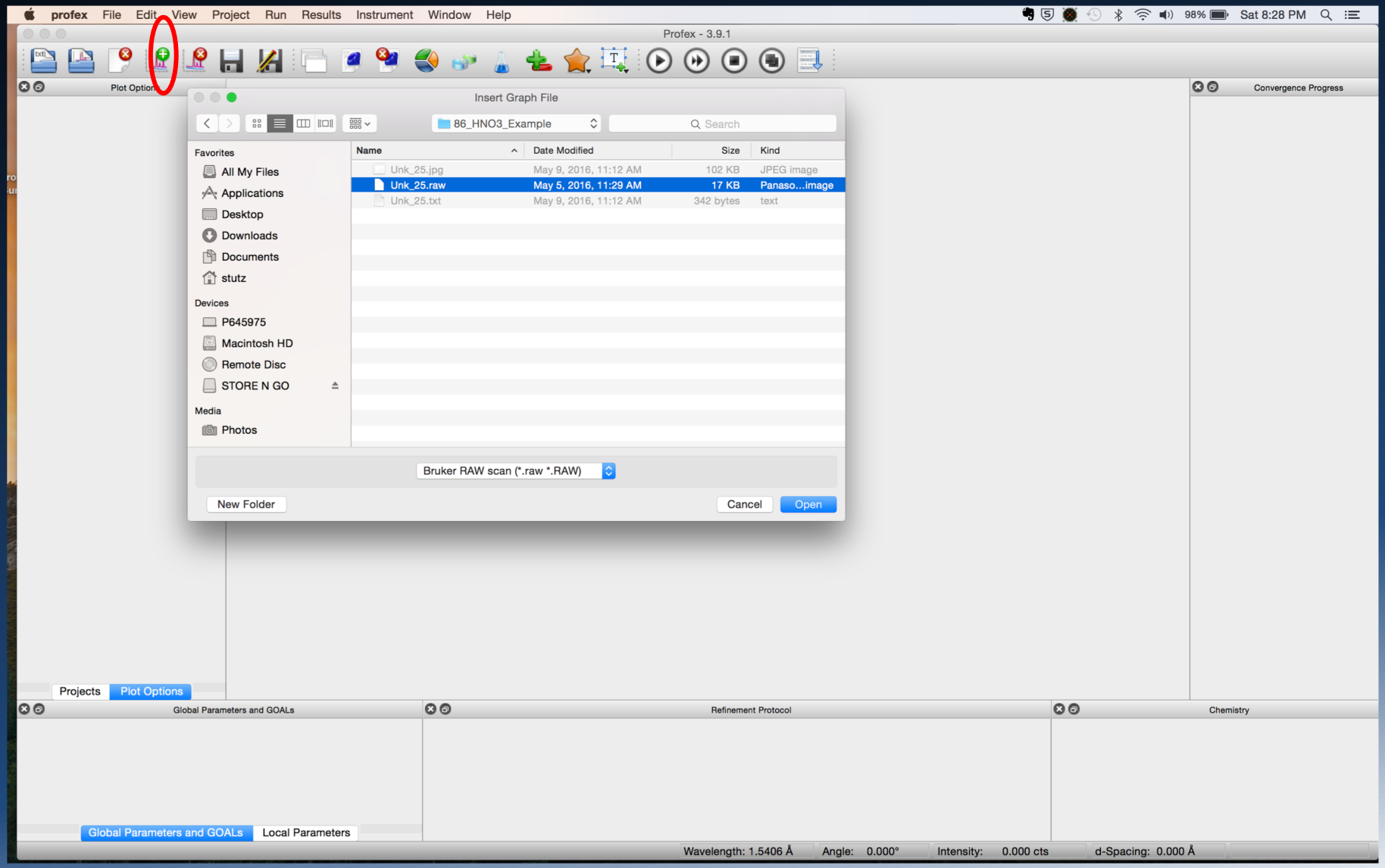
- Once added, select Display to see the copied data, make changes if desired



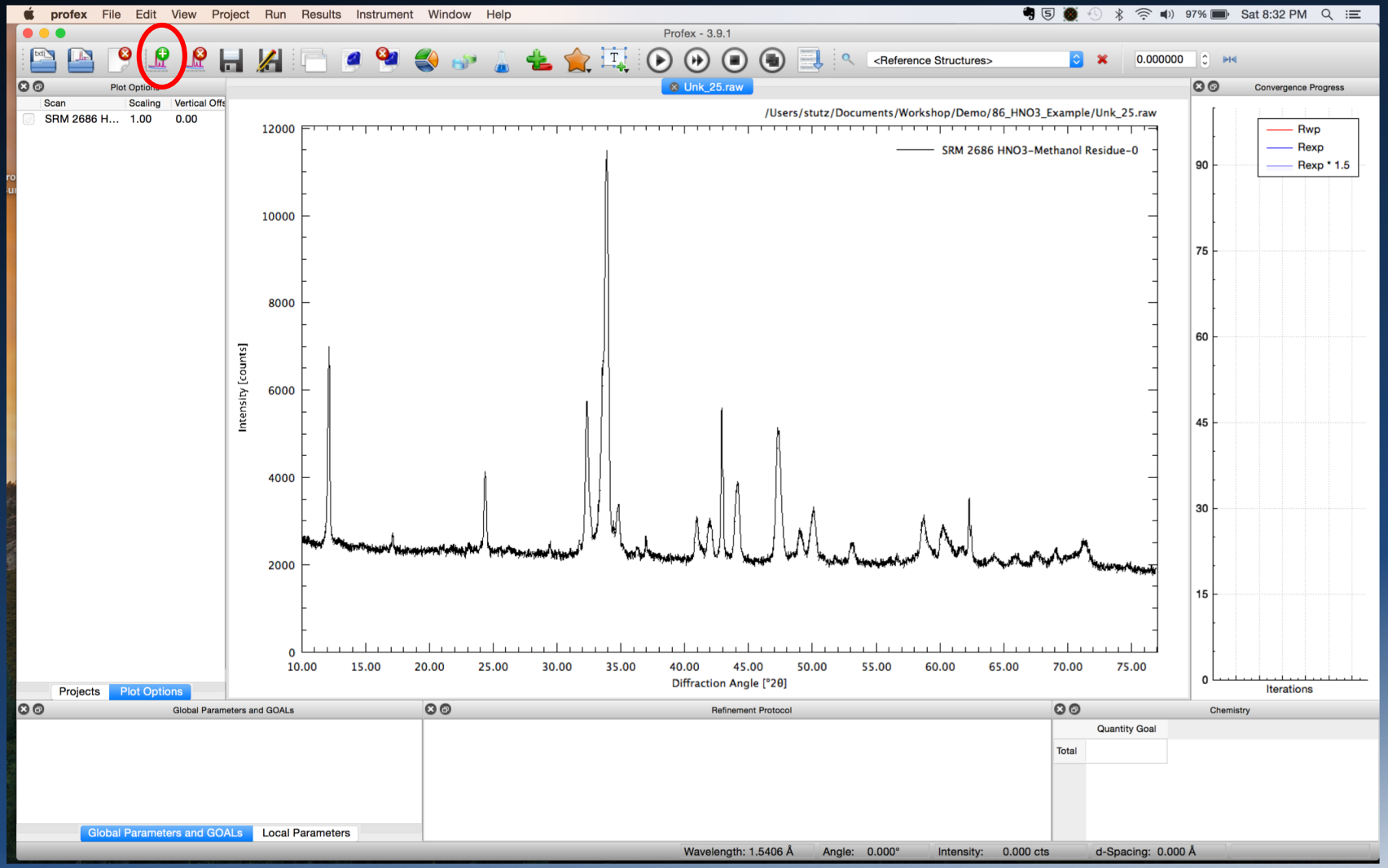
Profex: .raw, .sav, .str display



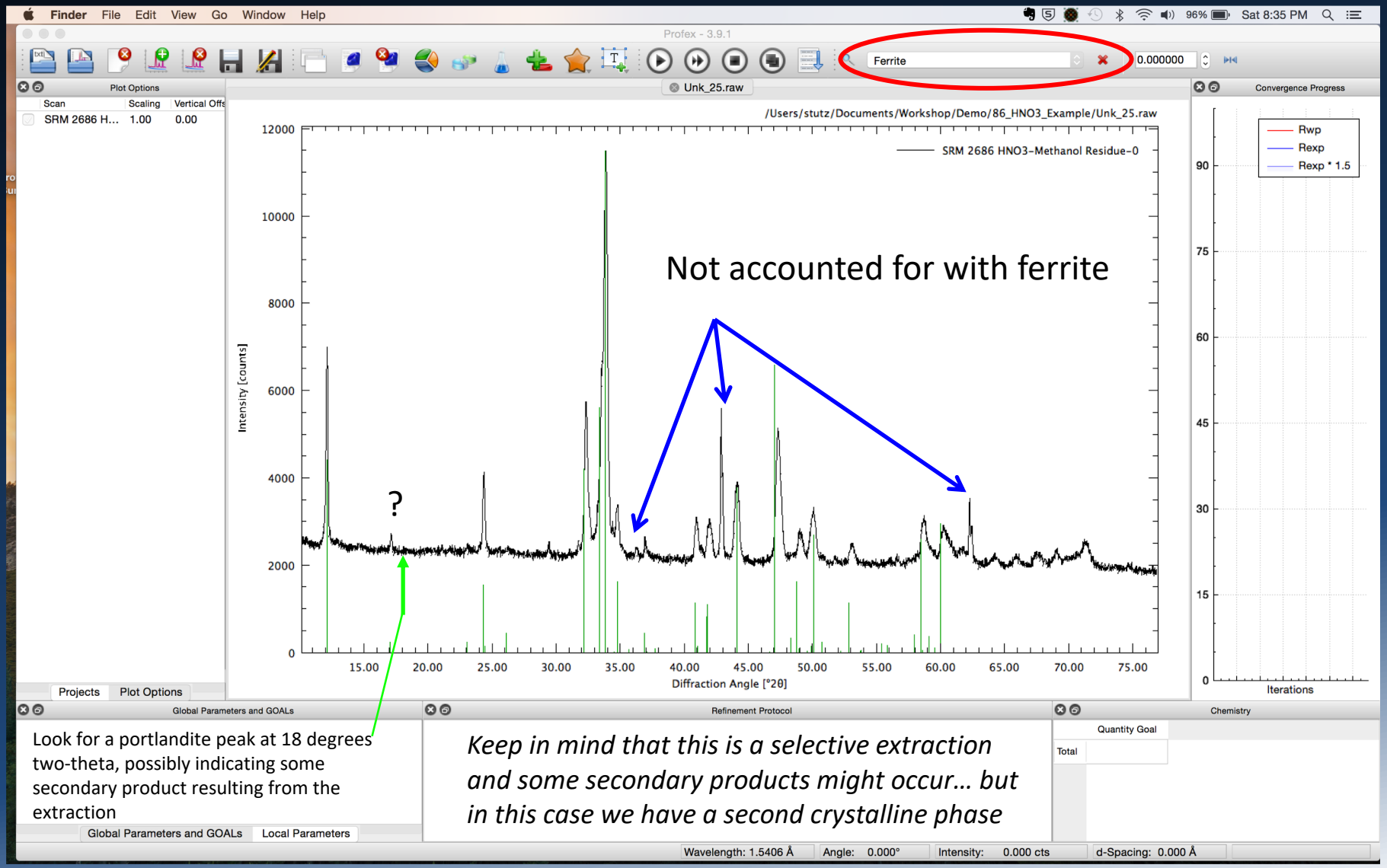
Initialize Profex, Insert Scan



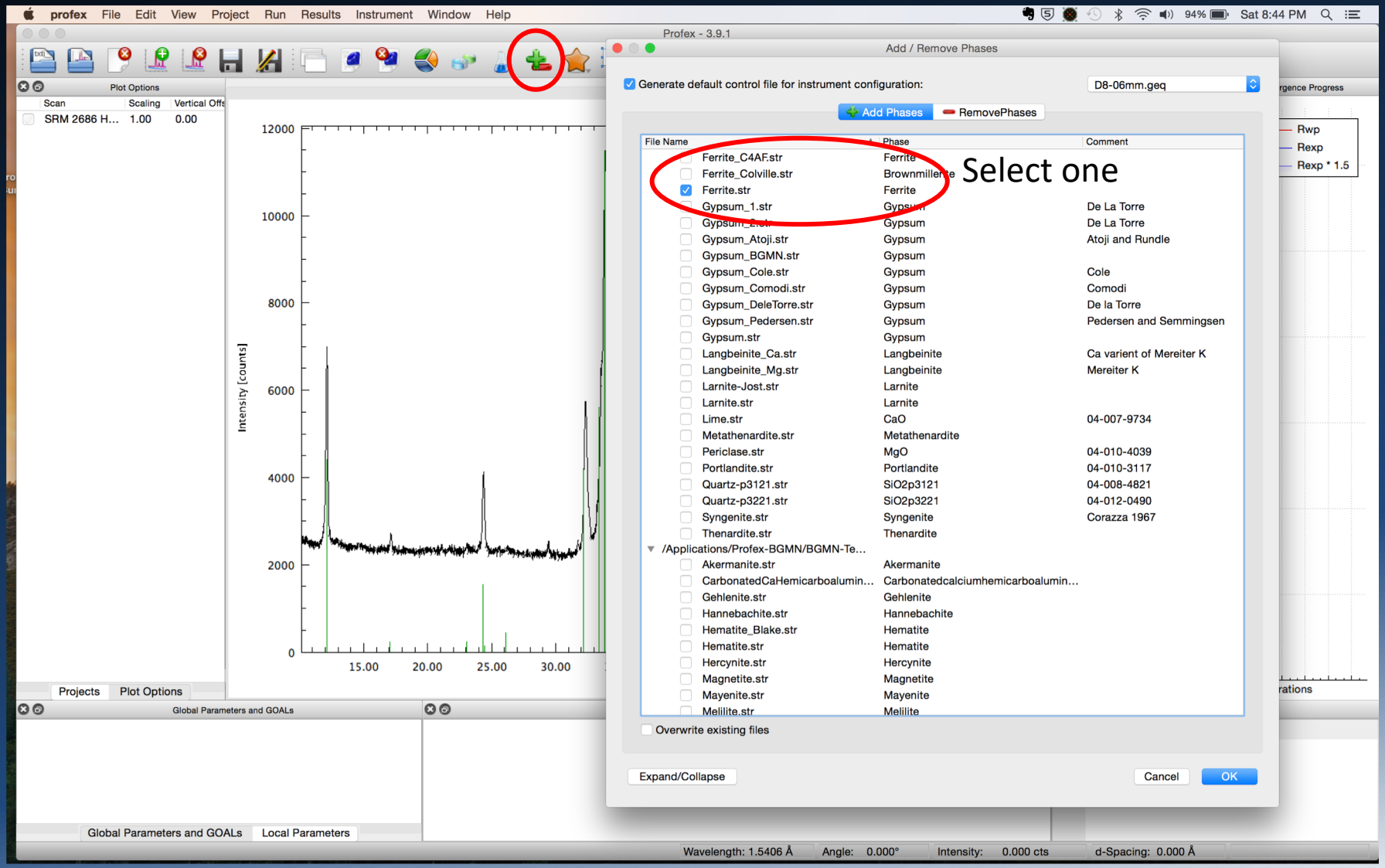
Initialize Profex, Insert Scan



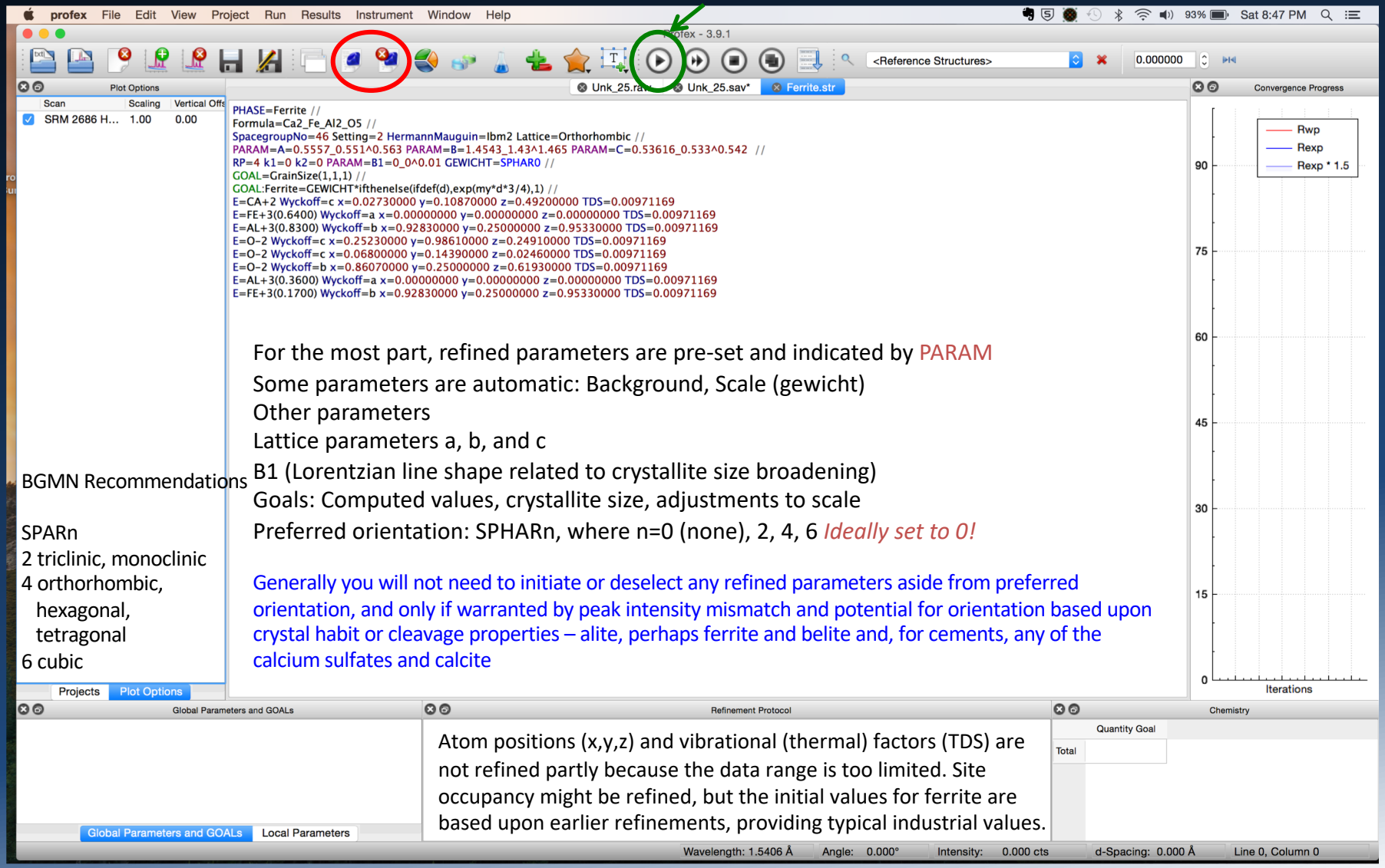
Phase d&I Search for Phase Identification



Add Phase


Add / Remove Phases
Generate default control file for instrument configuration:
D8-06mm.geq
Add Phases
RemovePhases
File Name
Phase
Comment
Select one
Ferrite_C4AF.str
Ferrite
Ferrite_Colville.str
Brownmillerite
Ferrite.str
Ferrite
Gypsum_1.str
Gypsum
De La Torre
Gypsum_2.str
Gypsum
De La Torre
Gypsum_Atoji.str
Gypsum
Atoji and Rundle
Gypsum_BGMN.str
Gypsum
Gypsum_Cole.str
Gypsum
Cole
Gypsum_Comodi.str
Gypsum
Comodi
Gypsum_DeleTorre.str
Gypsum
De la Torre
Gypsum_Pedersen.str
Gypsum
Pedersen and Semmingsen
Gypsum.str
Gypsum
Langbeinite_Ca.str
Langbeinite
Ca varient of Mereiter K
Langbeinite_Mg.str
Langbeinite
Mereiter K
Larnite-Jost.str
Larnite
Larnite.str
Larnite
Lime.str
CaO
04-007-9734
Metathenardite.str
Metathenardite
Periclase.str
MgO
04-010-4039
Portlandite.str
Portlandite
04-010-3117
Quartz-p3121.str
SiO2p3121
04-008-4821
Quartz-p3221.str
SiO2p3221
04-012-0490
Syngenite.str
Syngenite
Corazza 1967
Thenardite.str
Thenardite
/Applications/Profex-BGMN/BGMN-Te...
Akermanite.str
Akermanite
CarbonatedCaHemicarboalumin...
Carbonatedcalciumhemicarboalumin...
Gehlenite.str
Gehlenite
Hannebachite.str
Hannebachite
Hematite_Blake.str
Hematite
Hematite.str
Hematite
Hercynite.str
Hercynite
Magnetite.str
Magnetite
Mayenite.str
Mayenite
Melilite.str
Melilite
Overwrite existing files
Expand/Collapse
Cancel
OK
Profex - 3.9.1
Plot Options
Scan
Scaling
Vertical Offs
SRM 2686 H...
1.00
0.00
Intensity [counts]
12000
10000
8000
6000
4000
2000
0
15.00
20.00
25.00
30.00
Projects
Plot Options
Global Parameters and GOALs
Local Parameters
Rwp
Rexp
Rexp * 1.5
Wavelength: 1.5406 Å
Angle: 0.000°
Intensity: 0.000 cts
d-Spacing: 0.000 Å

Display Structure



For the most part, refined parameters are pre-set and indicated by PARAM

Some parameters are automatic: Background, Scale (gewicht)

Other parameters

Lattice parameters a, b, and c

B1 (Lorentzian line shape related to crystallite size broadening)

Goals: Computed values, crystallite size, adjustments to scale

Preferred orientation: SPHARn, where n=0 (none), 2, 4, 6 *Ideally set to 0!*

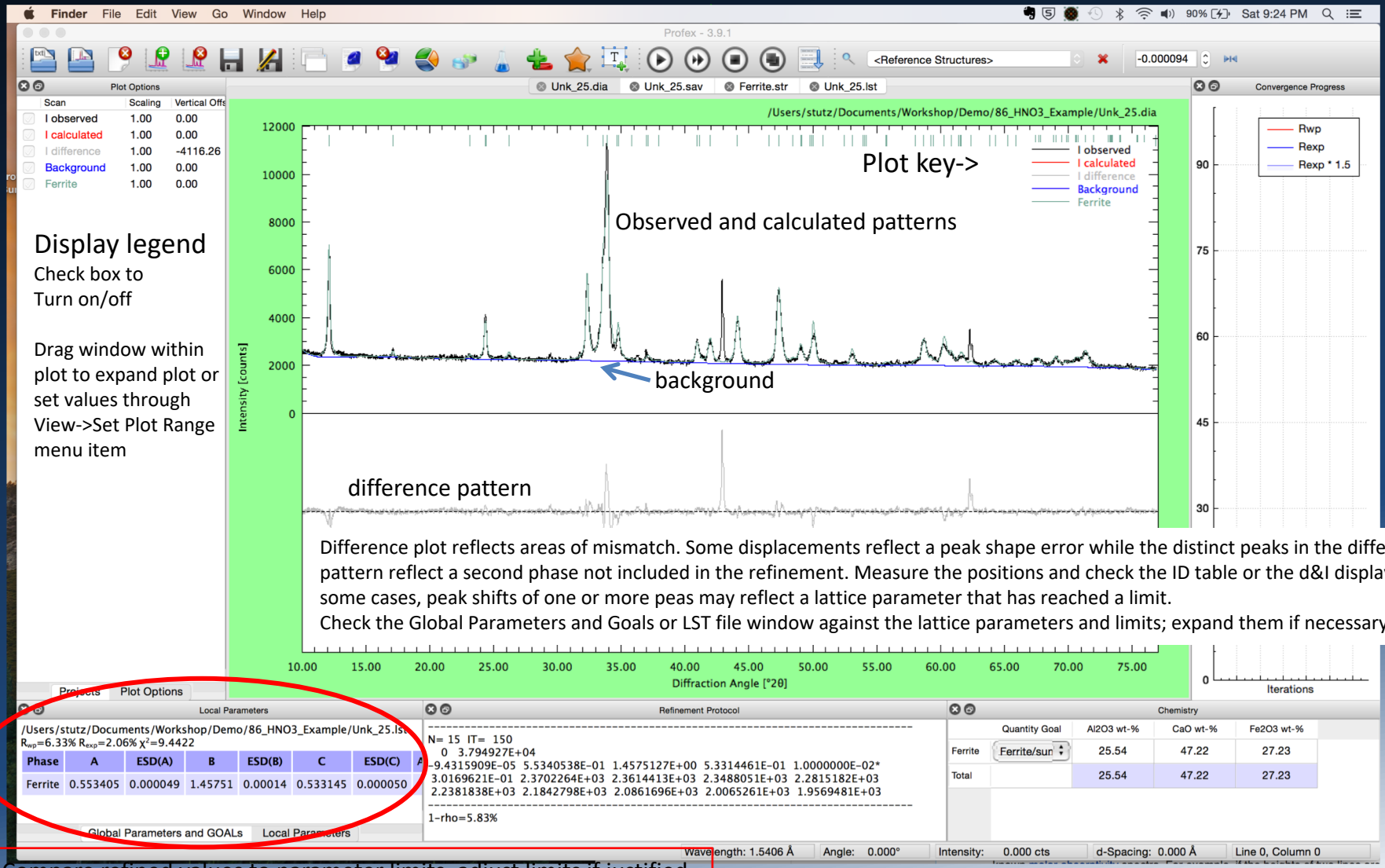
Generally you will not need to initiate or deselect any refined parameters aside from preferred orientation, and only if warranted by peak intensity mismatch and potential for orientation based upon crystal habit or cleavage properties – alite, perhaps ferrite and belite and, for cements, any of the calcium sulfates and calcite

BGMN Recommendations

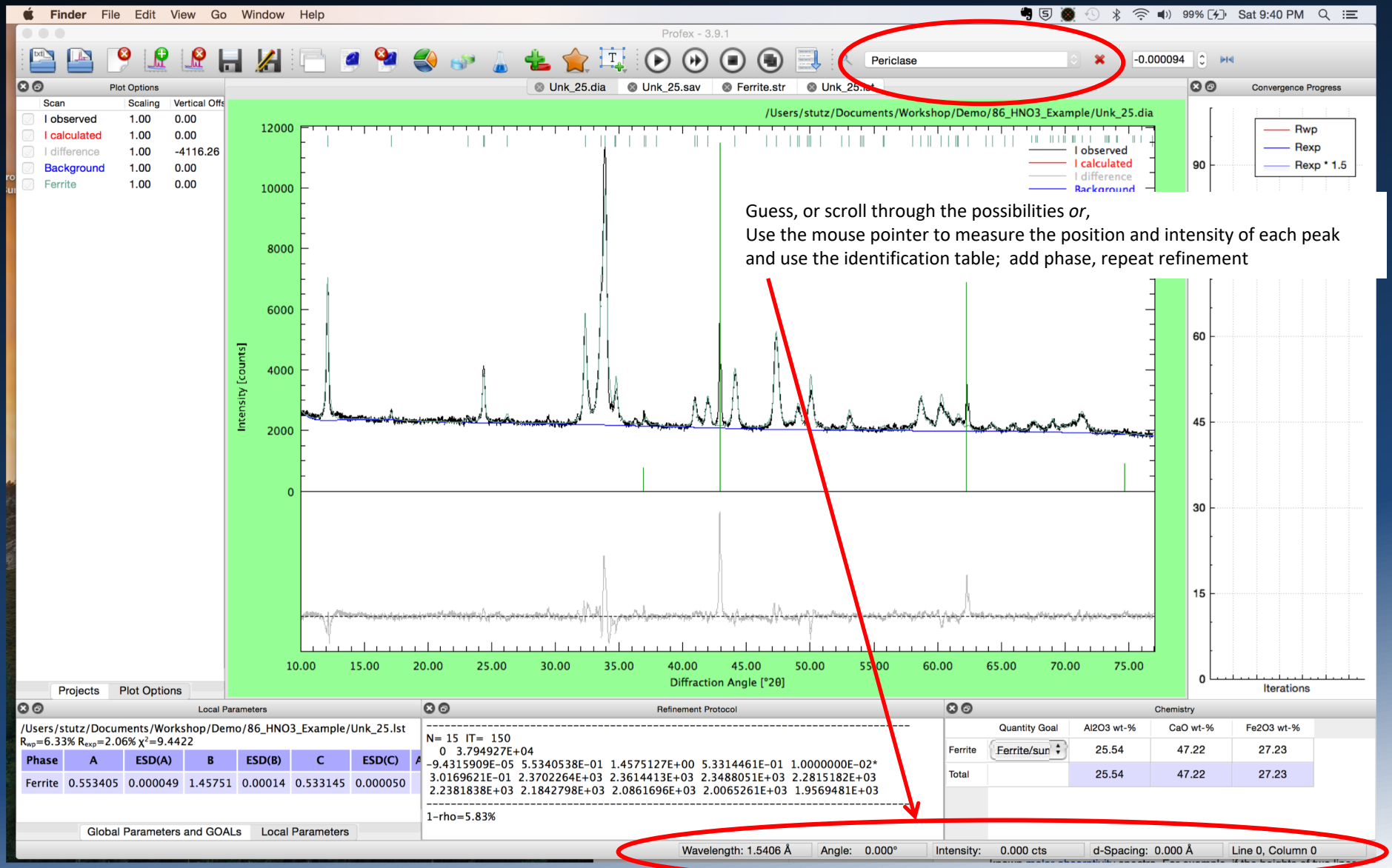
SPARn
2 triclinic, monoclinic
4 orthorhombic, hexagonal, tetragonal
6 cubic

Atom positions (x,y,z) and vibrational (thermal) factors (TDS) are not refined partly because the data range is too limited. Site occupancy might be refined, but the initial values for ferrite are based upon earlier refinements, providing typical industrial values.

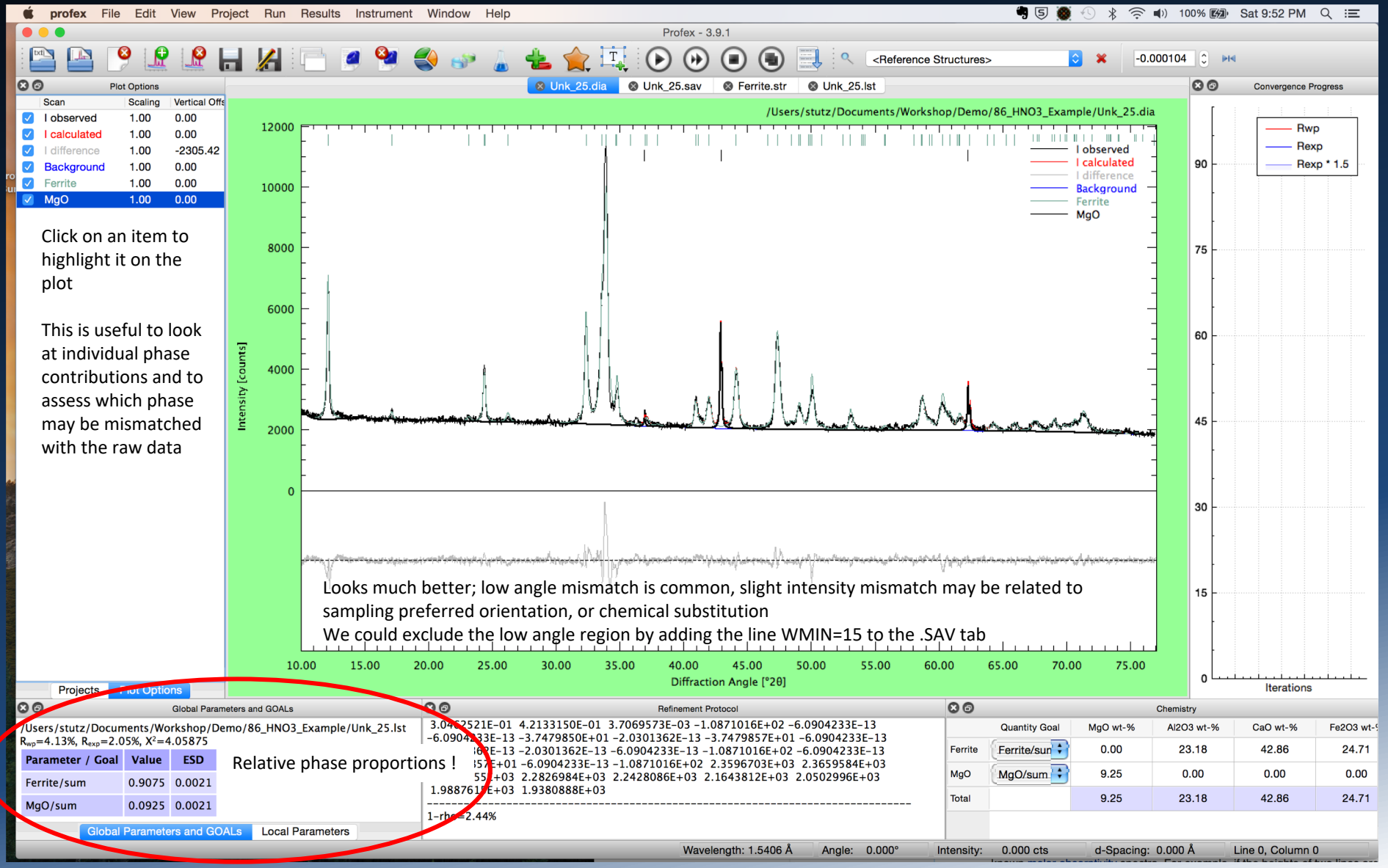
Examine Results: Plot, Parameters, LST



Search for Missing Phase

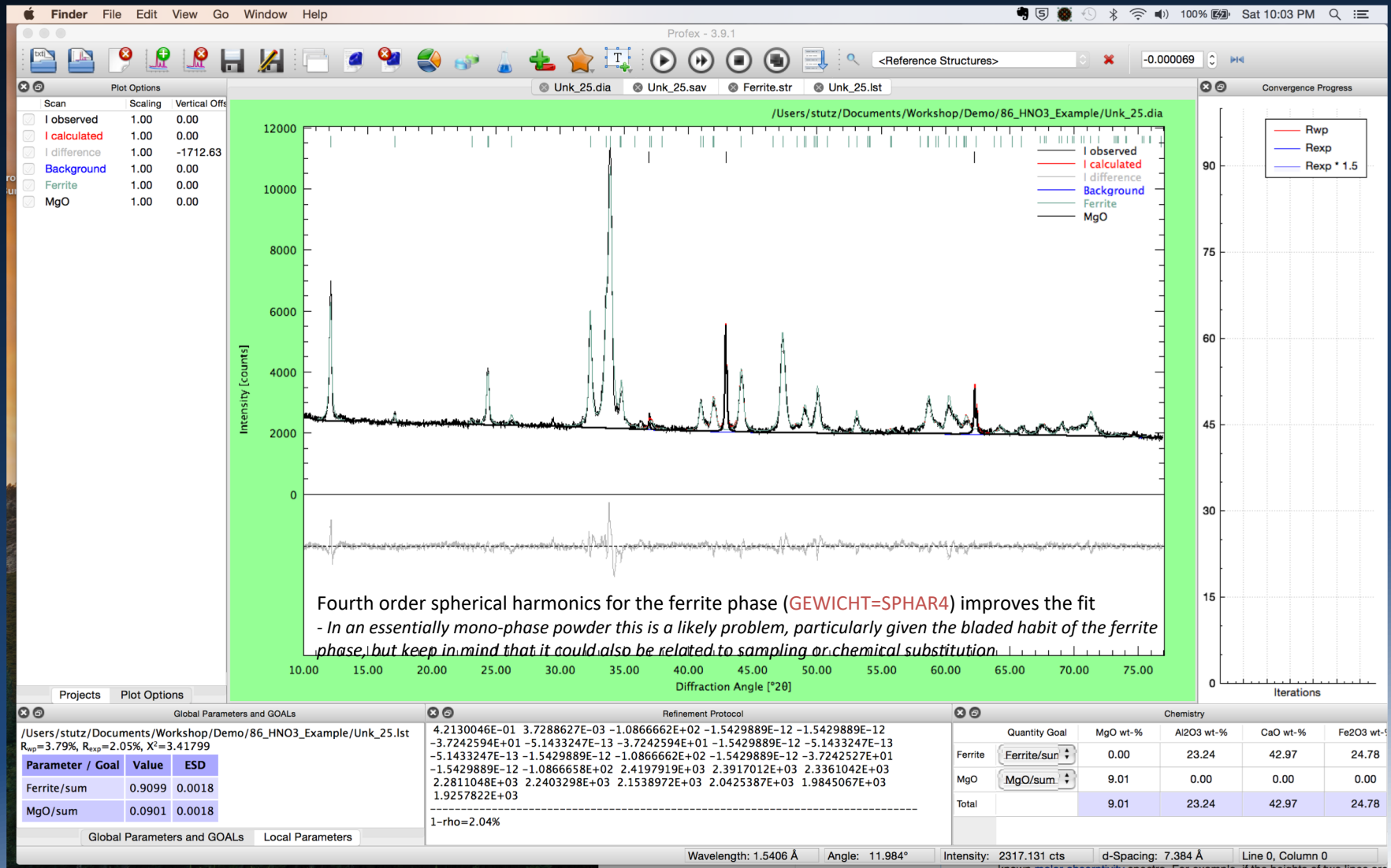


Ferrite and Periclase Refinement

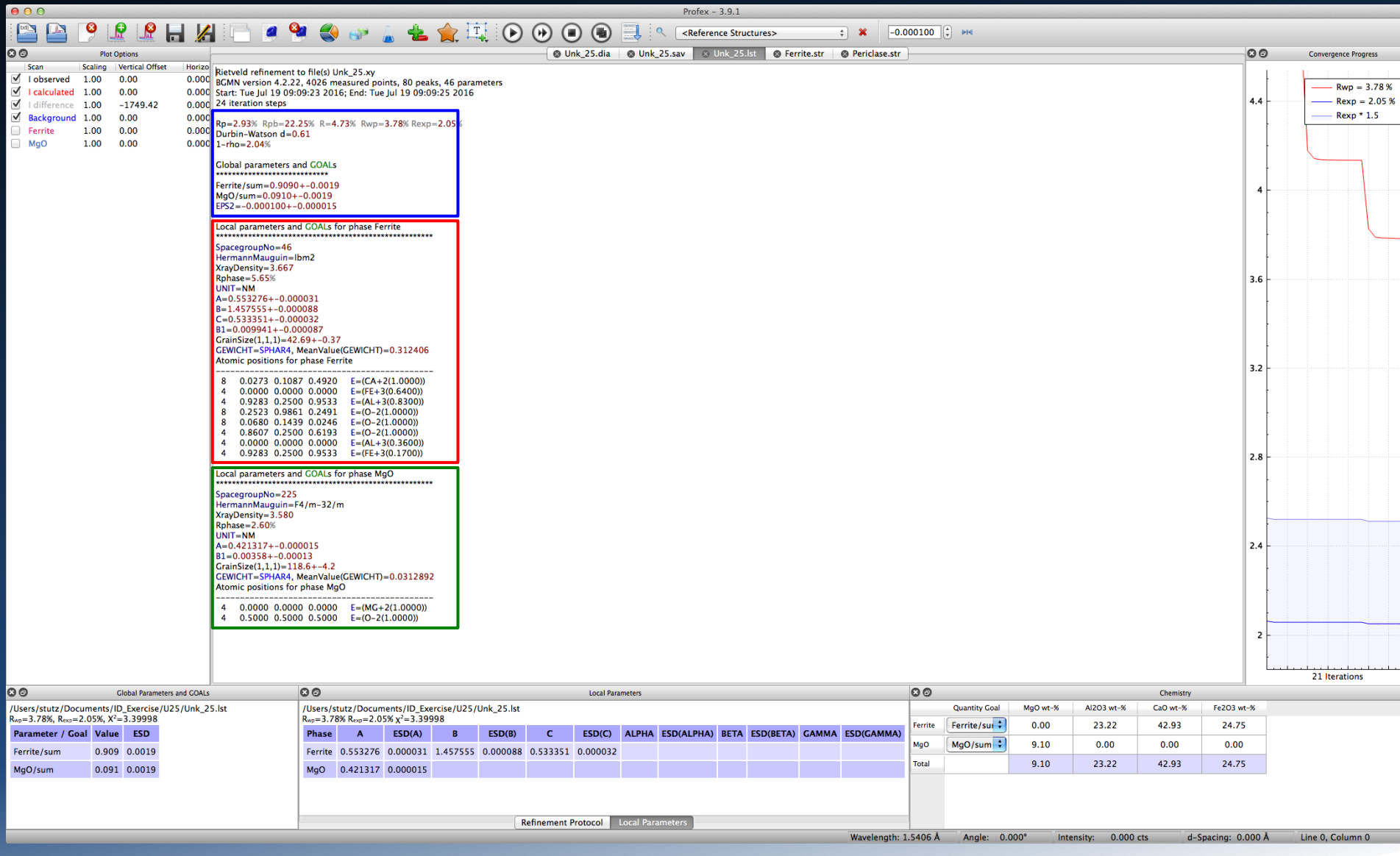


Preferred Orientation

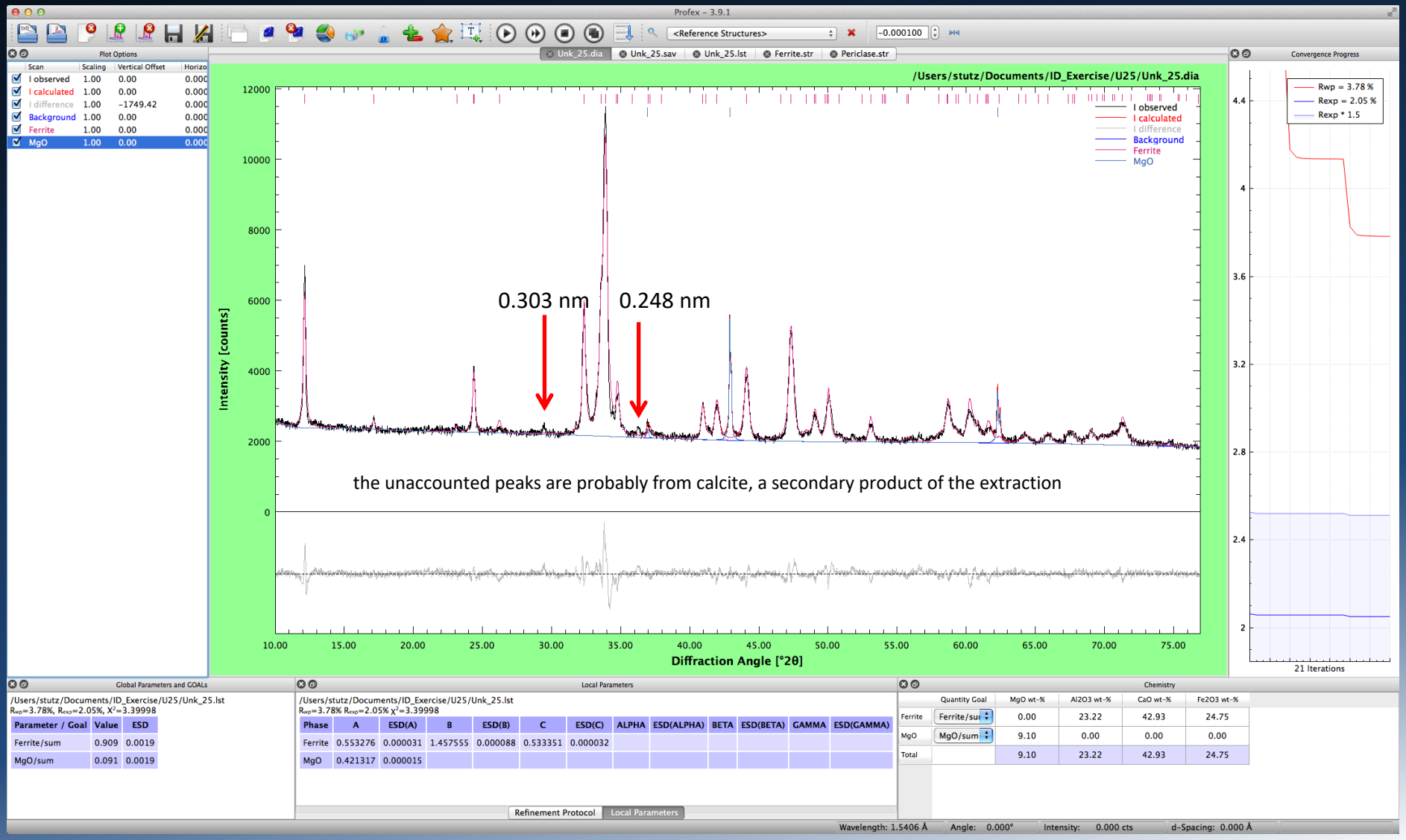
Since ferrite can exhibit a tabular habit it is possible it could be oriented, skewing the representative orientation ideal and so, the relative peak intensities. Change the zero to 2 and later 4 if necessary after the variable SPHAR and repeat the refinement to see if it improves. Profex runs the refinement and introduces the orientation correction at the last stages, but only if the phase fraction is above a minimum to make it practical.



Check Parameters



Look for additional phases



d-Spacing	Two-Theta	Phase
7.627	**11.593**	**gypsum (100)**
7.249	**12.200**	**C4AF (45)**
5.997	**14.759**	**bassanite (80)**
5.970	14.827	triclinic C3S (12)
5.953	14.869	triclinic C3S (12)
5.927	14.935	triclinic C3S (12)
5.927	14.935	mono. C3S (12)
5.610	15.784	γC2S (19)
5.107	17.350	C3Ao (10)
4.917	18.026	aphthitalite (10)
4.659	19.033	thenardite (71)
4.640	19.112	α´C2S (30)
4.316	20.561	γC2S (45)
4.284	**20.717**	**gypsum (100)**
4.253	20.869	langbeinite (30)
4.235	**20.959**	**C3Ac (6)**
4.222	21.024	langbeinite (25)
4.188	21.197	langbeinite (16)
4.175	**21.264**	**arcanite (28)**
4.158	**21.352**	**arcanite (23)**
4.091	21.706	aphthitalite (30)
4.079	**21.770**	**C3Ac (12)**
4.059	21.879	γ C2S (20)
3.900	22.783	αC2S (20)
3.886	22.866	triclinic C3S (10)
3.855	23.052	calcite (9)
3.838	23.156	thenardite (17)
3.817	23.285	γC2S (509)
3.810	23.328	α´C2S (30)
3.799	**23.397**	**gypsum (17)**
3.764	23.617	γC2S (119)
3.744	23.745	arcanite (18)
3.670	**24.231**	**aphthitalite (20)**
3.653	**24.346**	**C4AF (16)**
3.497	**25.450**	**anhydrite (100)**
3.468	**25.666**	**bassanite (40)**
3.462	25.711	langbeinite (12)
3.424	26.002	C3Ao (11)
3.385	26.307	arcanite (13)
3.379	26.354	γC2S (25)
3.370	26.426	α´C2S (30)
3.313	**26.889**	**langbeinite (95)**
3.271	**27.241**	**langbeinite (80)**
3.263	**27.309**	**langbeinite (80)**
3.225	**27.637**	**langbeinite (100)**
3.180	28.036	thenardite (52)
3.153	28.281	langbeinite (18)
3.114	28.643	langbeinite (18)
3.077	28.995	thenardite (55)
3.065	**29.111**	**gypsum (75)**
3.056	29.198	triclinic C3S (60)
3.045	29.306	bassanite (10)
3.038	29.375	M1 C3S (50)
3.036	29.400	calcite (100)
3.036	**29.395**	**mono C3S (40)**
3.034	**29.415**	**m1 C3S (50)**
3.025	**29.504**	**triclinic C3S (65)**
3.025	**29.504**	**mono C3S (75)**
3.011	29.645	γC2S (80)
3.002	**29.736**	**bassanite (80)**
3.000	**29.756**	**arcanite (77)**
2.985	**29.909**	**triclinic C3S (25)**
2.974	**30.022**	**triclinic C3S (18)**
2.972	30.043	M1 C3S (20)
2.968	30.084	mono C3S (12)
2.968	30.084	M1 C3S (20)
2.965	**30.115**	**triclinic C3S (20)**
2.961	**30.157**	**mono C3S (25)**
2.940	**30.378**	**aphthitalite (75)**
2.902	**30.785**	**arcanite (100)**
2.894	30.872	γC2S (25)
2.886	**30.960**	**arcanite (53)**
2.880	**31.026**	**langbeinite (18)**
2.876	**31.070**	**βC2S (21)**
2.872	31.115	gypsum (45)
2.870	31.137	α´C2S (30)
2.850	31.361	anhydrite (29)
2.843	31.440	calcite (2)
2.838	**31.497**	**aphthitalite (100)**
2.813	31.784	βC2S (22)
2.813	31.784	bassanite (100)
2.810	31.819	αC2S (80)
2.790	32.053	βC2S (97)
2.788	32.077	triclinic C3S (100)
2.788	32.077	gypsum (10)
2.786	32.101	langbeinite (45)
2.784	**32.124**	**C4AF (25)**
2.784	32.124	thenardite (100)
2.782	32.148	βC2S (100)
2.776	32.220	free lime (36)
2.775	32.231	M1 C3S (100)
2.775	32.231	langbeinite (50)
2.773	32.255	mono C3S (85)
2.767	32.327	triclinic C3S (70)
2.754	32.484	triclinic C3S (65)
2.750	32.533	γC2S (70)
2.750	32.533	langbeinite (45)
2.747	32.569	mono C3S (45)
2.747	32.569	M1 C3S (40)
2.745	32.593	βC2S (83)
2.743	32.618	M1 C3S (60)
2.743	32.618	langbeinite (45)
2.740	32.655	α´C2S (100)
2.737	32.691	mono C3S (75)

d-Spacing	Two-Theta	Phase
2.736	32.704	triclinic C3S (60)
2.717	32.939	βC2S (30)
2.714	**32.976**	**C3Ao (65)**
2.714	32.976	bassanite (10)
2.710	**33.026**	**αC2S (100)**
2.698	**33.178**	**C3Ac (100)**
2.692	**33.254**	**C3Ao (100)**
2.684	33.356	gypsum (35)
2.680	33.407	α´C2S (75)
2.673	33.497	C4AF (35)
2.647	33.836	thenardite (52)
2.644	**33.875**	**C4AF (100)**
2.618	34.222	triclinic C3S (60)
2.612	34.303	triclinic C3S (90)
2.610	**34.330**	**βC2S (42)**
2.607	34.371	M1 C3S (70)
2.605	34.398	M1 C3S (80)
2.603	34.425	mono C3S (100)
2.590	34.604	γC2S (14)
2.576	34.798	C4AF(17)
2.517	35.640	arcanite (13)
2.514	35.684	γC2S (25)
2.499	35.906	arcanite (15)
2.495	35.968	calcite (15)
2.494	35.980	gypsum (11)
2.458	36.526	triclinic C3S (12)
2.458	36.526	aphthitalite (10)
2.455	36.572	γC2S (17)
2.448	36.680	βC2S (12)
2.442	36.774	aphthitalite (16)
2.430	36.962	periclase (10)
2.422	37.088	arcanite (25)
2.409	37.296	βC2S (13)
2.405	**37.360**	**free lime (100)**
2.402	37.408	βC2S (18)
2.385	37.685	arcanite (13)
2.374	37.866	arcanite (17)
2.360	38.100	α´C2S (30)
2.339	38.455	triclinic C3S (15)
2.329	38.627	triclinic C3S (20)
2.329	38.627	thenardite (25)
2.329	38.627	aphthitalite (14)
2.328	38.644	anhydrite (20
2.325	38.696	γC2S (10)
2.323	38.725	M1 C3S (10)
2.319	38.800	M1 C3S (20)
2.315	38.870	triclinic C3S (25)
2.315	38.870	mono C3S (20)
2.285	39.408	calcite (20)
2.280	39.491	βC2S (22)
2.280	39.491	triclinic C3S (11)
2.270	39.672	α´C2S (10)
2.268	39.709	bassanite (10)
2.230	40.415	α´C2S (30)
2.220	40.605	αC2S (40)
2.218	40.643	gypsum (15)
2.209	40.816	anhydrite (20)
2.205	40.893	C3Ao (20)
2.205	40.893	arcanite (14)
2.203	40.932	C3Ac (10)
2.196	41.068	langbeinite (12)
2.195	41.088	triclinic C3S (75)
2.189	41.206	βC2S (51)
2.184	41.304	M1 C3S (40)
2.181	41.364	mono C3S (60)
2.180	41.383	α´C2S (30)
2.179	41.403	triclinic C3S (17)
2.179	41.403	M1 C3S (40)
2.171	41.563	triclinic C3S (11)
2.169	41.603	M1 C3S (10)
2.166	41.663	M1 C3S (10)
2.164	41.704	βC2S (13)
2.164	41.704	mono C3S (15)
2.163	41.724	triclinic C3S (11)
2.162	41.744	M1 C3S (10)
2.136	42.276	bassanite (20)
2.109	42.844	langbeinite (18)
2.105	**42.930**	**periclase (100)**
2.094	43.157	calcite (15)
2.093	43.188	langbeinite (20)
2.088	43.297	arcanite (25)
2.085	43.362	gypsum (25)
2.082	43.428	arcanite (25)
2.073	43.626	gypsum (15)
2.051	44.118	C4AF(35)
2.050	44.141	βC2S (14)
2.041	44.346	aphthitalite (45)
2.036	44.461	langbeinite (14)
2.026	44.692	βC2S (15)
2.024	44.738	γC2S (13)
2.020	44.832	α´C2S (30)
2.019	44.855	βC2S (15)
2.017	44.902	langbeinite (20)
2.009	45.091	langbeinite (14)
1.994	45.449	triclinic C3S (10)
1.982	45.740	M1 C3S (10)
1.981	45.764	βC2S (20)
1.973	45.960	mono C3S (10)
1.940	46.788	αc2s (60)
1.937	46.865	M1 C3S (10)
1.933	46.968	M1 C3S (10)
1.930	47.045	α´C2S (30)
1.930	47.045	mono C3S (13)
1.928	47.097	C4AF(35)