Fullprof refinement of commensurate structure from TOF data (magnetic space group approach)

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Magnetic Structure Determination from Neutron Diffraction Data (MagStr), October 3-7, 2022

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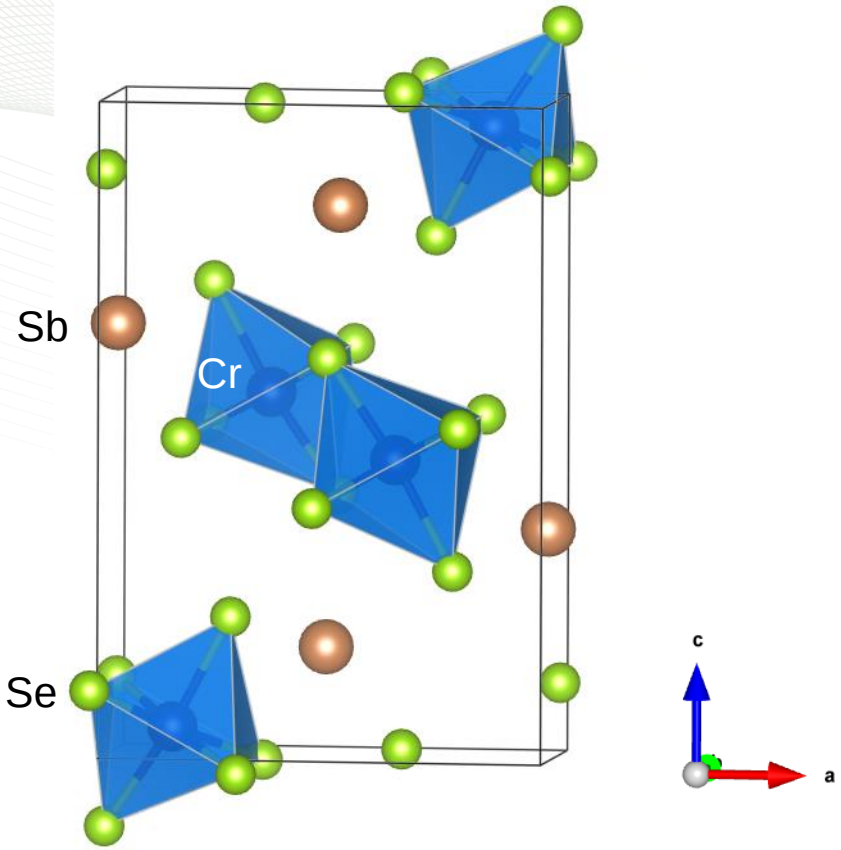
OAK RIDGE National Laboratory | HIGH FLUX ISOTOPE REACTOR | SPALLATION NEUTRON SOURCE

$CrSbSe_3$



- Orthorhombic structure: *Pnma (No. 62)*

a= 9.143086, b= 3.784552, c=13.416915;

- *Octahedra $CrSe_6$;*
- *Magnetic transition 75 K:*

Files provided for this tutorial

- Time-of-flight diffraction data at POWGEN:
  PG3_42702-2_300K.dat
  PG3_42704-2_10K.dat
- Instrumental resolution file:
  PG2018B_HighRes_60Hz_b2_Ddep.irf

- cif file for crystal structure
  $CrSbSe_3$.cif

- Final PCR files
  PG3_42702-2_300K.pcr
  CrSbSe3_N_M_10K.pcr (magnetic space group method)

- Mcif file:
  62.447.mcif

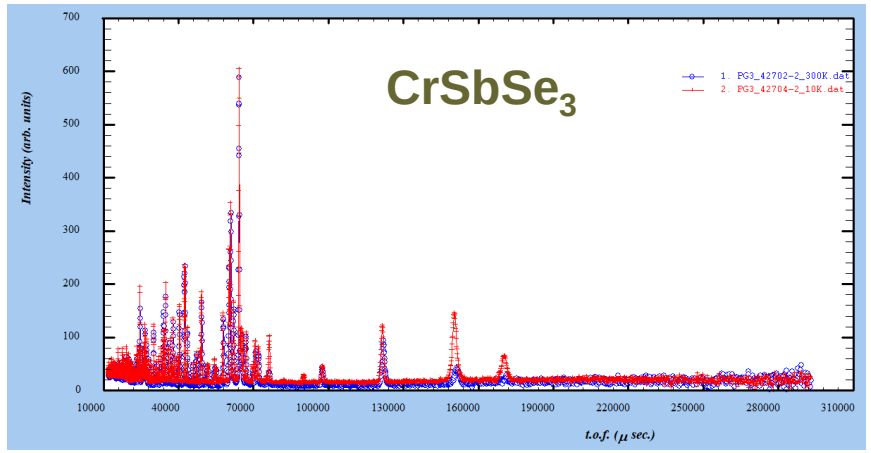
- Supporting information
  FullProf_QZhang.PDF (step-by-step instructions)
  jacs.1c09607.PDF

Time-of-flight powder diffractometer POWGEN

- **High resolution powder diffractometer** *to study crystal, magnetic and local structure of polycrystalline materials*

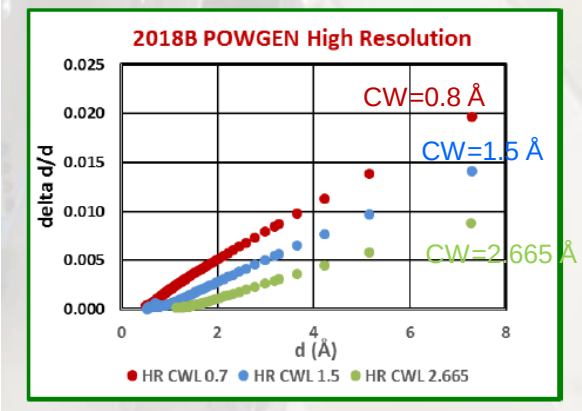
Current Instrument Capabilities (neutron band ~ 1 Å)

Freq (Hz)	WL center	WL min	WL max	dmin	dmax	Qmin	Qmax	Bank
60	0.533	0.15	1.066	0.075	7.50	0.82	83.45	0
60	0.800	0.27	1.333	0.134	8.00	0.76	46.88	1
60	1.500	0.97	2.033	0.485	13.00	0.48	12.95	2
60	2.665	2.13	3.198	1.070	21.00	0.30	5.87	3
60	4.797	4.26	5.33	2.140	38.00	0.17	2.94	4



Intensity vs TOF (NOT 2theta!)

Resolution: *best resolution Δd/d: 0.8x10^{-3}*



Dependent on wavelengths and *d* (or 2theta)

PG3_42702-2_300K.dat

```
XYDATA
# File generated by Mantid, Instrument POWGEN
# The X-axis unit is: Time-of-flight, The Y-axis unit
# Data for spectra :2
# Spectrum 1
# Time-of-flight          Y              E
    14300.17204      28.15968284     0.41506355
    14311.60584      26.26662343     0.40565232
    14323.04879      26.17181040     0.40476613
    14334.50089      25.28546181     0.40012561
```

TOF to D spacing (FullProf):

TOF(microseconds) = Zero + Dtt1 * D + Dtt2 * D^2 + Dtt_loverD)/D

(D in Angstroms)

POWGEN Peak Profile

TOF profile has Convolution of back-to-back exponentials with pseudo-Voigt (a linear combination of Gaussian and Lorentzian)

6 refinable parameters: position, intensity, α, β, σ and γ Position:

TOF(microseconds) = Zero + Dtt1 * D + Dtt2 * D^2 + Dtt_loverD)/D

Exponentials: Alpha largely affects the sharpness of the leading TOF edge of each peak; larger values mean sharper front edges. Beta terms affect the trailing TOF edge in the same way.

$$\alpha = \alpha_0 + \frac{\alpha_1}{d} + \frac{\alpha_q}{\sqrt{d}} \text{ and } \beta = \beta_0 + \frac{\beta_1}{d^4} + \frac{\beta_q}{d^2}$$

Gaussian and Lorentzian width: Sig terms affect the Gaussian shape component of the peak profiles; larger values result in broader peaks. The coefficients describe the sig and Gamma values as follows:

$$\sigma^2 = {\sigma_0}^2 + {\sigma_1}^2 d^2 + {\sigma_2}^2 d^4 + \frac{{\sigma_q}^2}{d^2}$$

→ σ_qd GSASII used

$$\gamma = \gamma 1 * d + \gamma 2 * d^2 + \gamma 0$$

- All these coefficients *marked as green* were implemented in the **2021 versions** of *FullProf* by Juan rodriguez-Carvajal recently.
- In other TOF instruments and previous versions of FullProf, there lack Dtt_1overD, β_q and σ_q.

Formats of the instrumental resolution files

Traditional Instrumental resolution files for old version FullProf

(2011 B- 2021 A cycle)

```
Instrumental resolution function for POWGEN/SNS 2018_B cycle  ireso: 5
! To be used with function NPROF=9 in FullProf  (Res=5)
! ----------------------------------------------------  Bank 3 CWL = 1.500
!  Type of profile function: back-to-back expon * pseudo-Voigt
NPROF   9
!       Tof-min(us)     step      Tof-max(us)
TOFRG  11300.0000       5.0000   315000.0000
!        Dtt1            Dtt2        Zero
D2TOF  22589.89258      -3.55042   -15.12841
!     TOF-TWOTH of the bank
TWOTH    90.0
!       Sig-2      Sig-1      Sig-0
SIGMA   57.460      10.000       0.000
!       Gam-2      Gam-1      Gam-0
GAMMA   26.000    -29.576       9.000
!          alph0       beta0       alph1       beta1
ALFBE      0.000000    0.100880    0.128660    0.003960
END
```

TOF region and step

TOF to D

Gaussian function

Lorentzian function

BB Exponentials

Listed the global coefficients without Dtt_1overD, β_q and σ_q for old version FullProf.

New Instrumental resolution files for new version FullProf

(2021 B cycle-future)

```
 Instrumental Resolution Parameters for TOF (numerical look-up table) for POWGEN/SNS 2018 B cycle
! To be used with functions NPROF=9 in FullProf (Res=5)
! ----------------------------------------------------  Bank 1
!  Type of profile function: back-to-back expon * pseudo-Voigt
NPROF   9
!       Tof-min(us)       step      Tof-max(us)
TOFRG   11308.69531       5  282062.50000
!             Dtt1              Dtt2       Dtt_1overD         Zero
D2TOF     22589.89258        -3.55042         0.24175       -15.12841
!     TOF-TWOTH of the bank
TWOTH      90.000
!       d-spacing       Sigma^2         Gamma           Alpha           Beta            Shift    for pattern # 1
LIST_SIG_GAM_ALF_BET_SHIFT  233
         0.44348         1.21809         2.85332         0.39237         0.10474         0.00000
         0.44515         1.21788         2.84730         0.39204         0.10578         0.00000
         ...
         0.89199         7.70333         3.42920         0.14657         0.10563         0.00000
         ...
         1.51101       757.12213        18.55641         0.11664         0.15918         0.00000
         1.54497       844.20258        19.59661         0.11365         0.15412         0.00000
         ...
         2.56205      7234.60986        59.75310         0.06182         0.08379         0.00000
         ...
         5.12410    116786.14844       252.91258         0.02469         0.05172         0.00000
         7.24657    462492.40625       465.01639         0.01463         0.04323         0.00000
END
```

TOF region and step

TOF to D

numerical coefficients

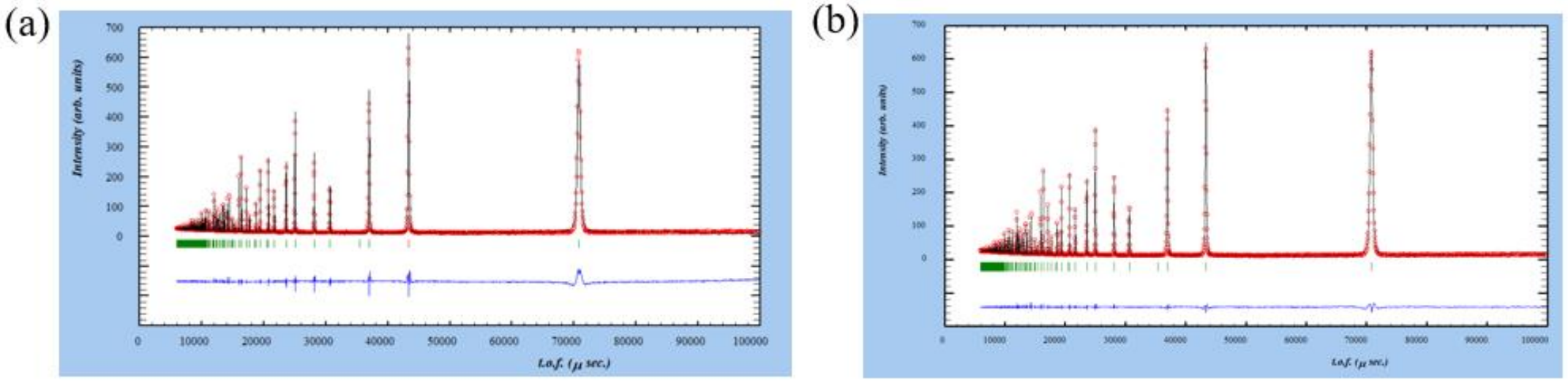
Provides d-dependent numerical coefficients for new 2021 versions of FullProf (used in this tutorial)

Allows a more accurate determination of instrumental resolution.

Comparison of the refinement quality using old and 2021 versions of FullProf and IRFs

Old versions of FullProf and old IRFs until 2020

2021 versions of FullProf and new IRFs



Same Si data at POWGEN

- New 2021 versions of FullProf and IRFs improved the refinement quality
- Old PCRs can be read automatically by the new 2021 versions of FullProf and converted to the new format PCR files

Recommend using the 2021 or newer version FullProf to refine POWGEN data for getting an improved refinement on the peakshape

Exercise steps

I. Import the cif file and create a PCR file for a single datafile at 300 K (T>Tm);

II. Refine the data to get accurate structural parameters at 300 K;

III. Save the PCR file at 300 K as a new PCR to refine the structural parameters at 10 K (T<Tm).

IV. Identify magnetic peaks/contributions and determine the propagation vector.

V. Use Bilbao Crystallographic Server to test magnetic space groups;

VI. Convert one mcif file to PCR file;

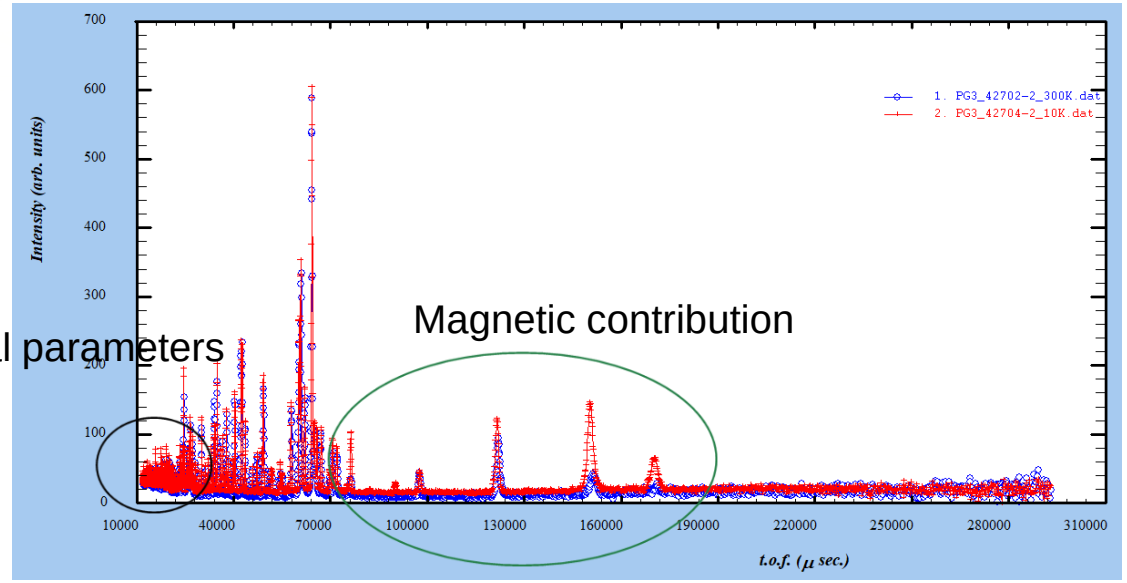
VII. Refine both nuclear and magnetic peaks at 10 K to obtain the magnetic structure and ordered moment.

VIII. Check if small Mz (AFM component) is real.

IX. Display the magnetic structure using FpStudio and Vesta.

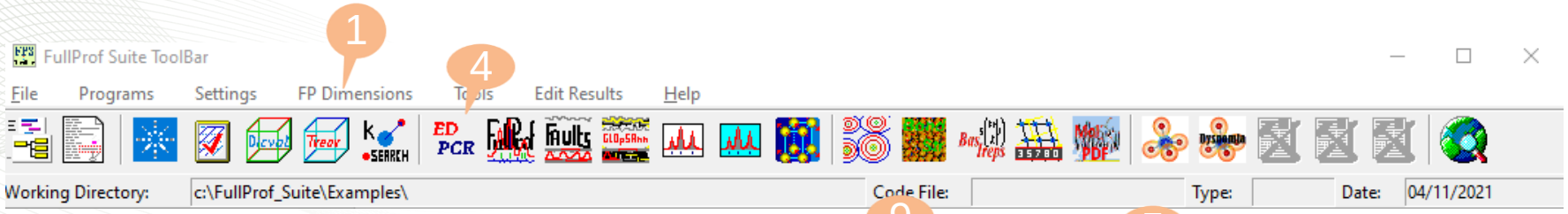
Comparison of the POWGEN data between 300 K and 10 K

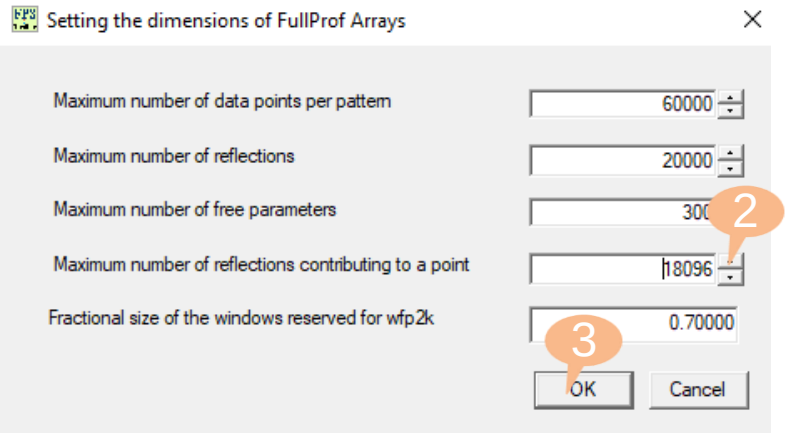
$CrSbSe_3$

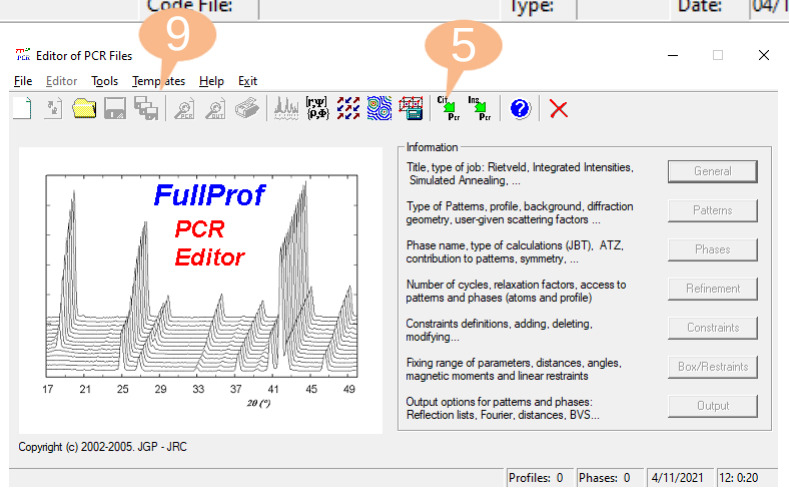


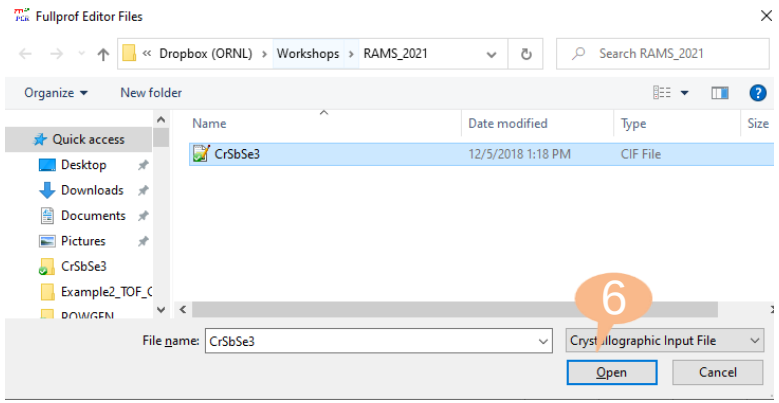
- Some magnetic peaks sit on the nuclear peaks. *Tricky sometimes!*
- At 10 K, the intensities at high *d* part and low *d* part both increased: different origins!

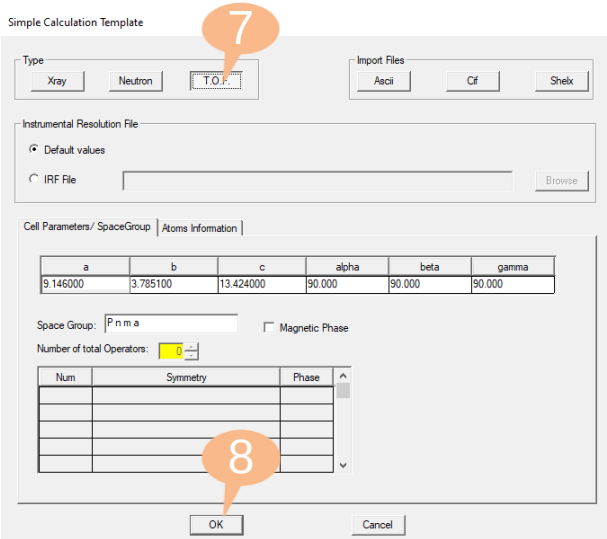
I. Import the cif file and create a PCR file for a single datafile at 300 K (T>Tm)

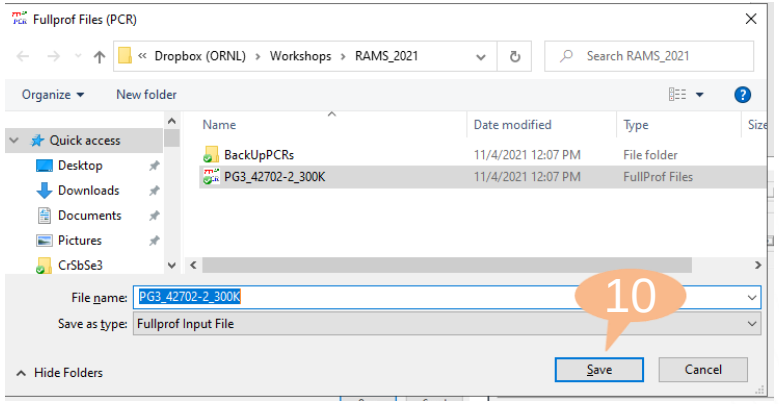












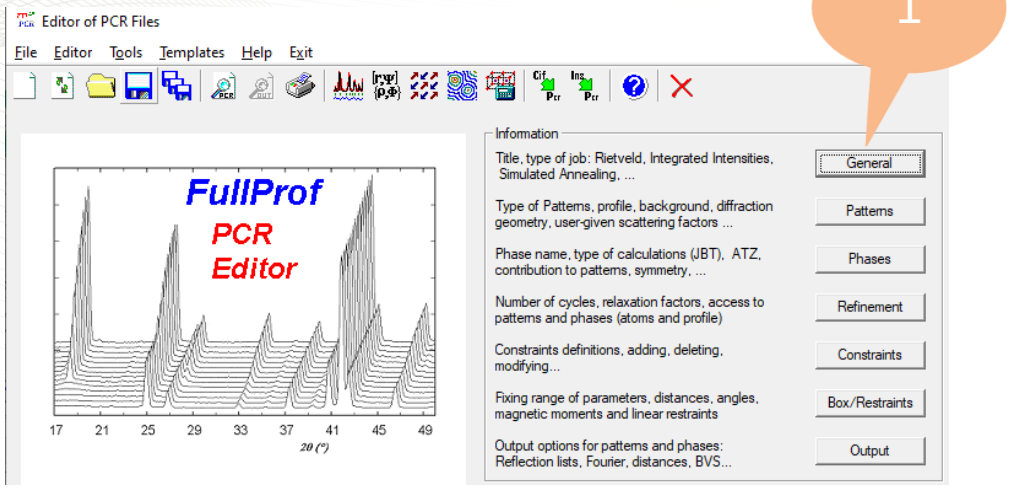
Important: check occ=
site multip/general multip

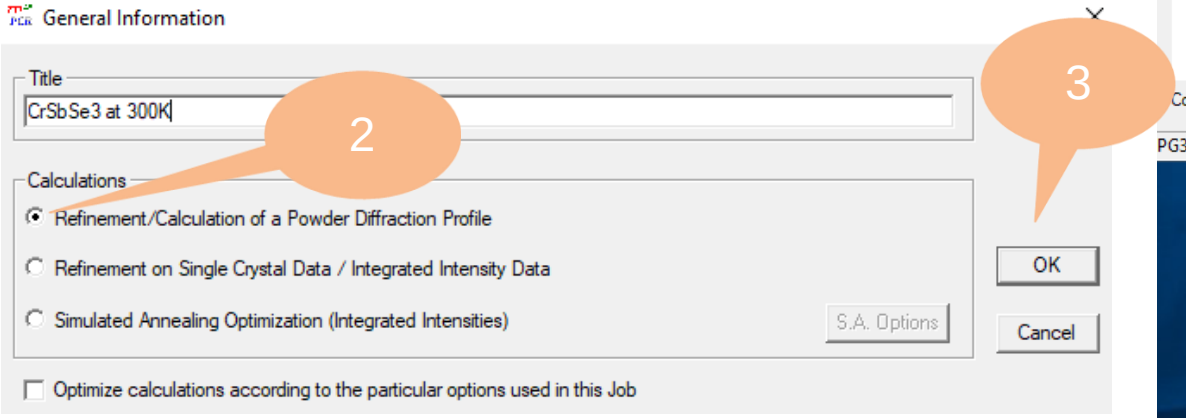
```
P n m a                    <--Space group symbol
!Atom   Typ      X        Y        Z      Biso      Occ
Sb      Sb    0.02950  0.25000  0.65786  1.00000  0.50000
                 0.00     0.00     0.00     0.00     0.00
Cr      Cr    0.15490  0.25000  0.04460  1.00000  0.50000
                 0.00     0.00     0.00     0.00     0.00
Se3     Se    0.17180  0.25000  0.48450  1.00000  0.50000
                 0.00     0.00     0.00     0.00     0.00
Se2     Se    0.28480  0.25000  0.21280  1.00000  0.50000
                 0.00     0.00     0.00     0.00     0.00
Se1     Se    0.50190  0.25000  0.60870  1.00000  0.50000
                 0.00     0.00     0.00     0.00     0.00
```

Sb: 4c; General mulp: 8;

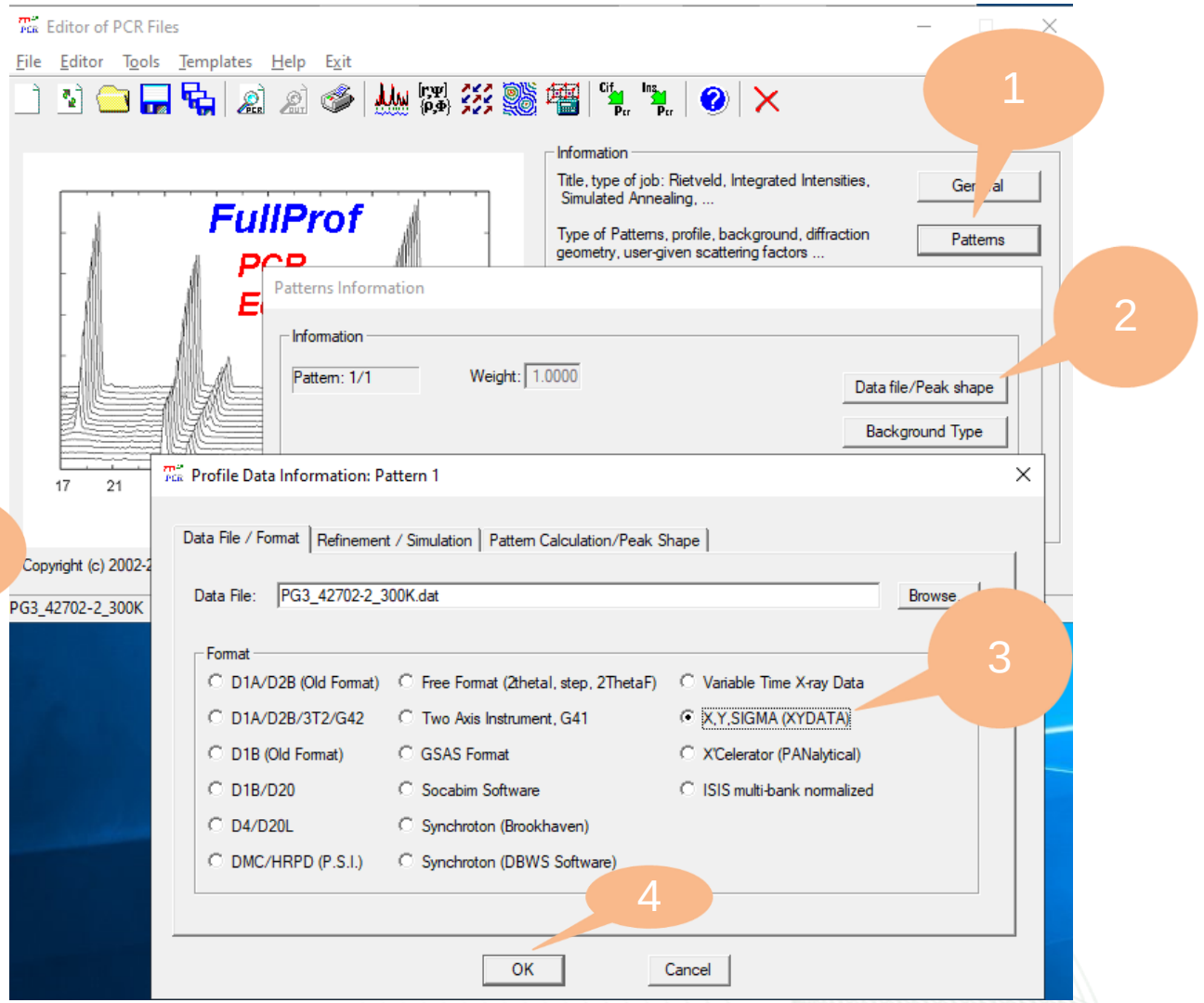
II. Refine the data to get accurate structural parameters at 300 K

General tab



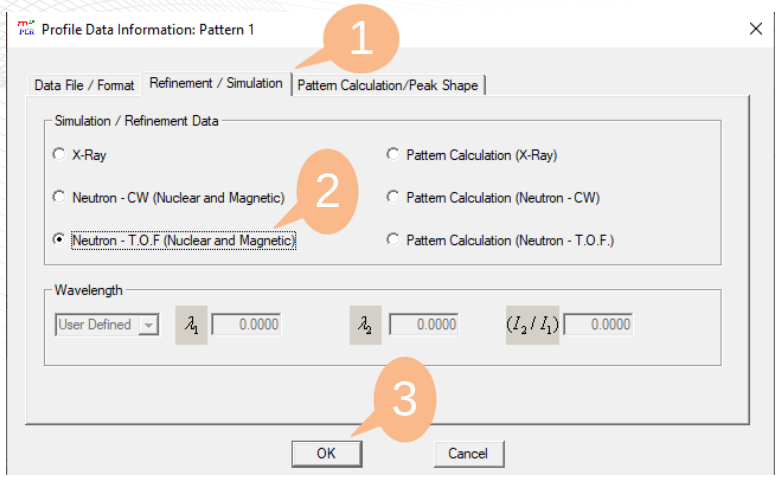


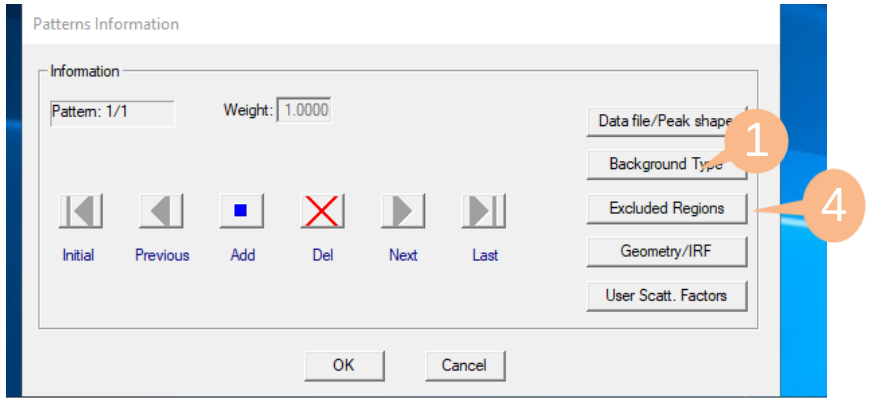
Pattern-Datafile/Peak shape tab

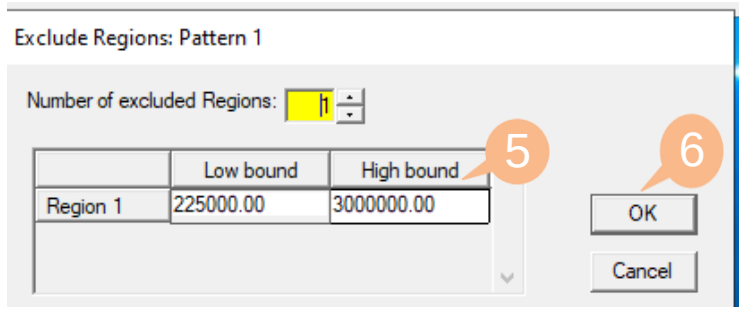


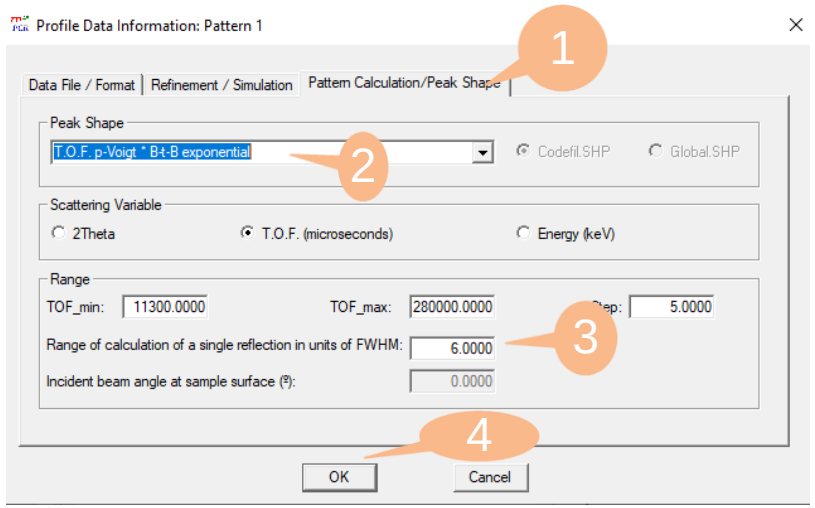
Pattern-Background Type

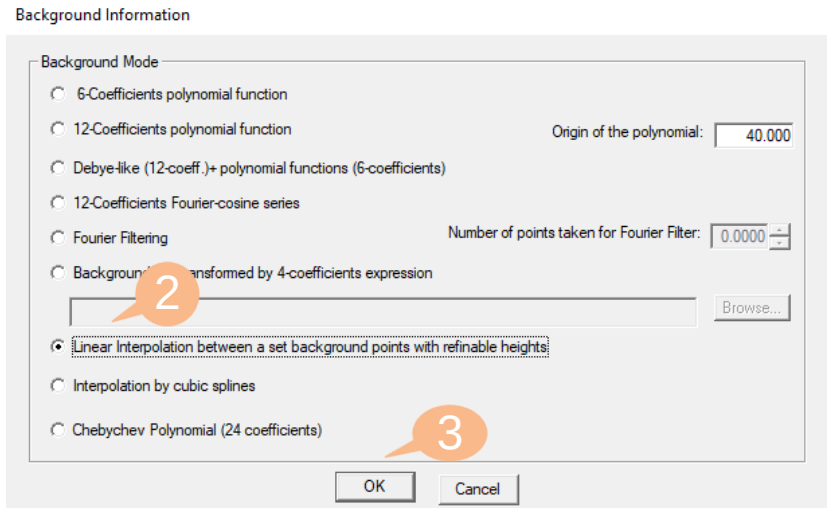
Pattern-Excluded Regions

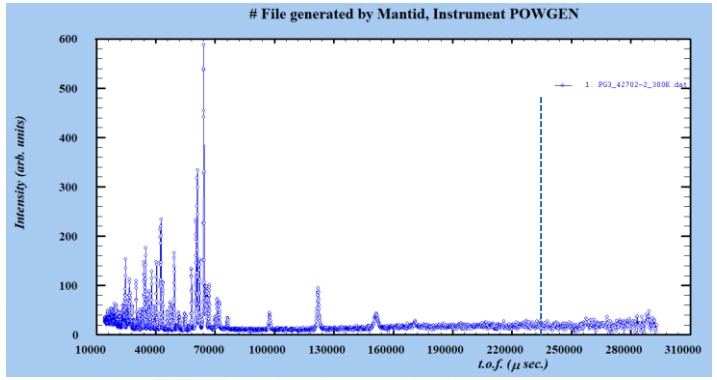




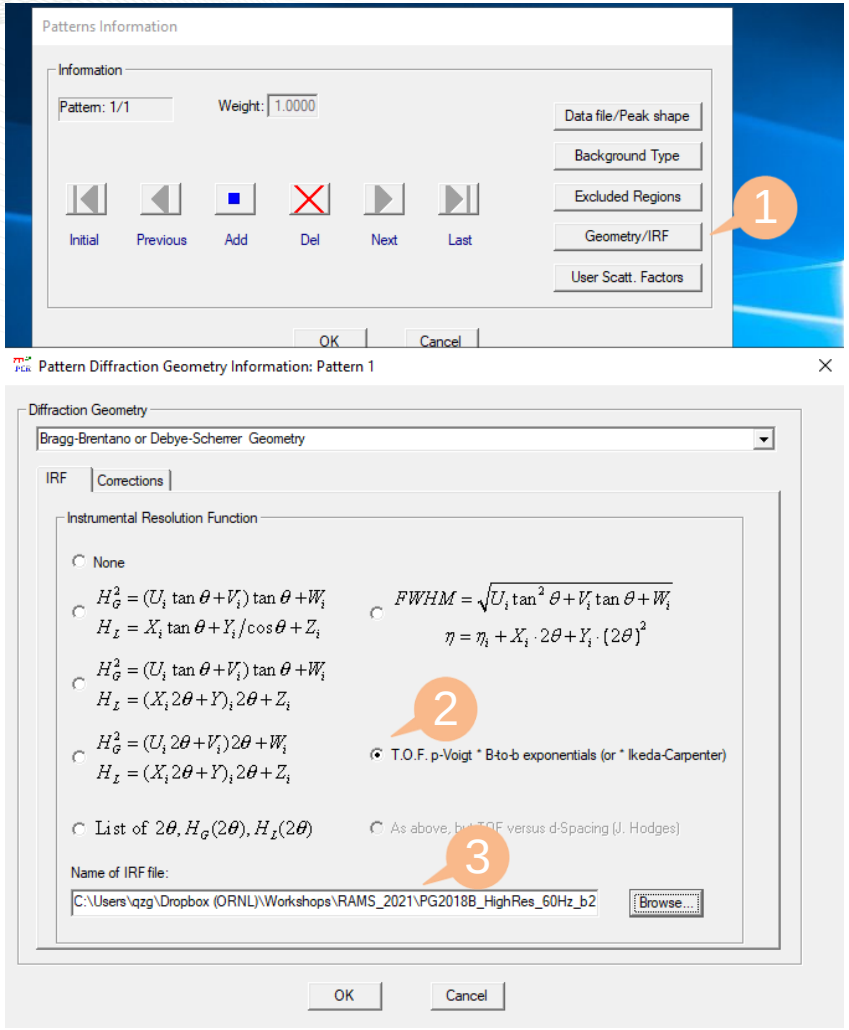




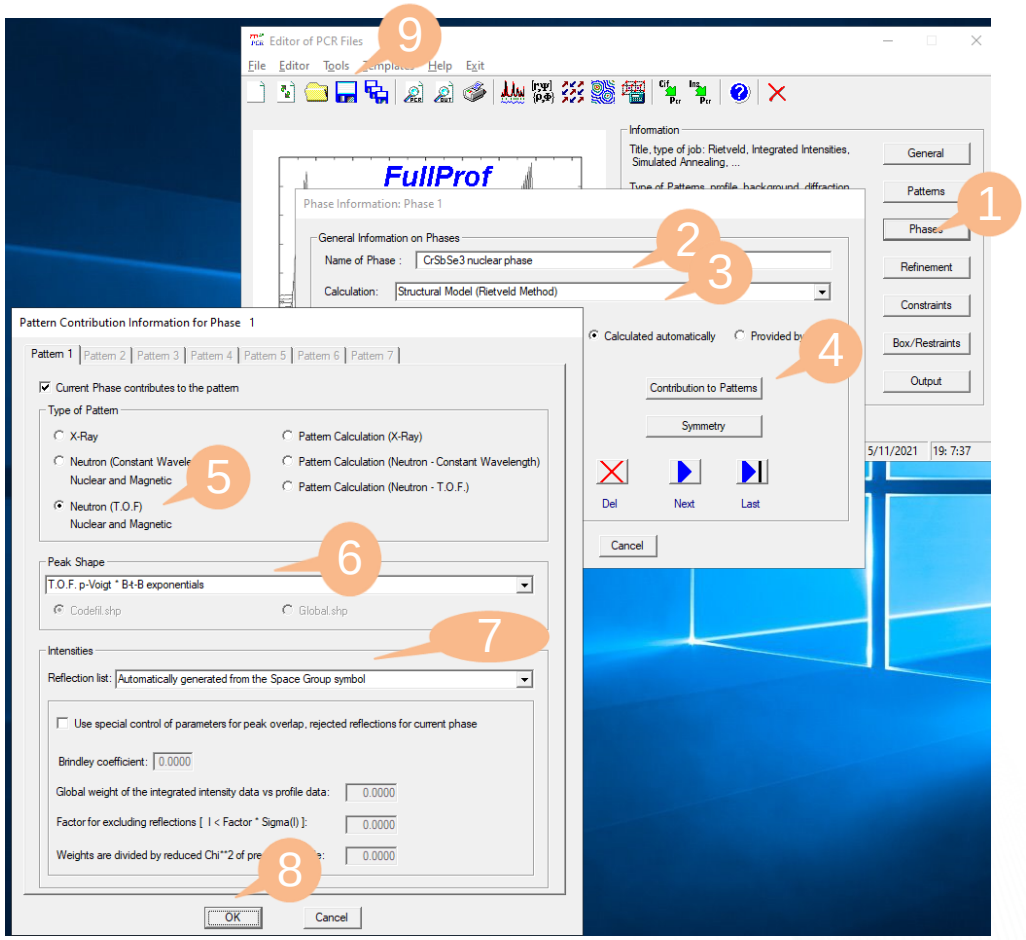




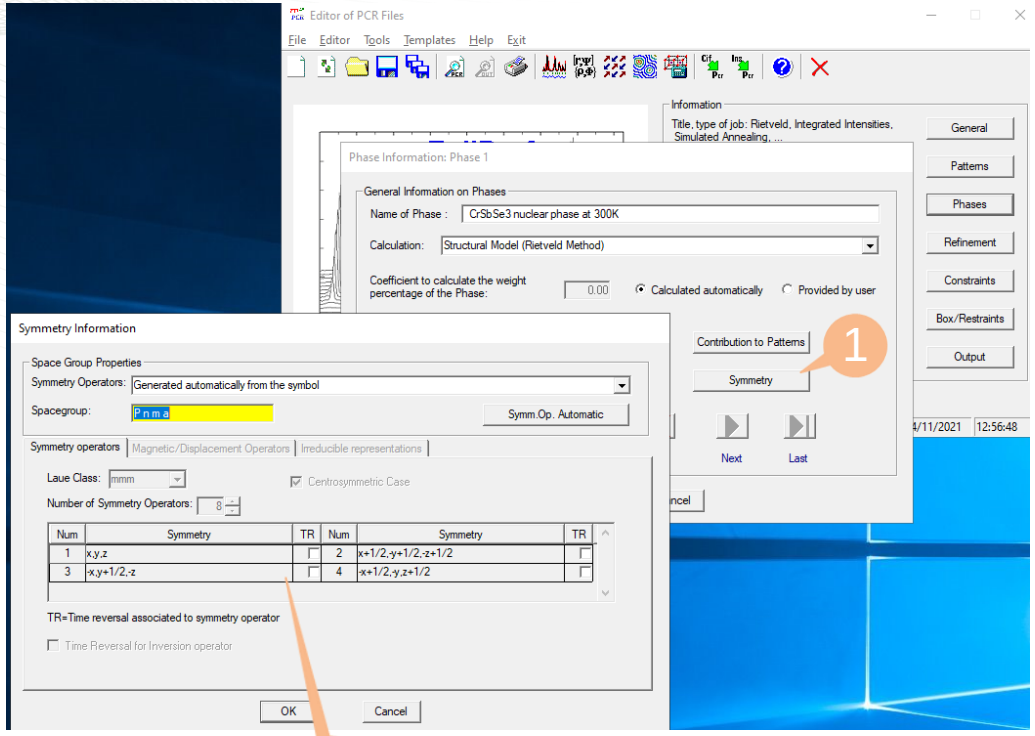
Pattern-Geometry/IRF tab



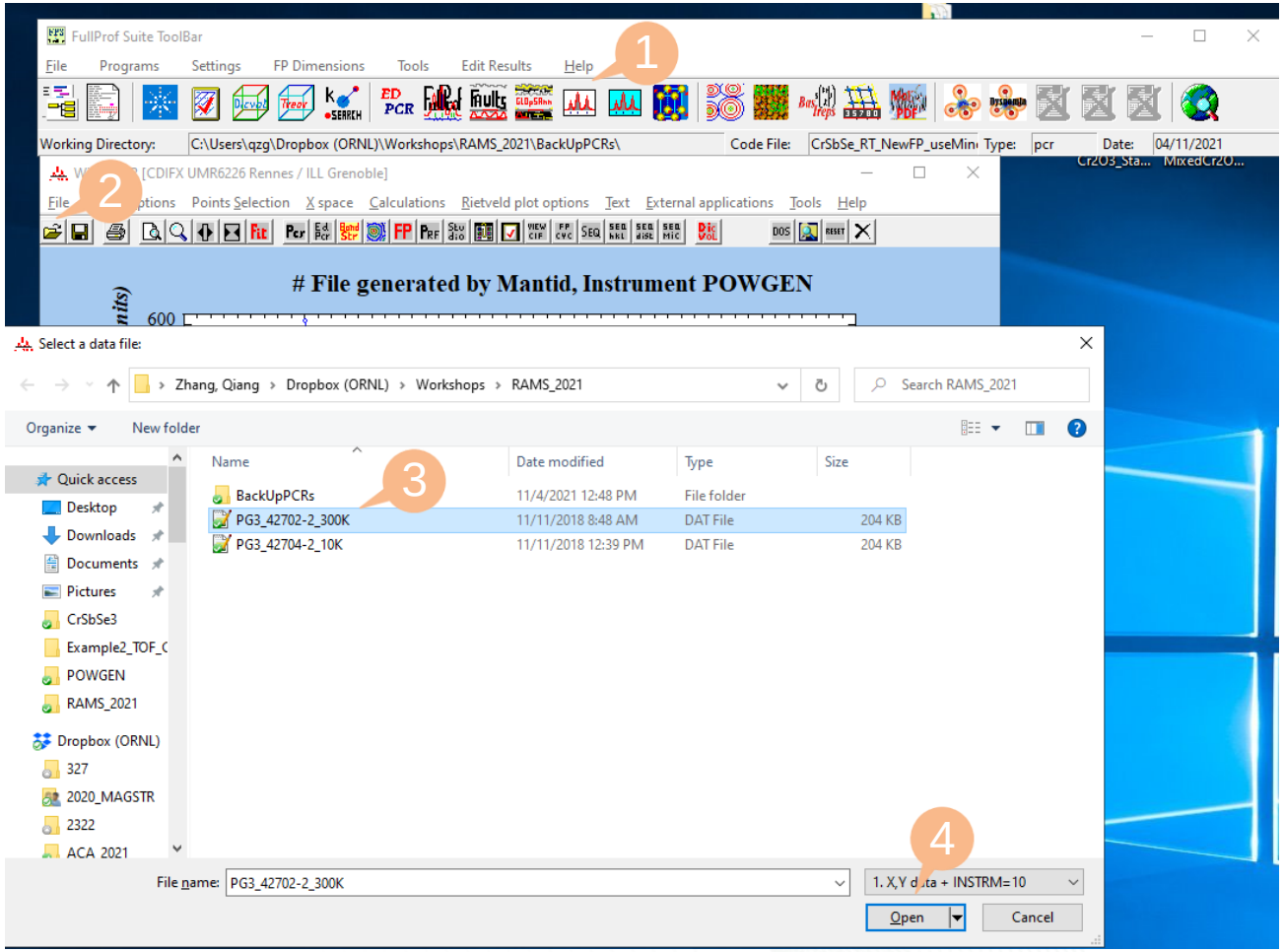
Phase-tab

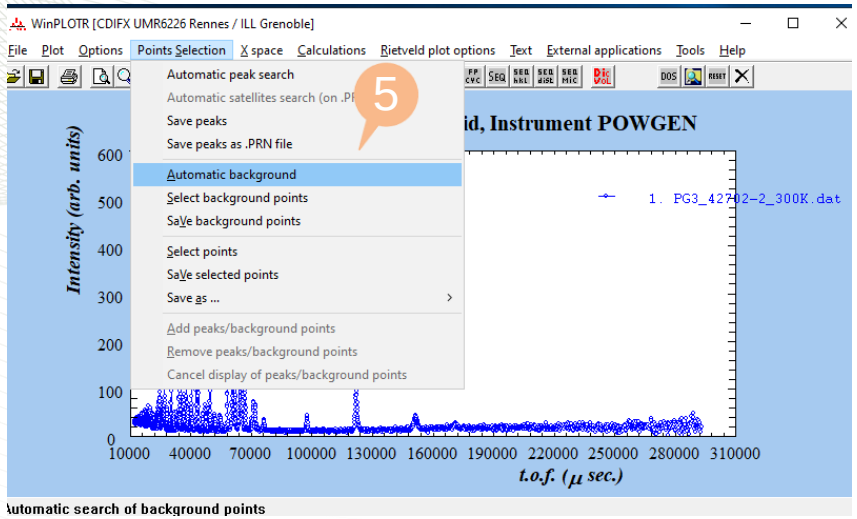


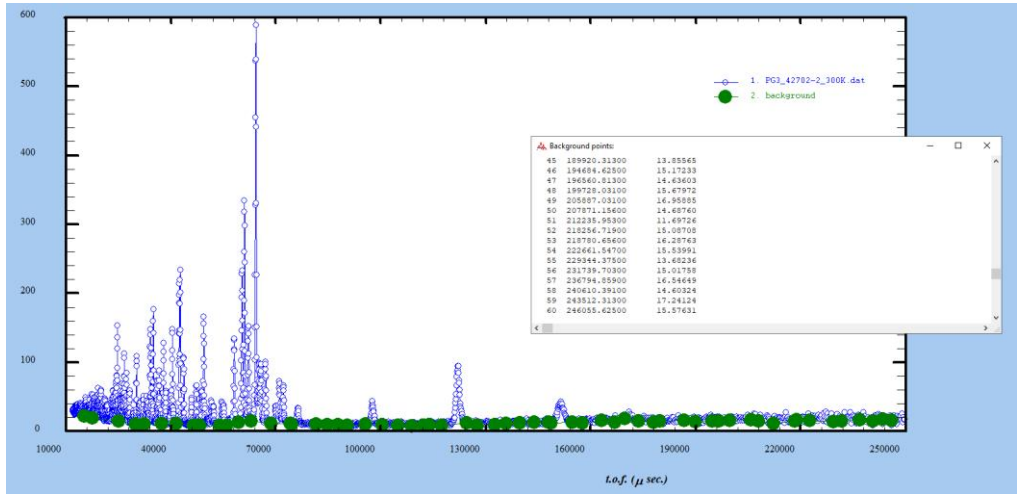
Use Winplotr to get the BG of the data

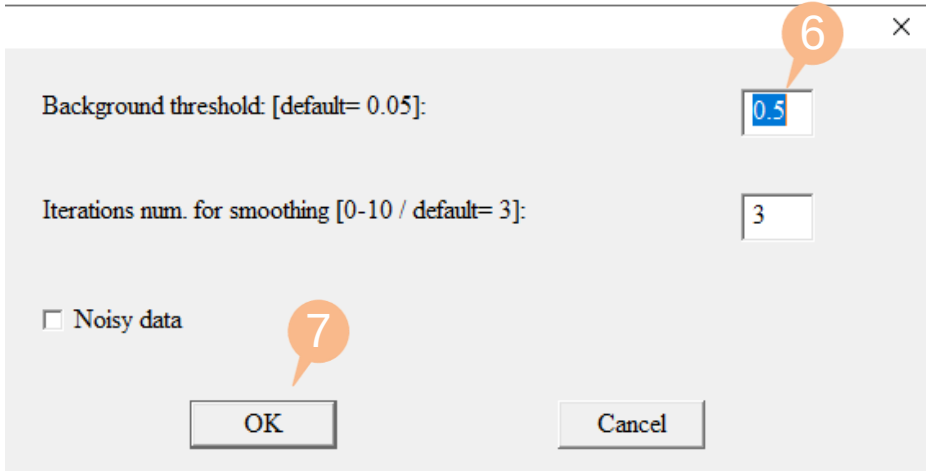


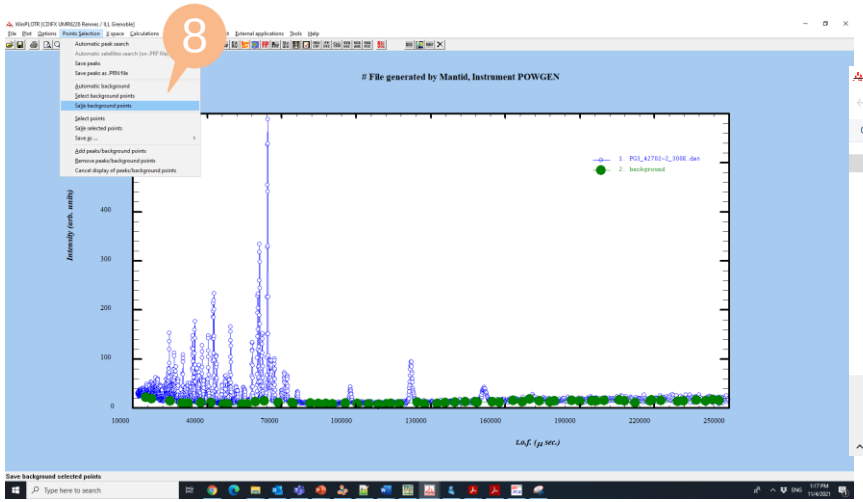
Verify the correct symmetry information

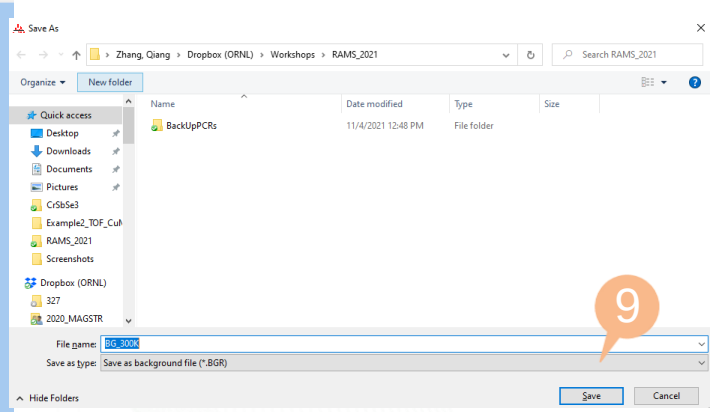


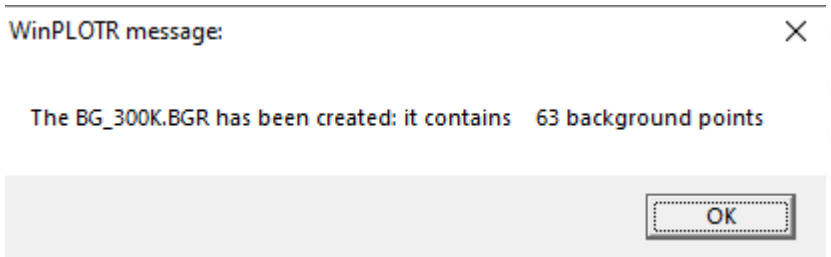

WinPLOTR [CDIFX UMR6226 Rennes / ILL Grenoble]
File Plot Options Points Selection X space Calculations Rietveld plot options Text External applications Tools Help
Automatic peak search
Automatic satellites search (on .PR
Save peaks
Save peaks as .PRN file
Automatic background
Select background points
SaVe background points
Select points
SaVe selected points
Save as ...
Add peaks/background points
Remove peaks/background points
Cancel display of peaks/background points
5
id, Instrument POWGEN
1. PG3_42702-2_300K.dat
Intensity (arb. units)
t.o.f. (μ sec.)
utomatic search of background points


1. PG3_42702-2_300K.dat
2. background
Background points:
t.o.f. (μ sec.)

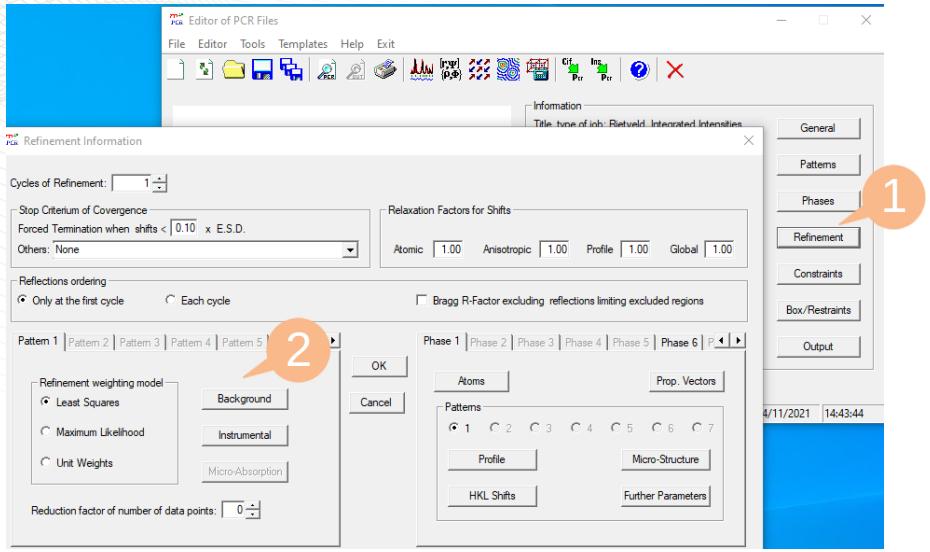

6
Background threshold: [default= 0.05]:
0.5
Iterations num. for smoothing [0-10 / default= 3]:
3
Noisy data
7
OK
Cancel

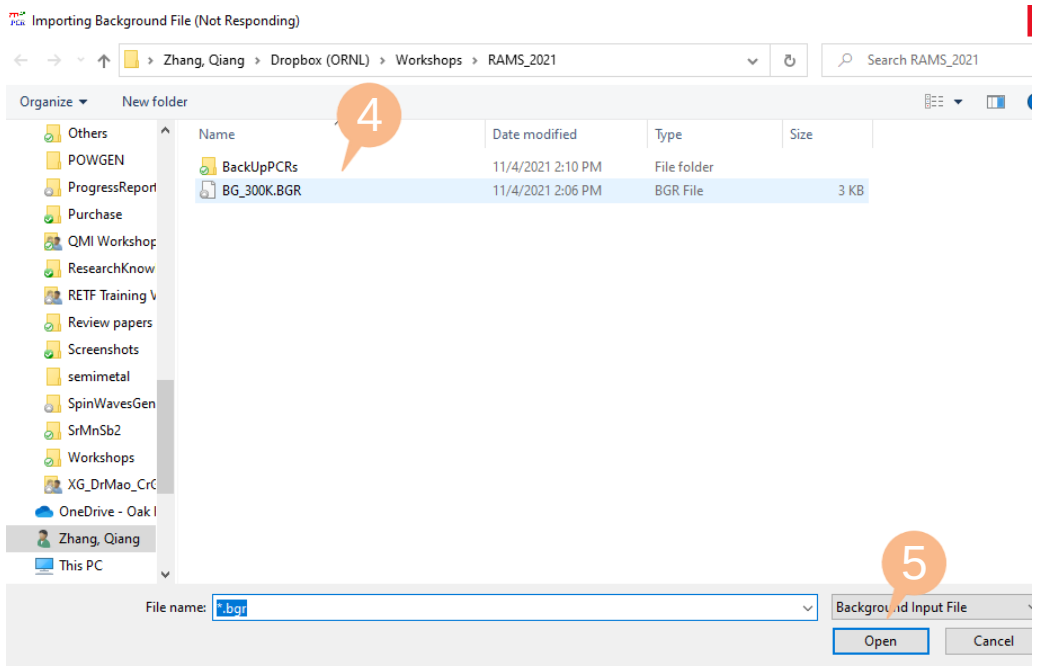

8
File generated by Mantid, Instrument POWGEN
1. PG3_42702-2_300K.dat
2. background
Intensity (arb. units)
t.o.f. (μ sec.)

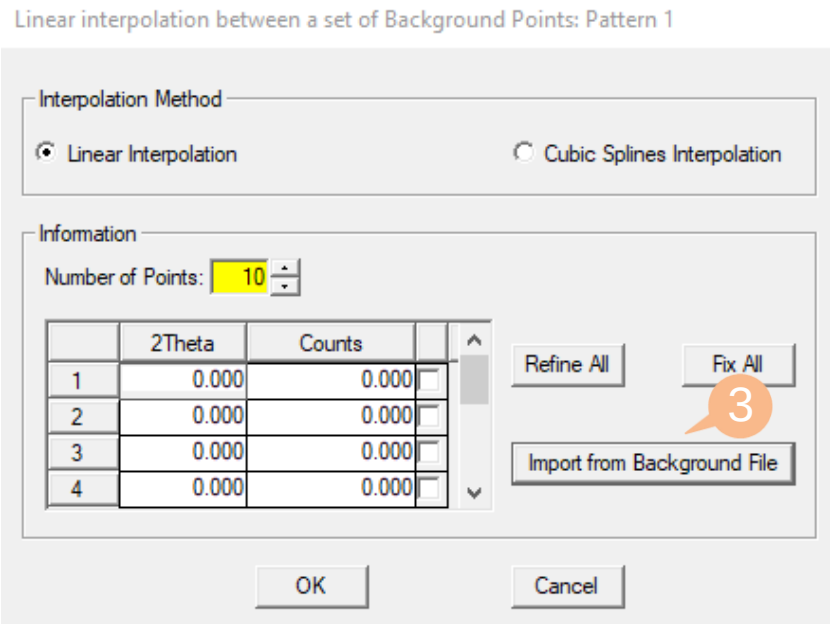

Save As
Zhang, Qiang › Dropbox (ORNL) › Workshops › RAMS_2021
Organize
New folder
Quick access
Desktop
Downloads
Documents
Pictures
CrSbSe3
Example2_TOF_Cuh
RAMS_2021
Screenshots
Dropbox (ORNL)
327
2020_MAGSTR
Name
Date modified
Type
Size
BackUpPCRs
11/4/2021 12:48 PM
File folder
9
File name:
BG_300K
Save as type:
Save as background file (*.BGR)
Hide Folders
Save
Cancel

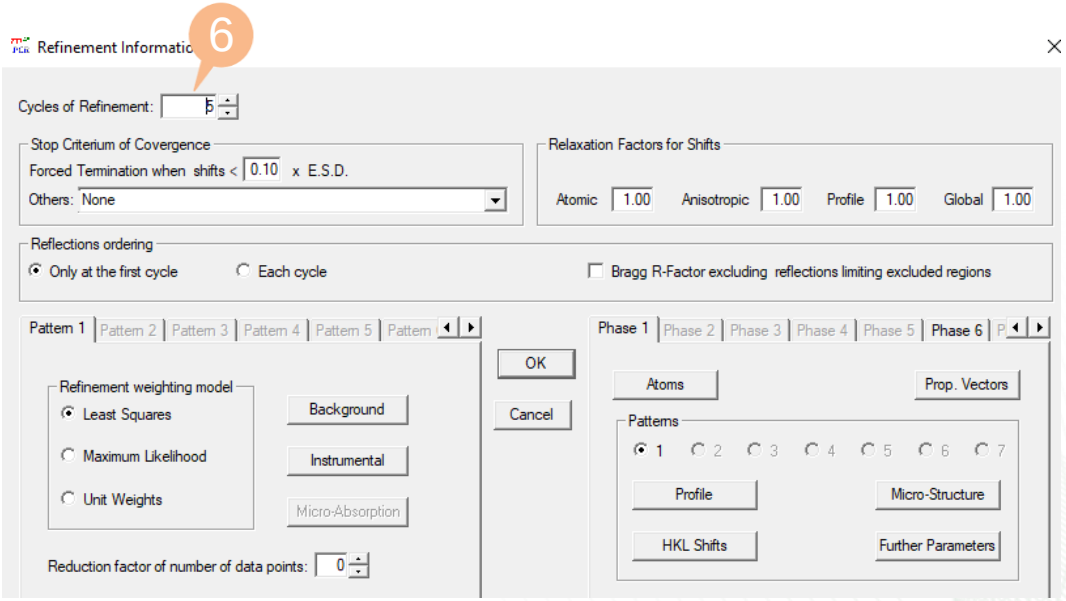

WinPLOTR message:
The BG_300K.BGR has been created: it contains 63 background points
OK

Refinement tab

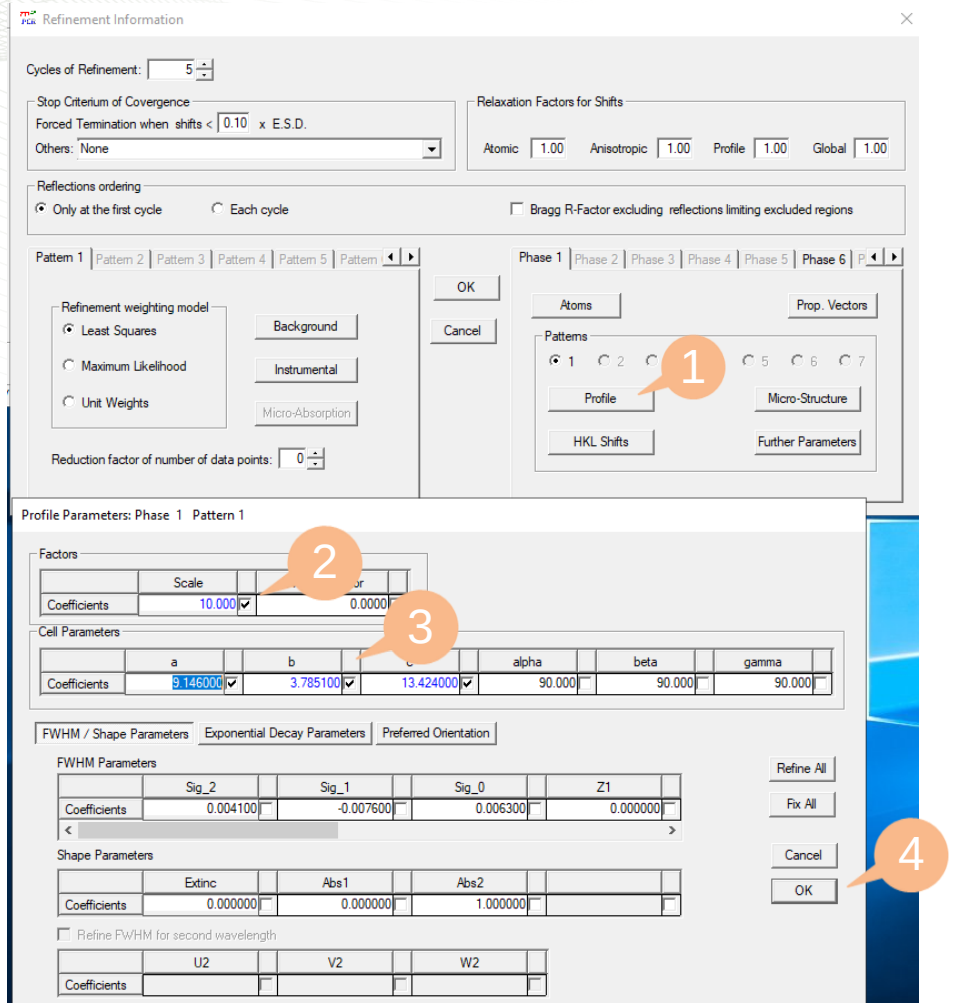


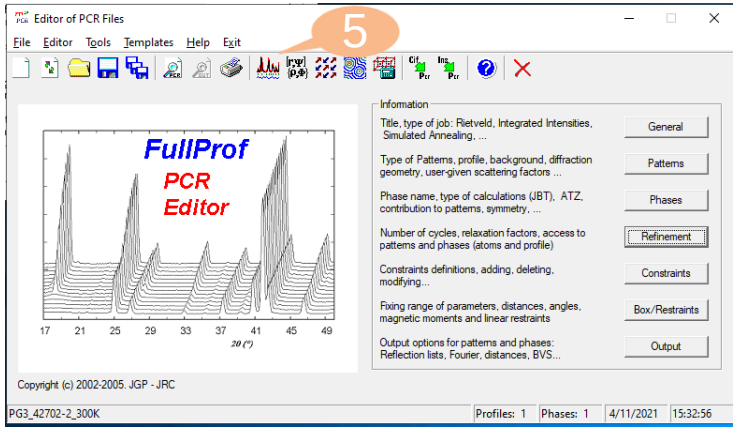




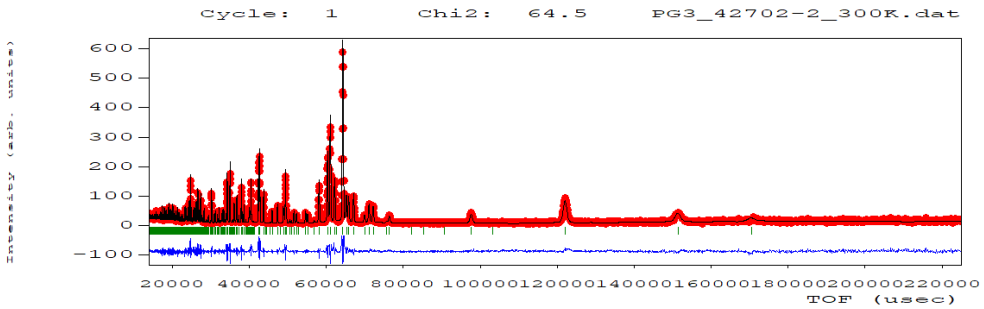


Refinement-Profile tab

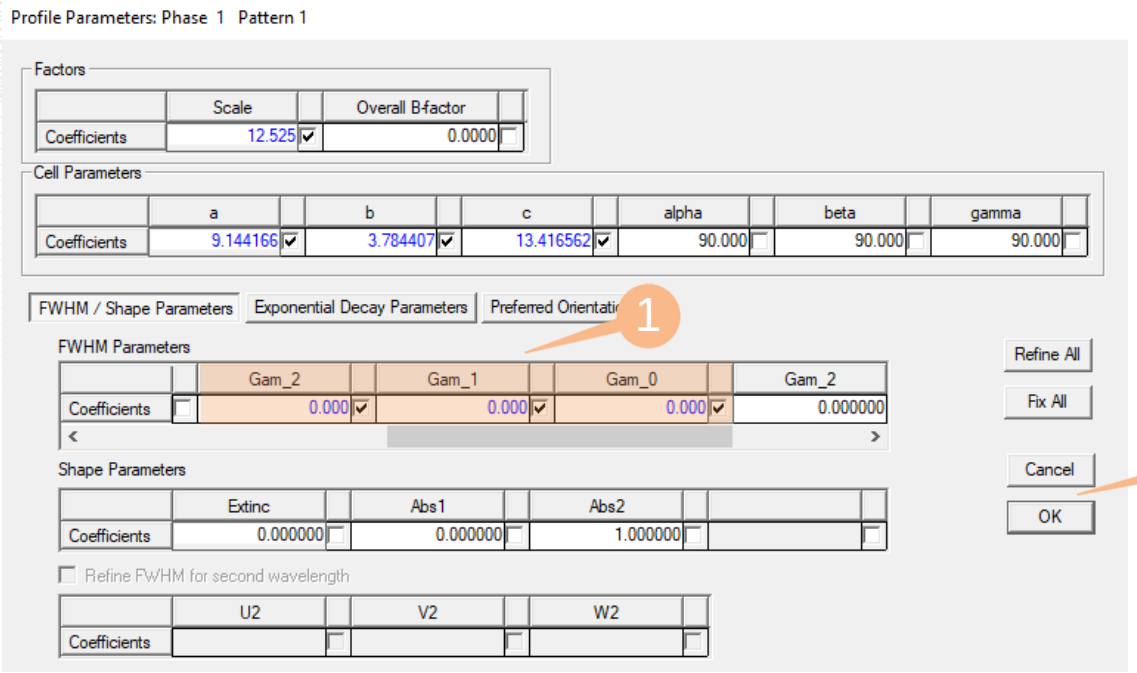


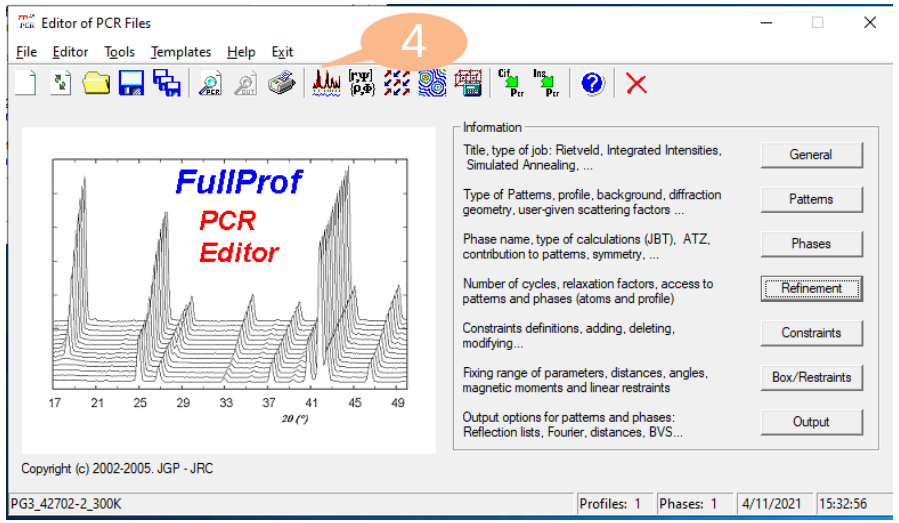



```
=> Phase:       1
=>    Bragg R-factor:   15.01
=>    RF-factor     :   10.72
=> Normal end, final calculations and writing...

=> CPU Time:     0.898 seconds
=> 0.015 minutes

=> END   Date:04/11/2021  Time => 15:52:18.456
```

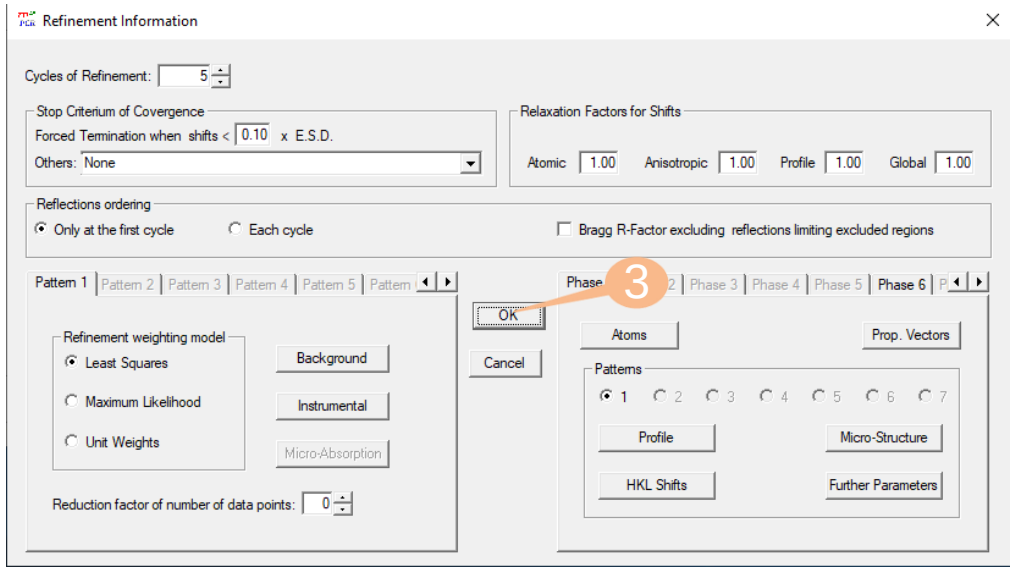


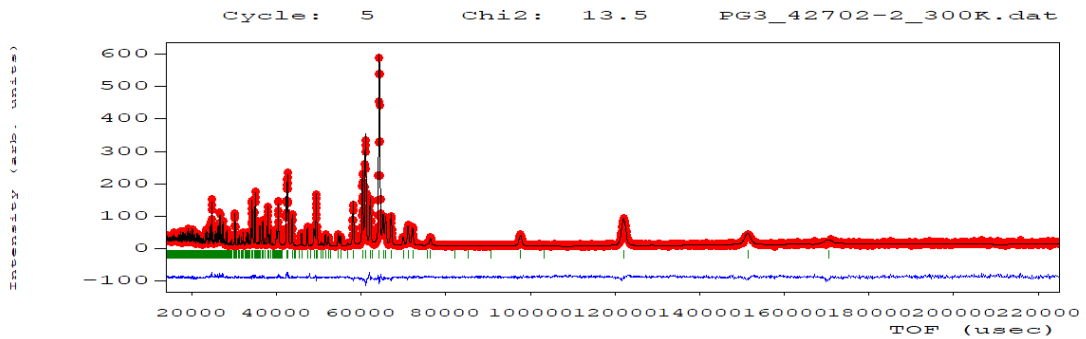
Add the refinement on the peak profile coefficients Gam_2, Gam_1, and Gam_0



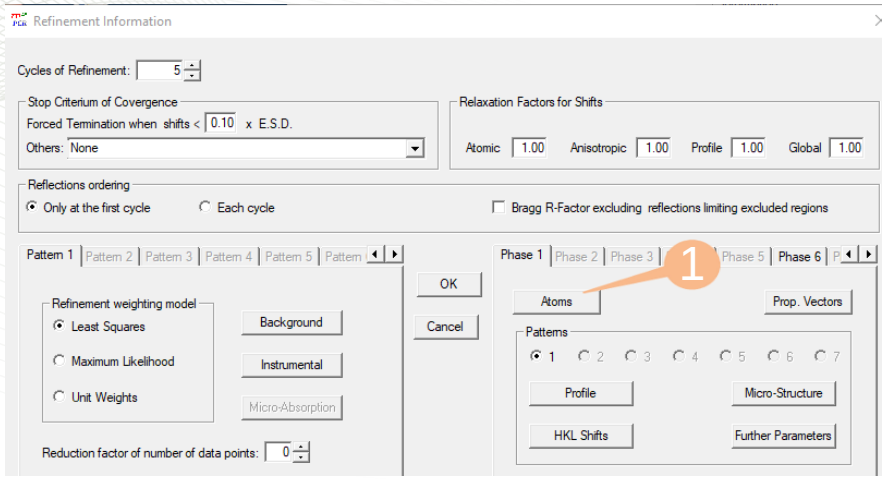


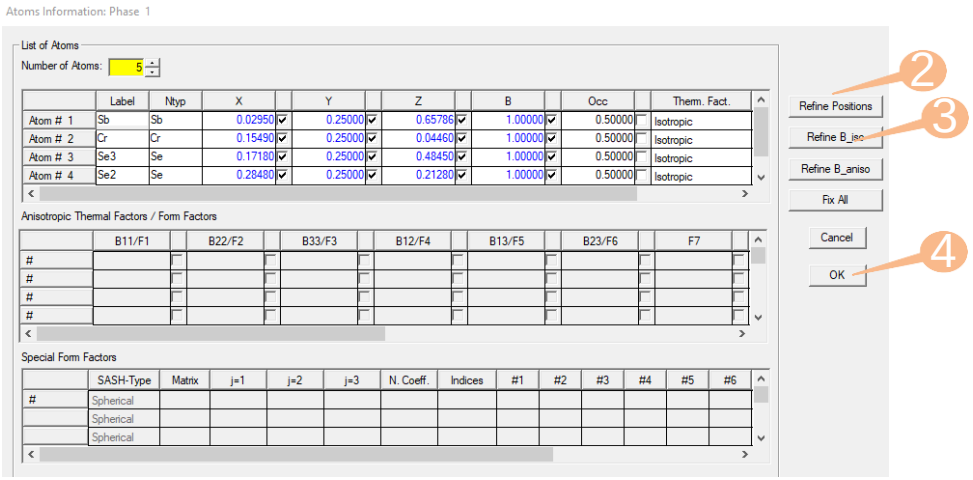

```
=> Phase:       1
=>    Bragg R-factor:    7.922
=>    RF-factor     :    7.961
=> Normal end, final calculations and writing...

=> CPU Time:     3.594 seconds
=> 0.060 minutes

=> END   Date:04/11/2021  Time => 15:49:06.360
```

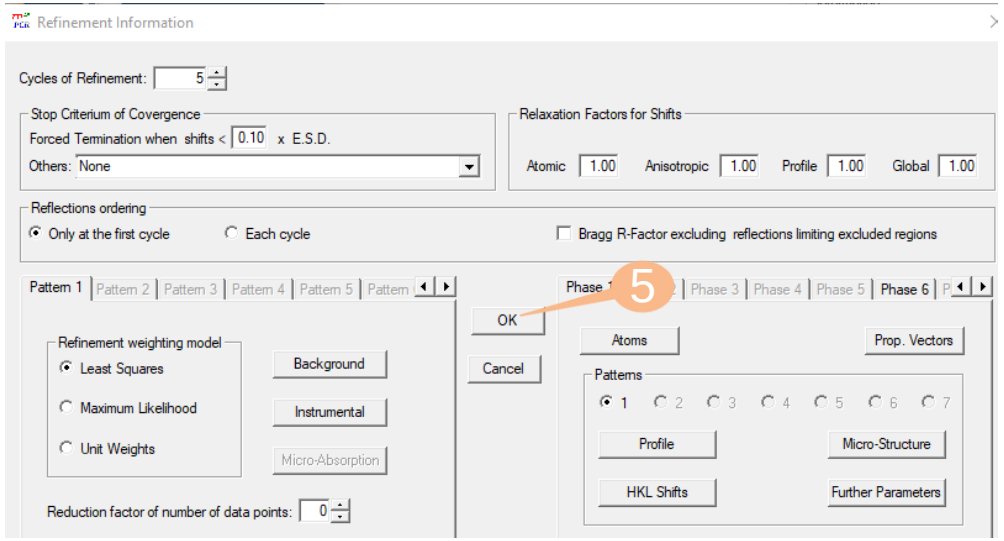




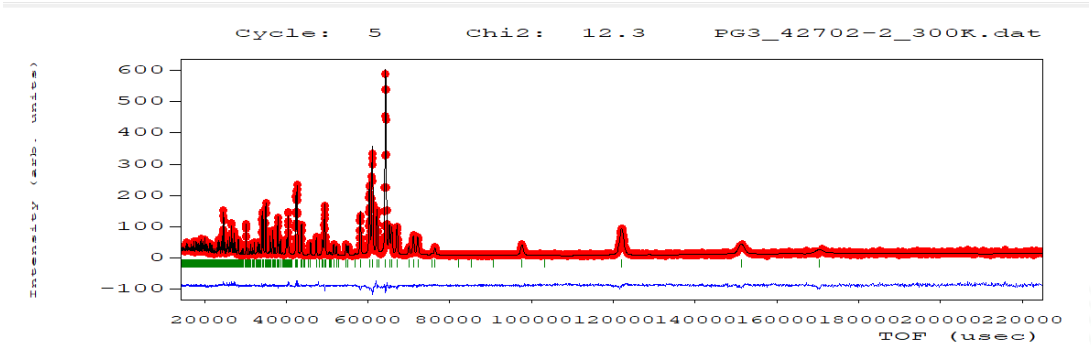
Refinement-Atoms tab (to refine atomic positions and temperature factors B)



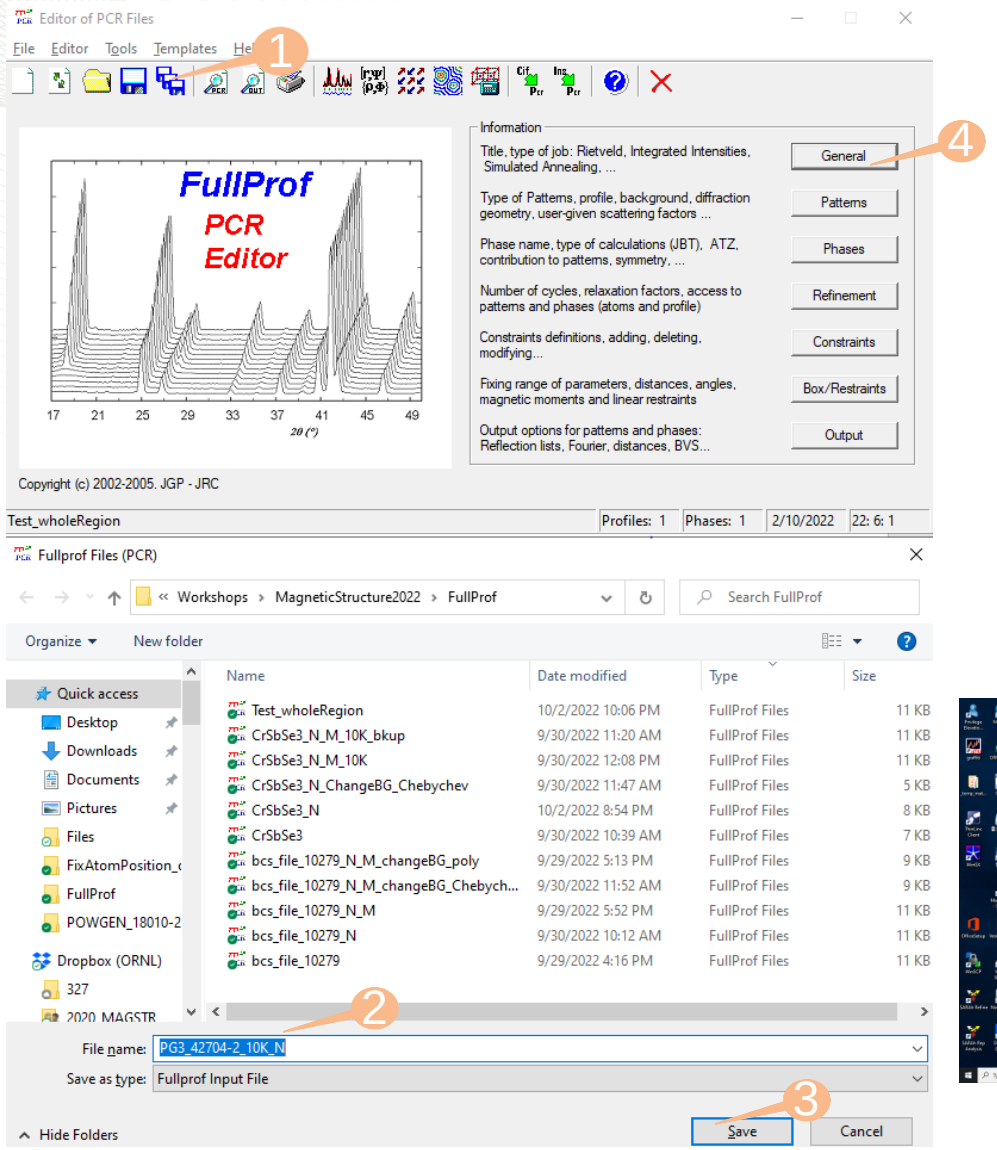




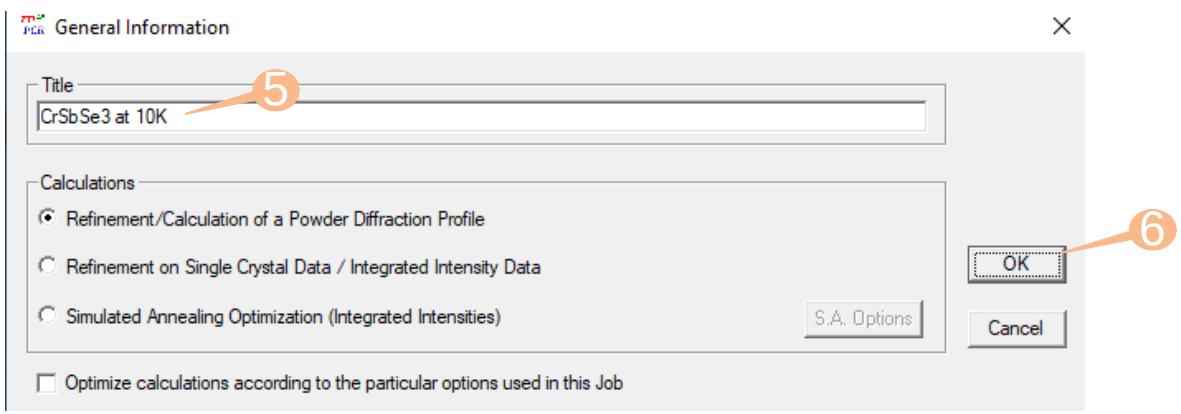

```
   =>    Bragg R-factor:   8.640
   =>    RF-factor     :   6.833
=> Conv. not yet reached -> [Max] Shift(Biso_Cr_ph1)/(eps*Sigma)=   -1.08 abs> 1
=> Normal end, final calculations and writing...


=> CPU Time:     3.750 seconds
=> 0.062 minutes

=> END   Date:04/11/2021  Time => 17:15:08.698
```



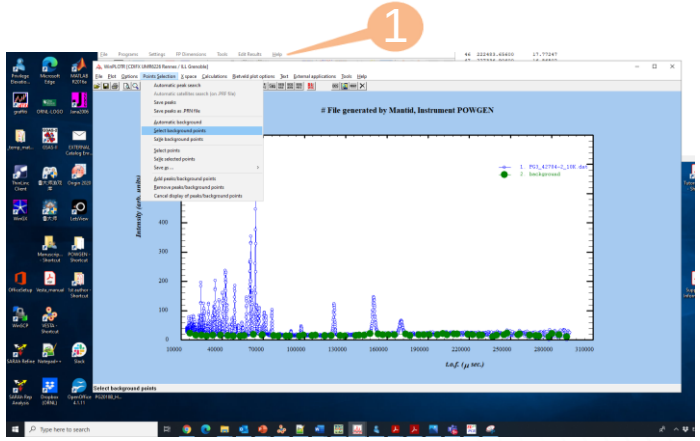
III. Save the PCR file at 300 K as a new PCR to refine the structural parameters at 10 K (T<Tm).

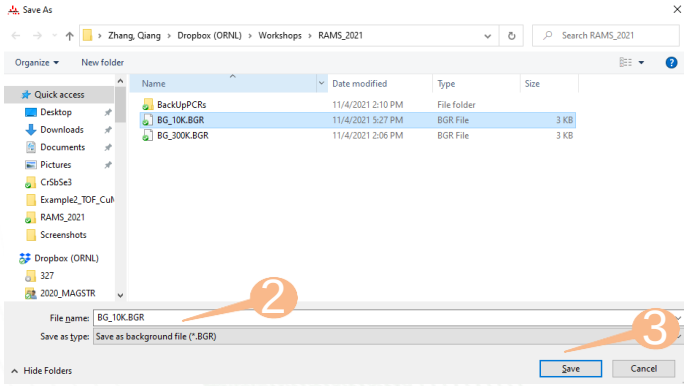


Save as PG3_42704-2_10K_N.pcr

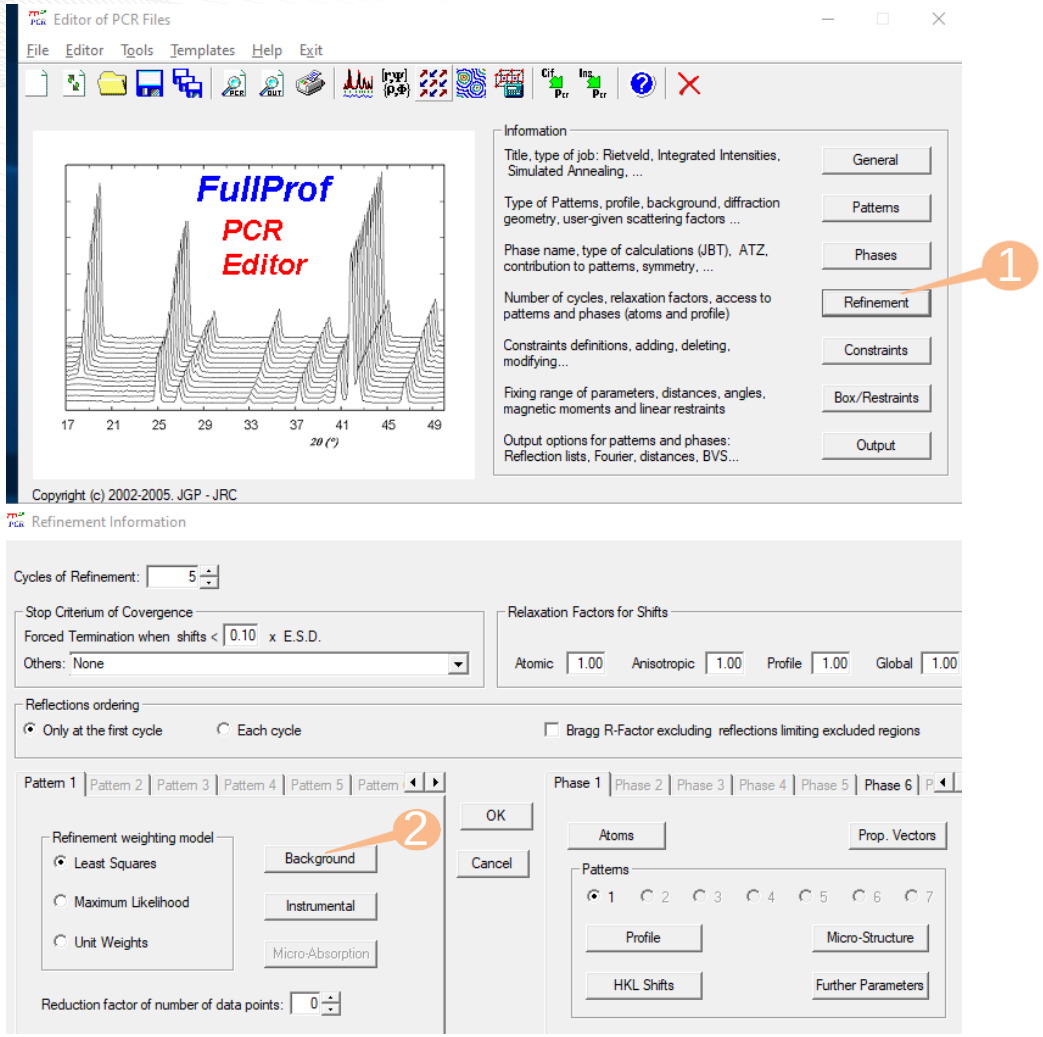


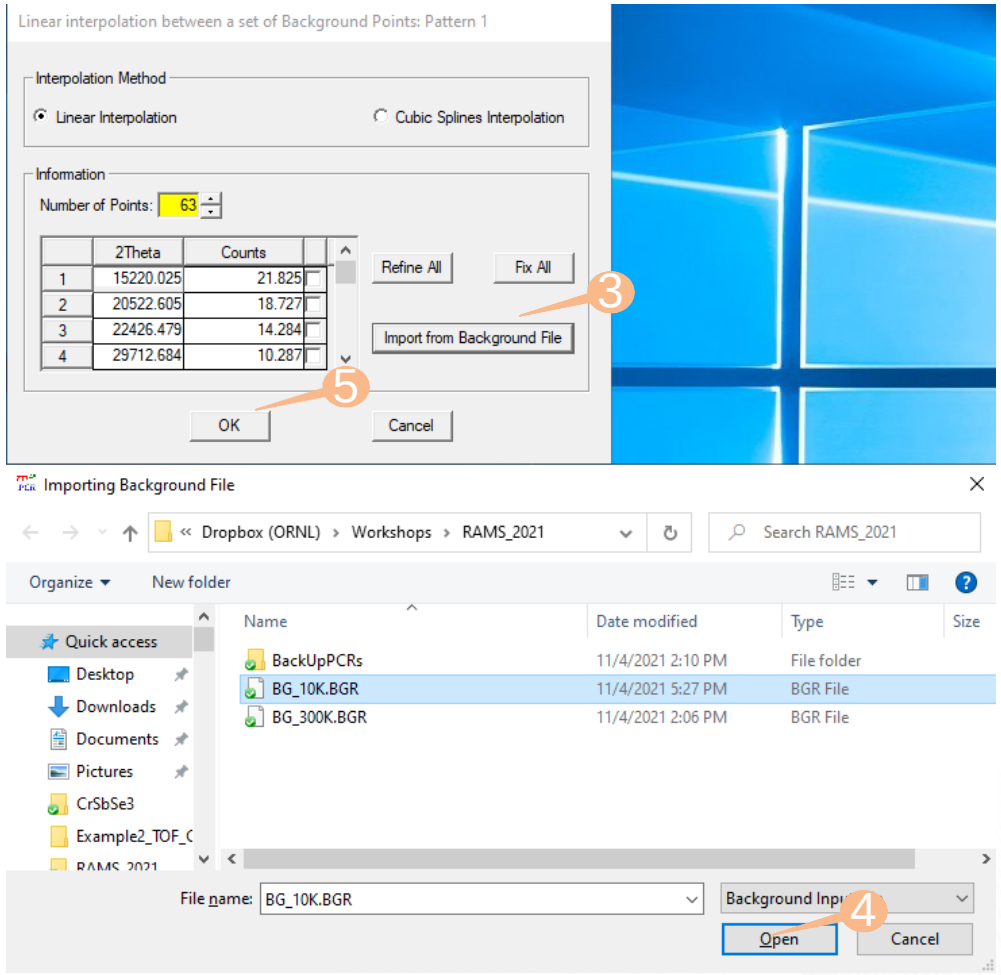
Use Winplotr to get the BG for PG3_42704-2_10K.dat (see slide 12-13)



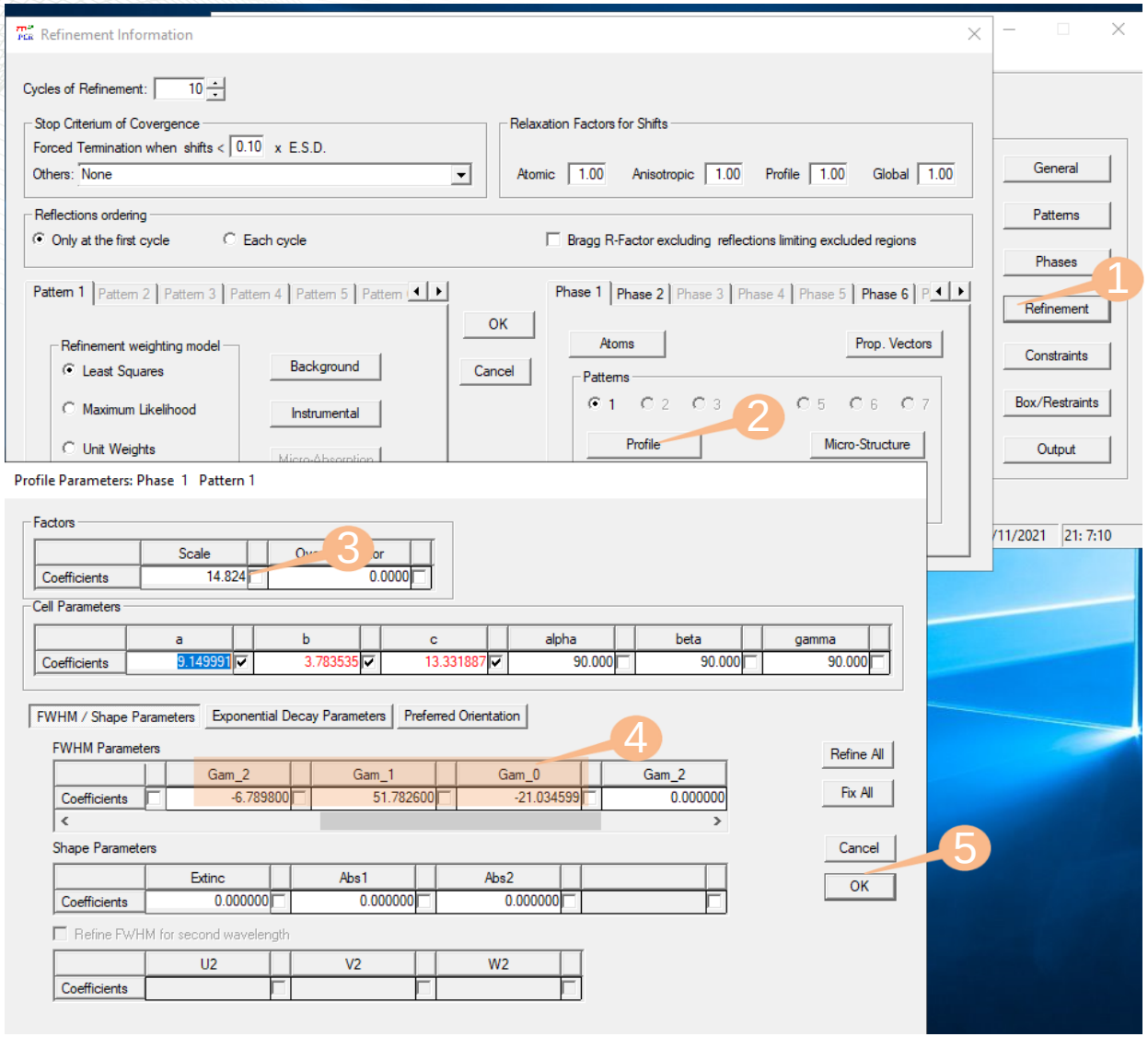


Use the BG for PG3_42704-2_10K.dat

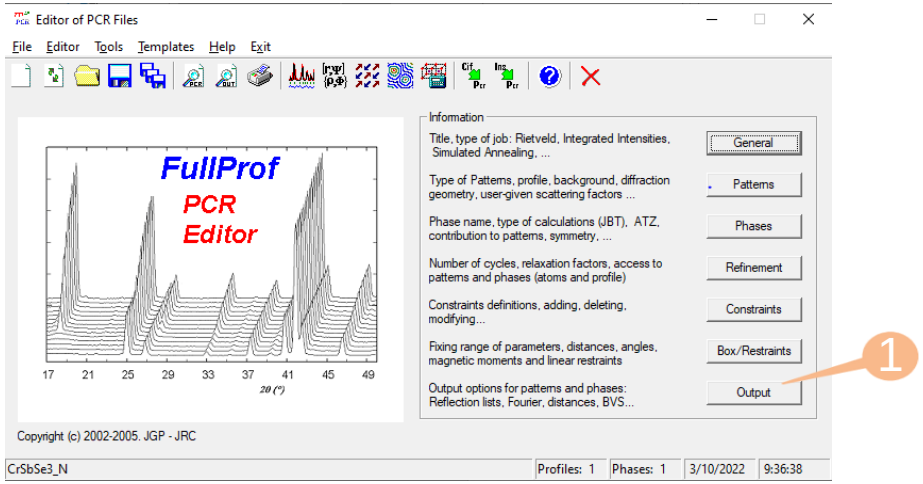


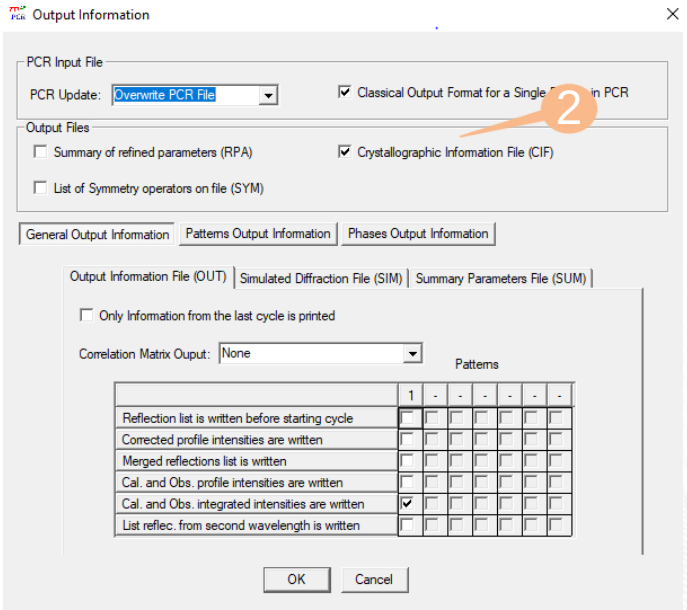


Refinement-Profile tab: unselect scale factor, Gam_2, Gam_1 and Gam_0 and only refine lattice constants, the atomic positions and B factors

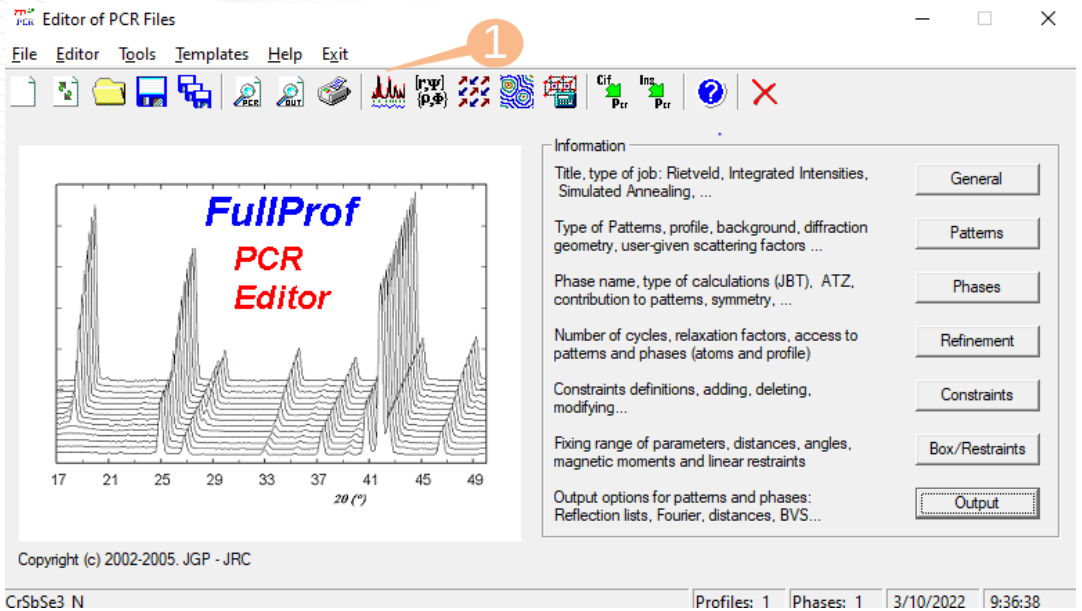


Output cif file at 10 K: used to generate mcif file

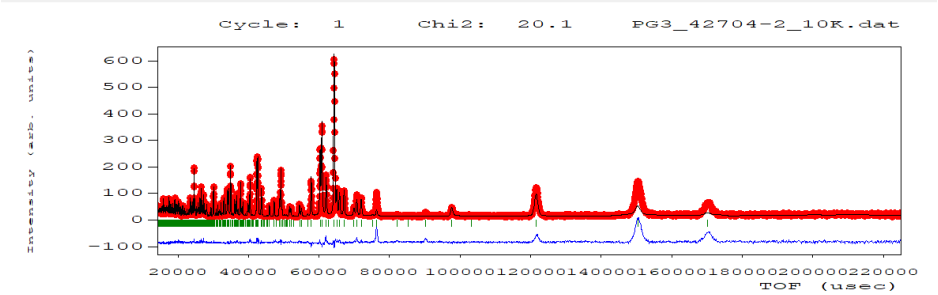




Run refinement:




```
=> Phase:       1
=>    Bragg R-factor:   25.46
=>    RF-factor     :   6.479
=> Normal end, final calculations and writing...

=> CPU Time:     1.219 seconds
=> 0.020 minutes

=> END   Date:04/11/2021  Time => 23:02:54.750
```

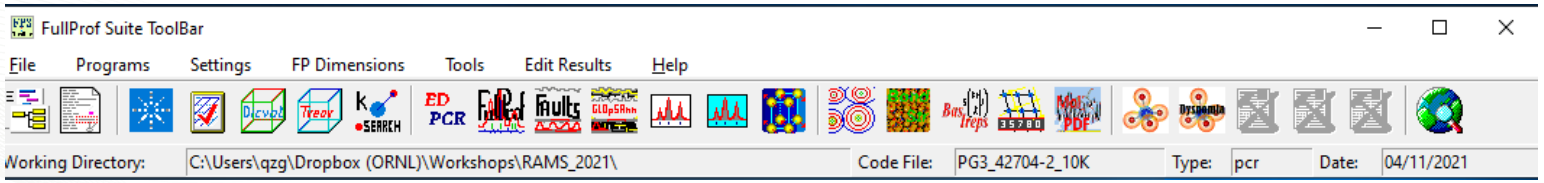


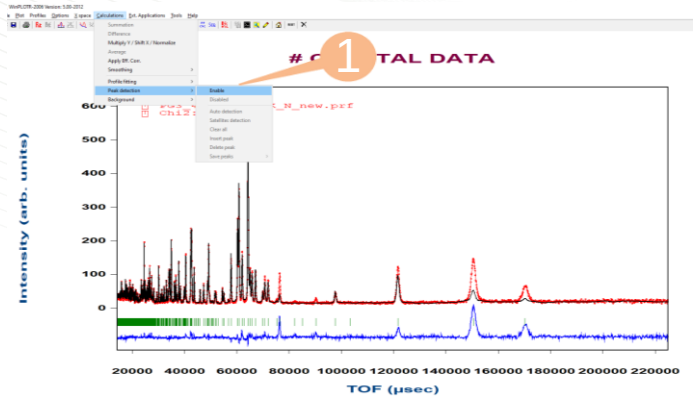
The B factors at 10 K are lower than those at 300 K as expected

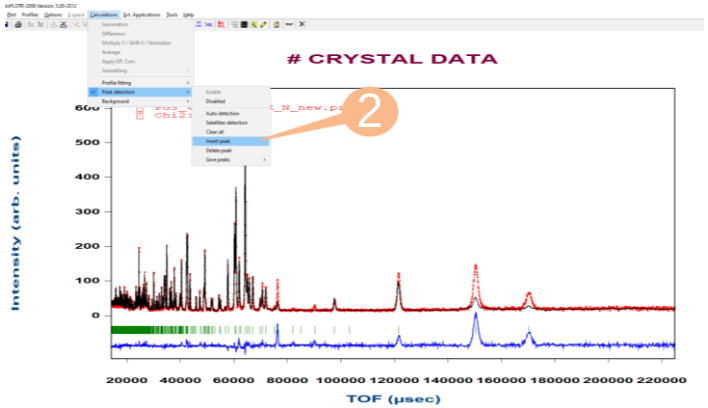
!Atom	Typ	X	Y	Z	Biso
Sb	Sb	0.02909	0.25000	0.65861	0.41769
		61.00	0.00	81.00	71.00
Cr	Cr	0.15737	0.25000	0.04474	0.34746
		91.00	0.00	111.00	101.00
Se3	Se	0.17096	0.25000	0.48400	0.41703
		121.00	0.00	141.00	131.00
Se2	Se	0.28632	0.25000	0.21412	0.38427
		151.00	0.00	41.00	51.00
Se1	Se	0.50108	0.25000	0.60829	0.42097
		31.00	0.00	11.00	21.00

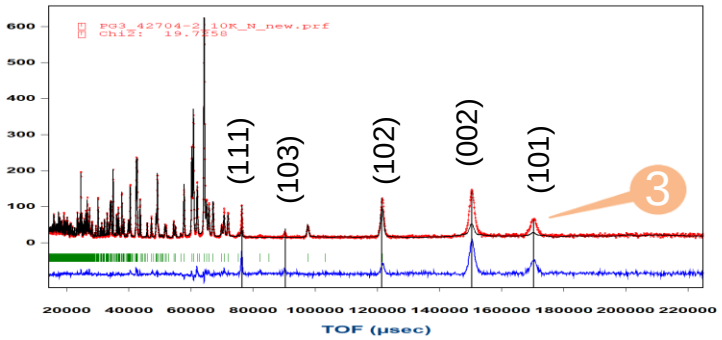
Cif file was output as “PG3_42704-2_10K_N.cif”.

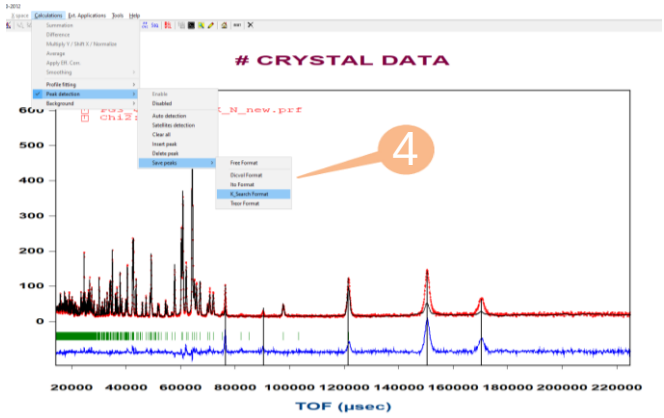
IV. Identify magnetic peaks/contributions and determine the propagation vector

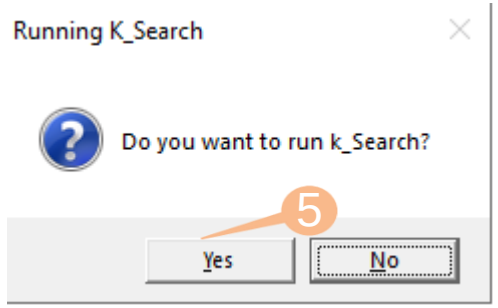













```
=> Testing 90 internal k-vectors
    Solution:       1 k =( 0.0000 0.0000 0.0000)  R-F:   0.5342
    Solution:       2 k =( 0.0000 0.3333 0.3333)  R-F:   3.5499
    Solution:       3 k =( 0.1250 0.5000 0.1250)  R-F:   4.2302
=> Special k-vector solutions found!

=> List of the best incommensurate 10 solutions for  5 satellites

      Kx          Ky          Kz         R-factor
   0.000000    0.000000    0.000000      0.534234
   0.000000    0.333330    0.333330      3.549911
   0.125000    0.500000    0.125000      4.230232

=> The best commensurate solution is the special kvector ks =(  0.0000  0.0000  0.0000)
=> The corresponding R-factor is:       0.5342 to be compared with incommensurate R-factors
```

k=0!

Magnetic peaks sit on the nuclear peak positions!

$$\boldsymbol{k} = \boldsymbol{Q}_M - \boldsymbol{Q}_N = 0$$

V. Use Bilbao Crystallographic Server to check magnetic space groups

Use the cif file output from the refinement on the 10 K data!

Bilbao Crystallographic Server → MAXMAGN Help

MAXMAGN: Maximal magnetic space groups for a given a propagation vector and resulting magnetic structural models

MAXMAGN: Maximal magnetic space groups for a given a propagation vector and resulting magnetic structural models

MAXMAGN provides the possible magnetic space groups that can be assigned to a 1-k commensurate magnetic phase assuming that the magnetic symmetry is a maximal one. The space group of the paramagnetic phase (parent group) and the observed propagation vector are required as input. Optionally, the parent paramagnetic structure can be introduced (by hand or by a cif file). In this latter case the program provides the constrains for the different possible symmetries and cif-like files can be produced. These files permit the different alternative models to be analyzed, refined, shown graphically, transported to ab-initio codes etc., with programs as ISODISTORT, JANA2006, StrConvert, VESTA, etc. These cif-like files can also be submitted to the program MVISUALIZE, which allow 3D visualization of magnetic structures with Jmol. A controlled descent to lower symmetries is also possible.

1

☑ *Structure data of the paramagnetic phase will be included* ☐ *Non-conventional setting*

Please, enter the label of the space group of the paramagnetic phase (parent group) choose it

Please, enter the propagation vector k: k_x 0 k_y 0 k_z 0

2

3

Submit

Bilbao Crystallographic Server → MAXMAGN - Maximal magnetic space groups

Parent paramagnetic structure cif file

Option 1: Please submit a structure file (CIF format):

Choose File PG3_42704-2_10K_N.cif Upload the file

Bilbao Crystallographic Server → MAXMAGN - Maximal magnetic space groups

Parent phase structure data: Magnetic Atoms

Parent space group: *Pnma* (No. 62)
Lattice parameters (Angstroms and degrees): a=9.14968, b=3.78330, c=13.33090, alpha=90.0000, beta=90.0000, gamma=90.0000

Atoms: Please select the magnetic ones

N	Atom name	Atom type	Wyckoff Position	Coordinates	Magnetic?
1	Sb	Sb	4c	0.02910 0.25000 0.65879	☐
2	Cr	Cr	4c	0.15735 0.25000 0.04497	☑
3	Se3	Se	4c	0.17073 0.25000 0.48381	☐
4	Se2	Se	4c	0.28660 0.25000 0.21393	☐
5	Se1	Se	4c	0.50111 0.25000 0.60835	☐

4

Submit

5

Maximal magnetic space groups for the parent space group *Pnma* (No. 62) and the propagation vector k = (0, 0, 0)

Maximal subgroups which allow non-zero magnetic moments for at least one atom are coloured

N	Group (BNS)	Transformation matrix	General positions	Properties	Magnetic structure
1	*Pn'm'a'* (#62.449) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
2	*Pn'ma'* (#62.448) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
3	*Pnm'a'* (#62.447) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
4	*Pn'm'a* (#62.446) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
5	*Pnma'* (#62.445) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
6	*Pnm'a* (#62.444) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
7	*Pn'ma* (#62.443) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
8	*Pnma* (#62.441) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show

6

groups

Magnetic Structure

Selected magnetic space group: 3- *Pnm'a'* (#62.447)

Setting of the parent group

Parent space group *Pnma* (No. 62)

Lattice parameters: a=9.14600, b=3.78510, c=13.42400, alpha=90, beta=90, gamma=90

[Go to setting standard (a, ... 0)]
[Go to an altern...]

Export data to MCIF file/Visualize | Go to a subgroup

8

Atomic positions, Wyckoff positions and Magnetic Moments

N	Atom	New WP	Multiplicity	Magnetic moment	Values of M_x, M_y, M_z
1	Sb Sb 0.02950 0.25000 0.65786	(x,1/4,z | m_x,0,m_z) (-x+1/2,3/4,z+1/2 | m_x,0,-m_z) (-x,3/4,-z | m_x,0,m_z) (x+1/2,1/4,-z+1/2 | m_x,0,-m_z)	4	-	-
2	Cr Cr 0.15490 0.25000 0.04460	(x,1/4,z | m_x,0,m_z) (-x+1/2,3/4,z+1/2 | m_x,0,-m_z) (-x,3/4,-z | m_x,0,m_z) (x+1/2,1/4,-z+1/2 | m_x,0,-m_z)	4	(M_x,0,M_z)	M_x = 1.00000 M_z = 2.00000
3	Se3 Se 0.17180 0.25000 0.48450	(x,1/4,z | m_x,0,m_z) (-x+1/2,3/4,z+1/2 | m_x,0,-m_z) (-x,3/4,-z | m_x,0,m_z) (x+1/2,1/4,-z+1/2 | m_x,0,-m_z)	4	-	-
4	Se2 Se 0.28480 0.25000 0.21280	(x,1/4,z | m_x,0,m_z) (-x+1/2,3/4,z+1/2 | m_x,0,-m_z) (-x,3/4,-z | m_x,0,m_z) (x+1/2,1/4,-z+1/2 | m_x,0,-m_z)	4	-	-
5	Se1 Se 0.50190 0.25000 0.60870	(x,1/4,z | m_x,0,m_z) (-x+1/2,3/4,z+1/2 | m_x,0,-m_z) (-x,3/4,-z | m_x,0,m_z) (x+1/2,1/4,-z+1/2 | m_x,0,-m_z)	4	-	-

7

Bilbao Crystallographic Server → MAXMAGN - Maximal magnetic space groups

mCIF file of the structure

Submit this mcif file to MVISUALIZE for 3D visualization of the estructure usi... Submit to MVISUALIZE

9 10

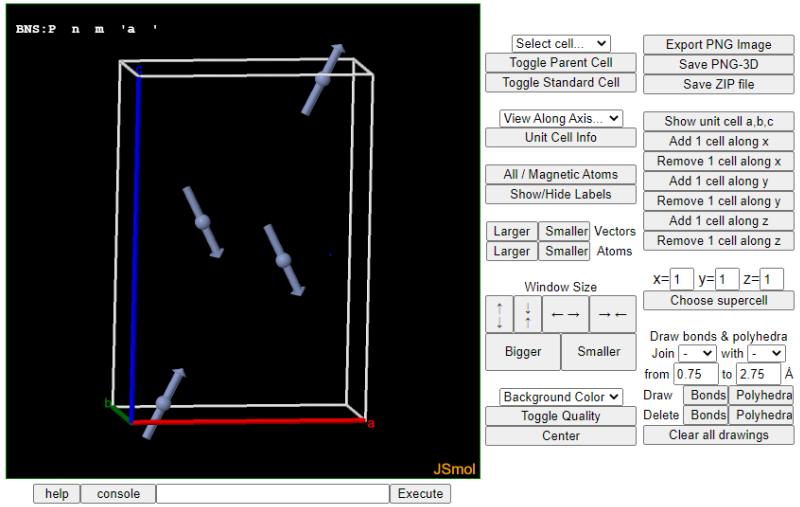
Download mCIF file: bcs_file.mcif
[The preview text below is non-editable, only copy-allowed]

```
# Created by the Bilbao Crystallographic Server
# http://www.cryst.ehu.es
# Date: 29/09/2022 17:55:34
# CrSbSe3.cif

data_5yOhtAoR
_audit_creation_date            2022-09-29
_audit_creation_method          "Bilbao Crystallographic Server"

_citation_journal_abbrev        ?
_citation_journal_volume        ?
_citation_page_first            ?
_citation_page_last             ?
_citation_article_id            ?
_citation_year                  ?
_citation_DOI                   ?

loop_
_citation_author_name
?

_atomic_positions_source_database_code_ICSD  ?
_atomic_positions_source_other               ?

_transition_temperature  ?
_experiment_temperature  ?

loop_
_irrep_id
_irrep_dimension
_irrep_small_dimension
_irrep_direction_type
_irrep_action
_irrep_modes_number
_irrep_presence
```

MVISUALIZE: 3D Visualization of magnetic structures with Jmol

MVISUALIZE Main Page

Show/Hide File



Canted order:
FM alignment along the *a* axis,
AFM alignment along the *c* axis.

Rename mcif file as 62p447.cif

VI. Covert one mcif file to PCR file

Bilbao Crystallographic Server → mCIF2PCR

mCIF2PCR: Transformation from mCIF to PCR format (FullProf).

mCIF_to_PCR

Given a magCIF file, it produces a PCR template that can be used as input for FullProf (the program mCIF_to_PCR (FullProf) is used). By default the provided PCR file is intended for a simulation. The file can be then modified by hand for a refinement of the model.

Choose a structure file (mCIF format):

1 Choose File 64p447.mcif

2 Convert

Bilbao Crystallographic Server → mCIF2PCR

mCIF2PCR: Transformation from mCIF to PCR format (FullProf).

mCIF_to_PCR

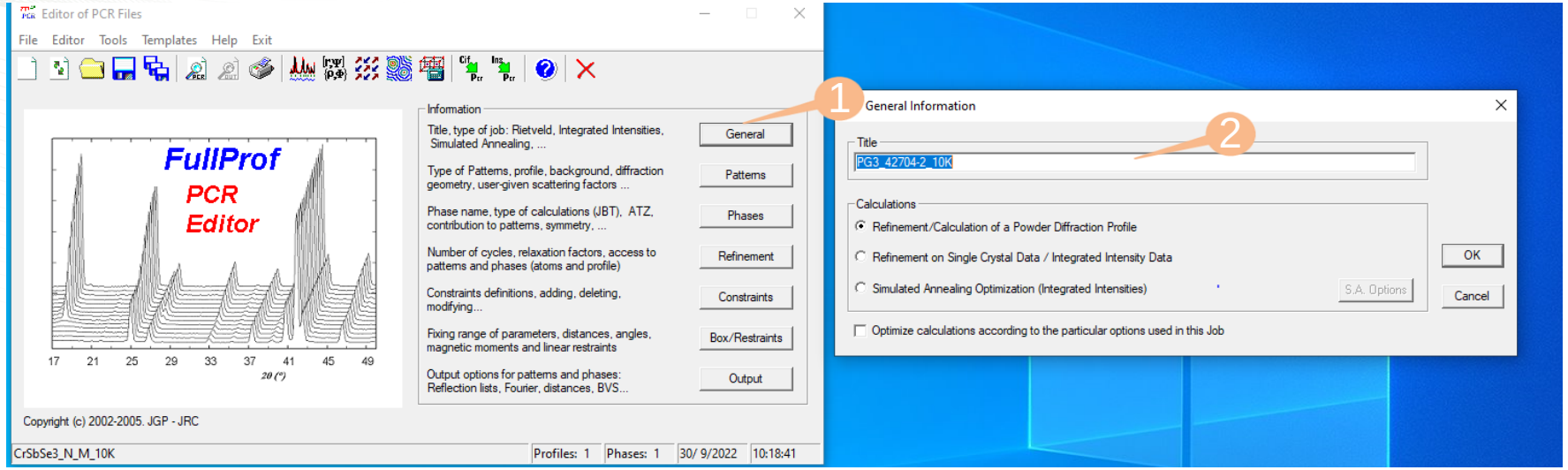
Given a magCIF file, it produces a PCR template that can be used as input for FullProf (the program mCIF_to_PCR (FullProf) is used). By default the provided PCR file is intended for a simulation. The file can be then modified by hand for a refinement of the model.

The file has been successfully converted.

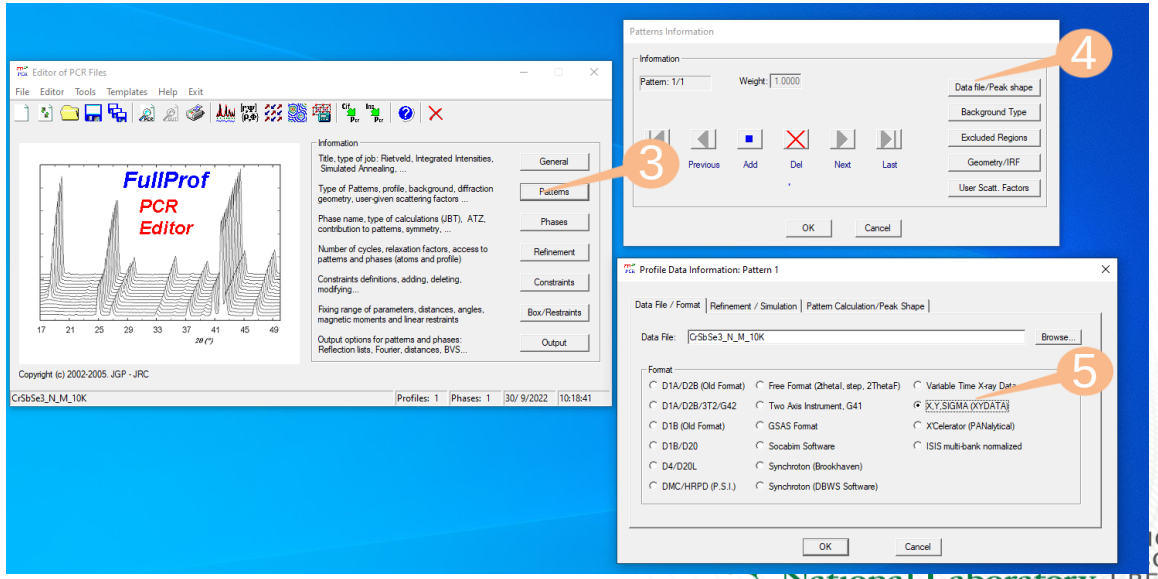
3 Click to download it

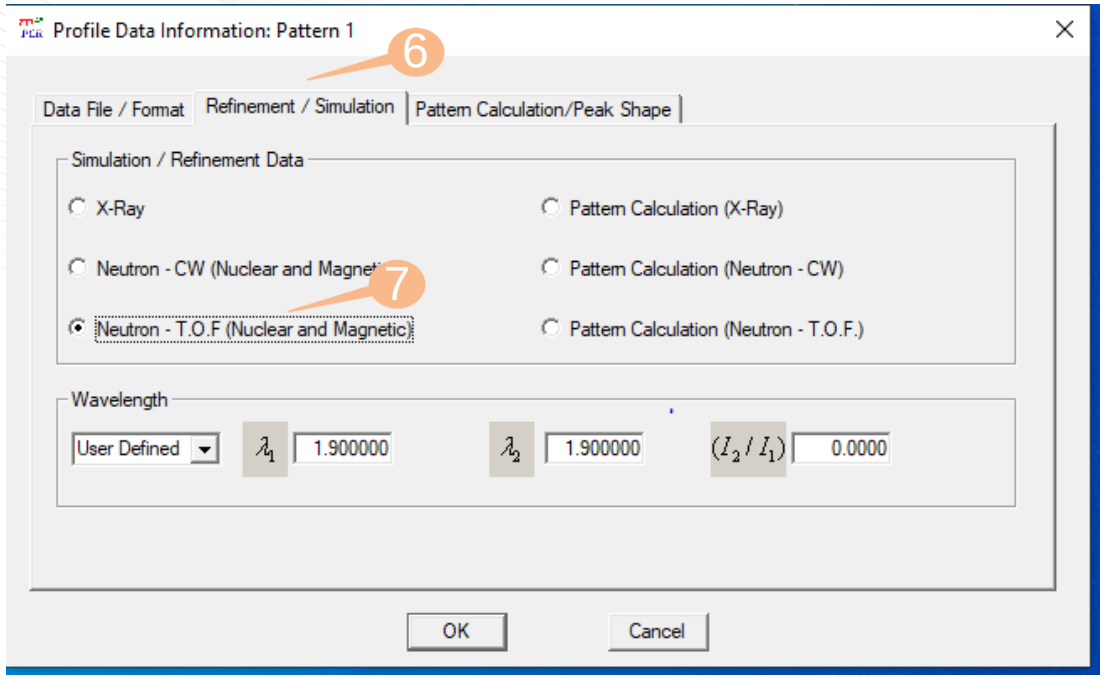
VII. Refine both nuclear and magnetic peaks at 10 K to obtain the magnetic structure and ordered moment

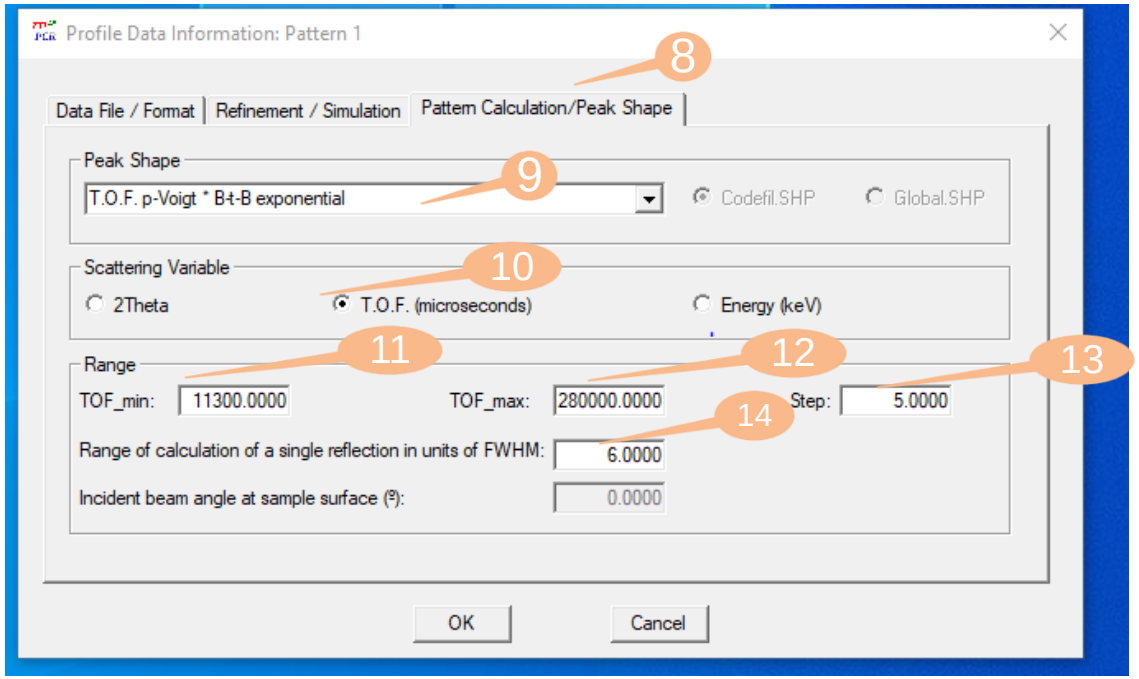
General tab



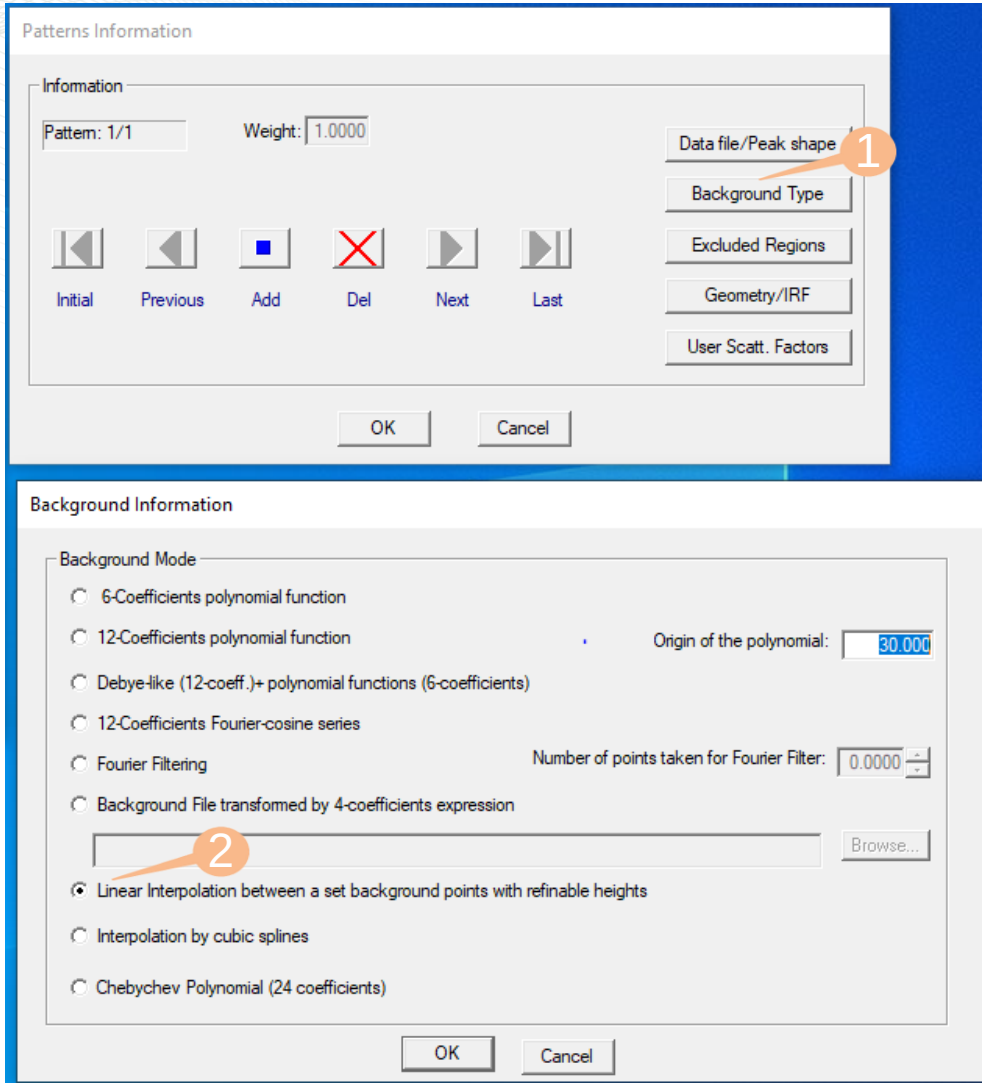
Pattern-Datafile/Peak shape tab



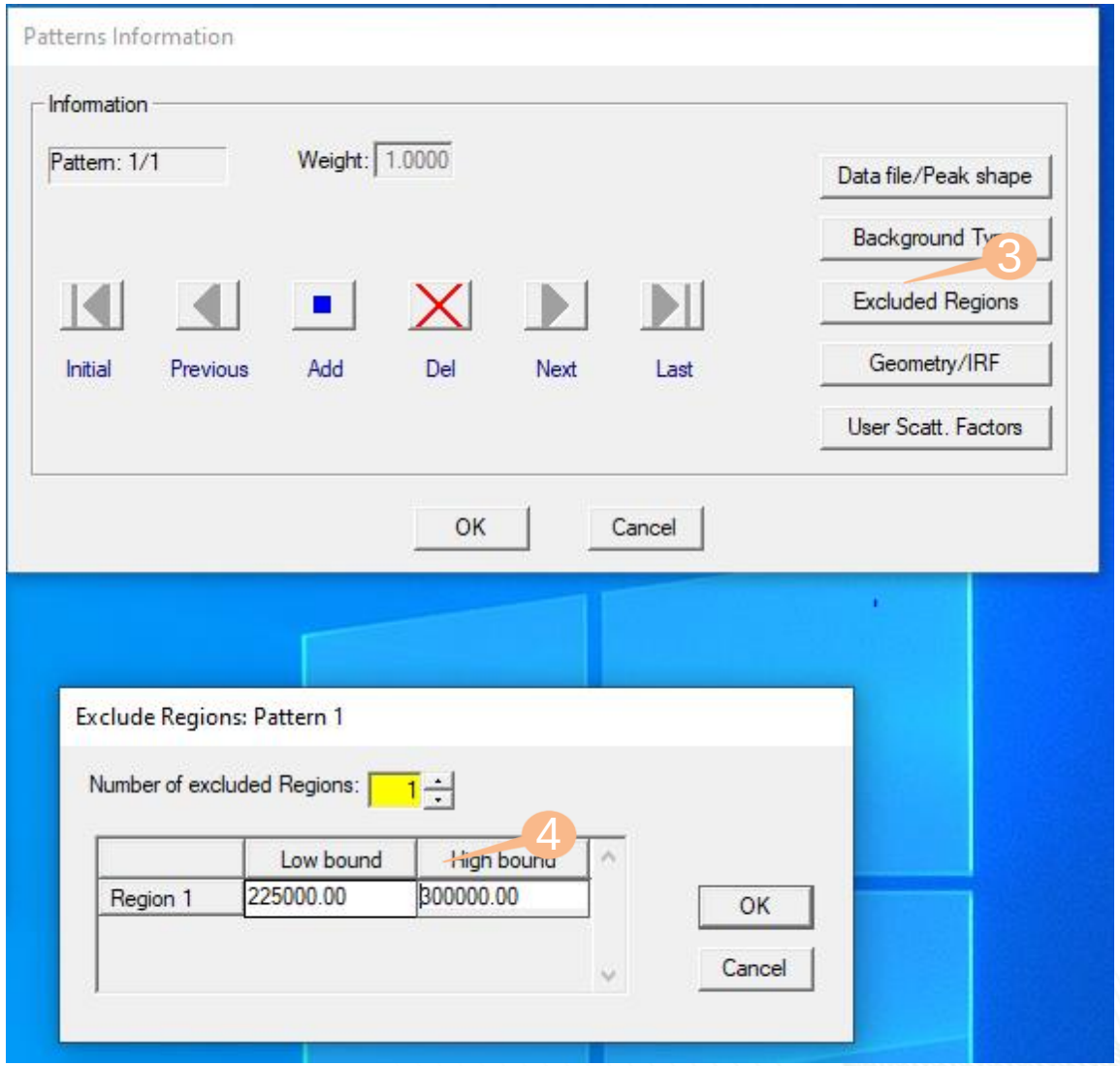

Profile Data Information: Pattern 1
6
Data File / Format
Refinement / Simulation
Pattern Calculation/Peak Shape
Simulation / Refinement Data
X-Ray
Pattern Calculation (X-Ray)
Neutron - CW (Nuclear and Magnet
7
Pattern Calculation (Neutron - CW)
Neutron - T.O.F (Nuclear and Magnetic)
Pattern Calculation (Neutron - T.O.F.)
Wavelength
User Defined
λ1
1.900000
λ2
1.900000
(I2 / I1)
0.0000
OK
Cancel


Profile Data Information: Pattern 1
8
Data File / Format
Refinement / Simulation
Pattern Calculation/Peak Shape
Peak Shape
9
T.O.F. p-Voigt * B-t-B exponential
Codefil.SHP
Global.SHP
Scattering Variable
10
2Theta
T.O.F. (microseconds)
Energy (keV)
Range
11
12
13
TOF_min:
11300.0000
TOF_max:
280000.0000
Step:
5.0000
14
Range of calculation of a single reflection in units of FWHM:
6.0000
Incident beam angle at sample surface (º):
0.0000
OK
Cancel

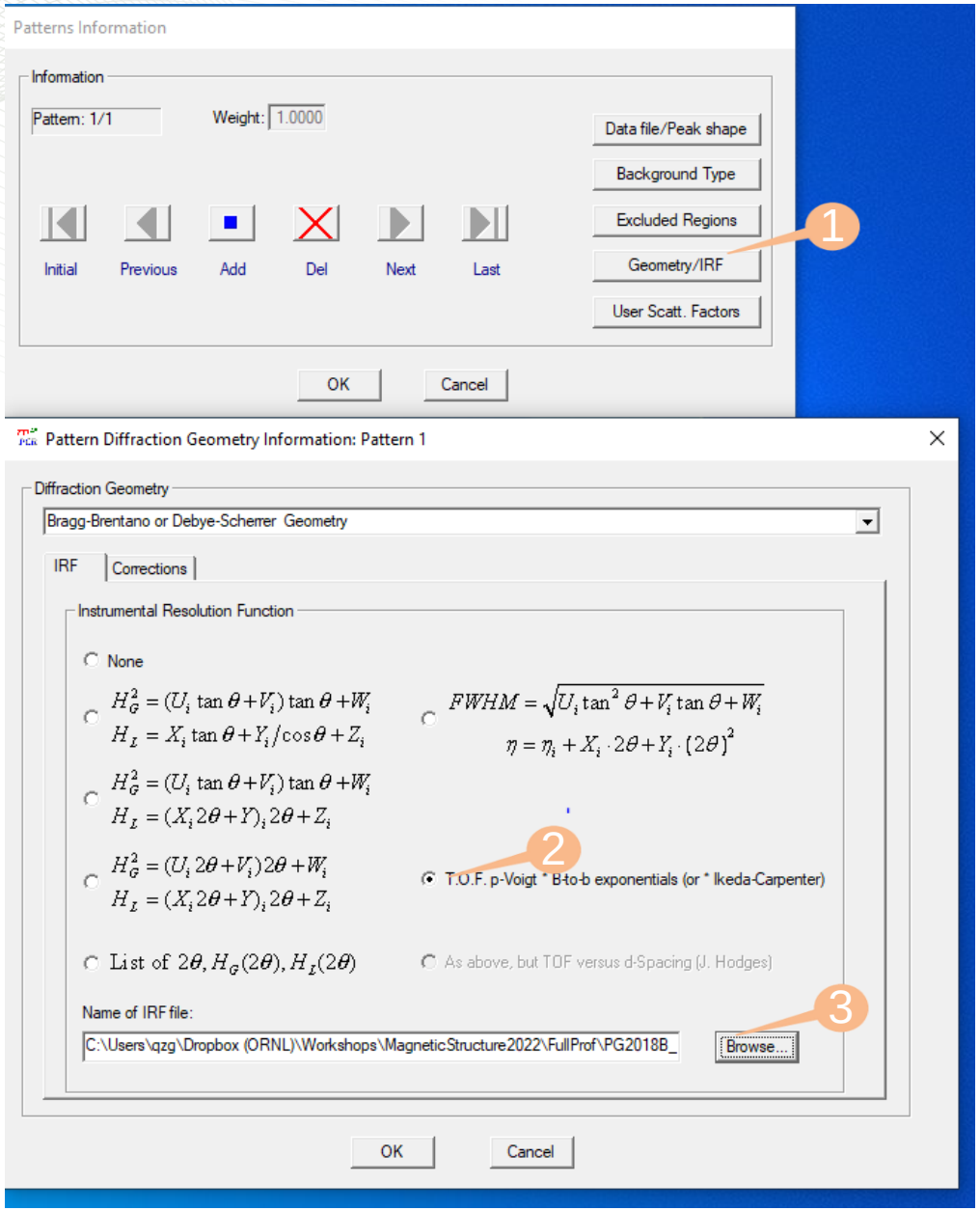
Pattern-Background Type



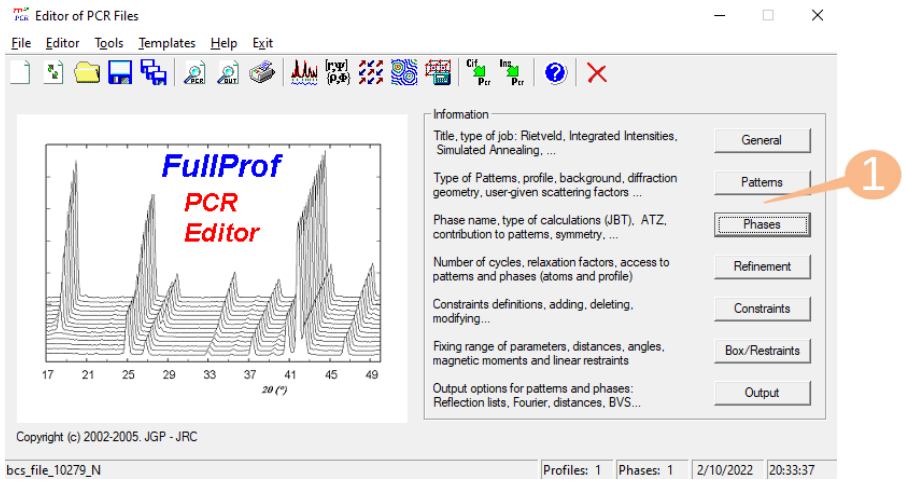
Pattern-Excluded Regions

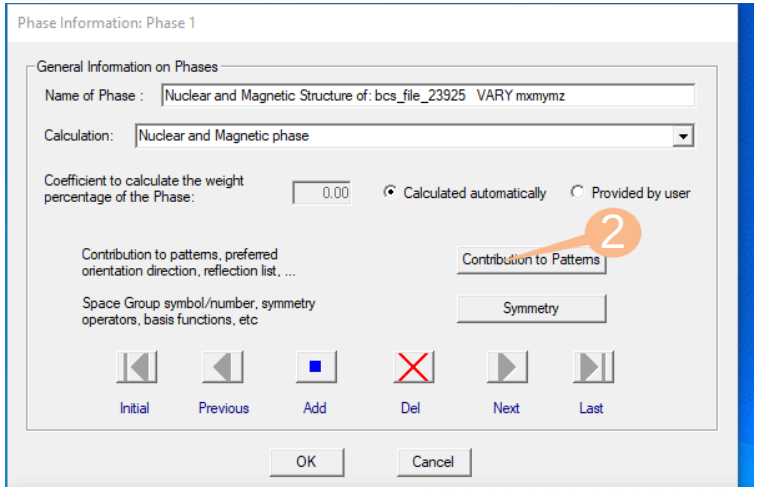


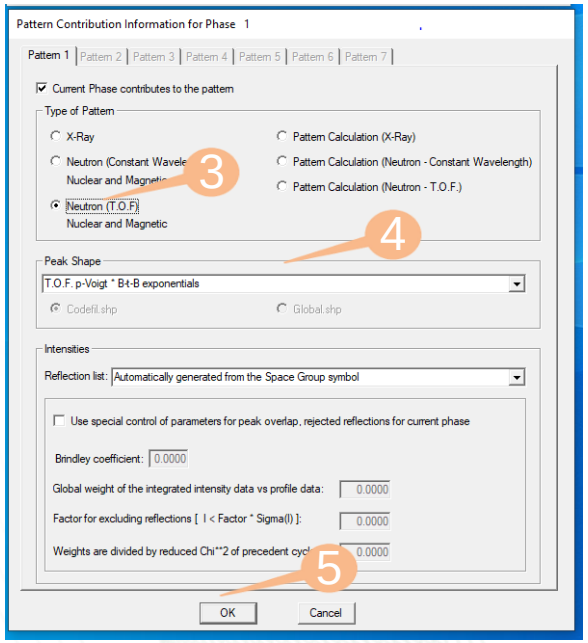
Pattern-Geometry/IRF



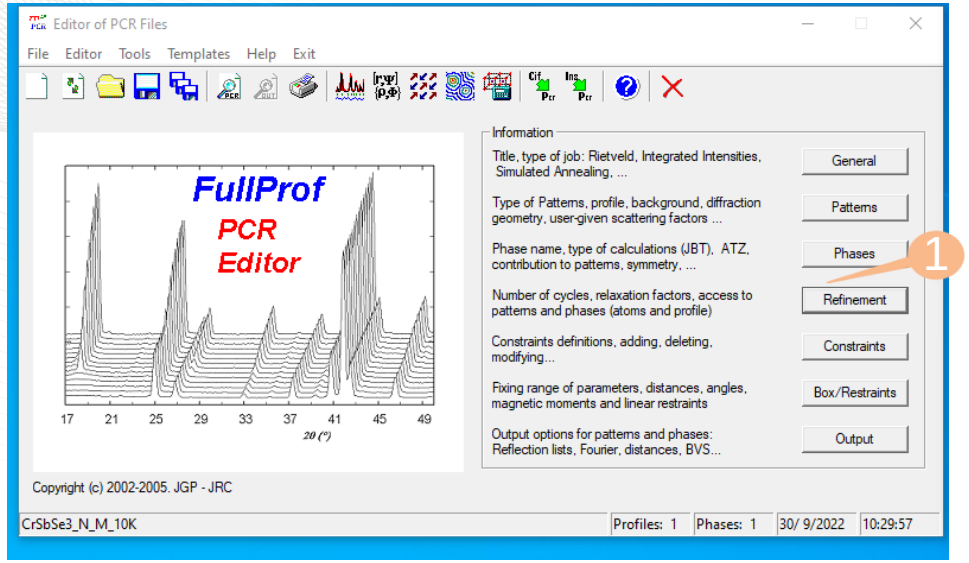
Phases

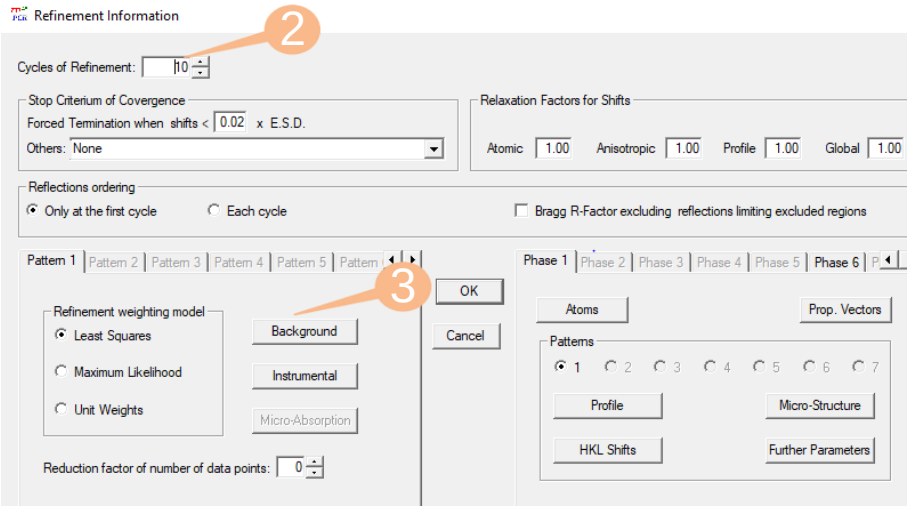


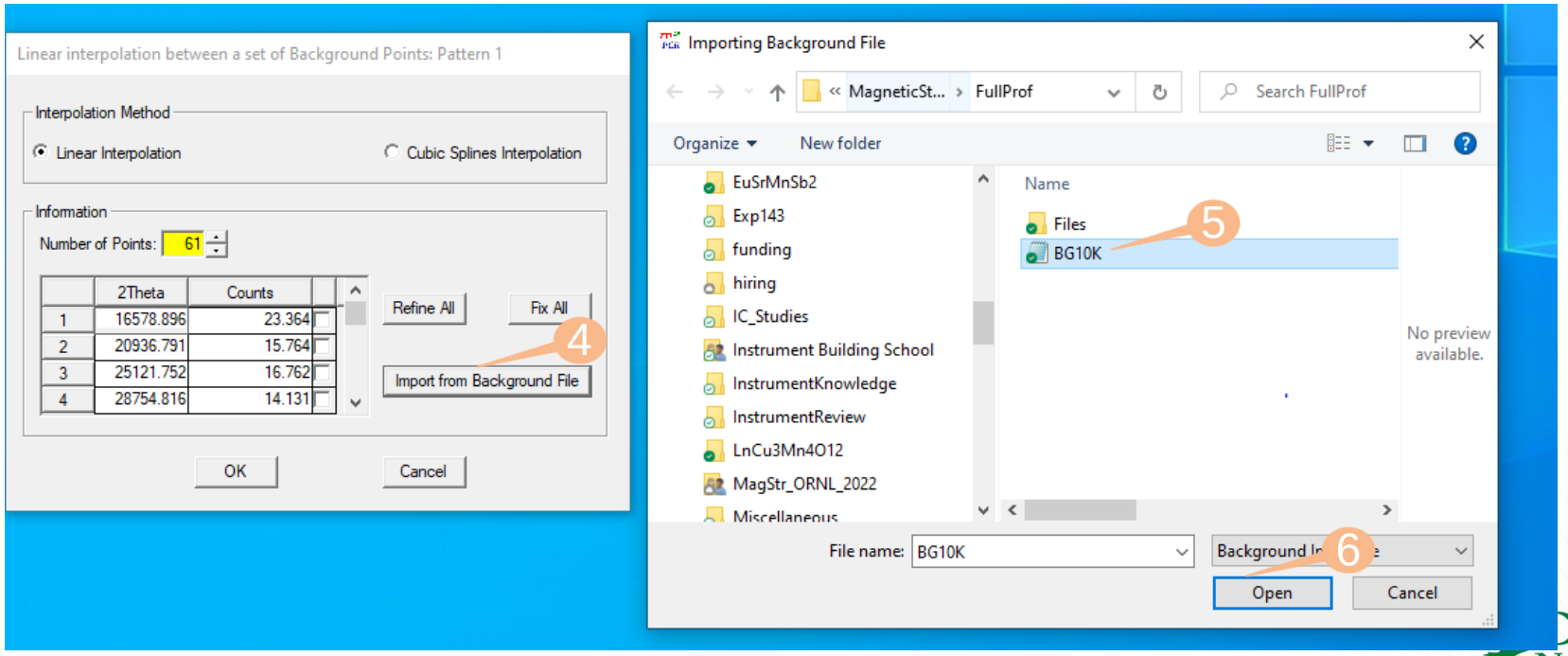




Refinement tab







Copy “profile parameters for pattern” part from PG3_42704-2_10K_N.pcr file

External EdPCR Text Editor - [C:\Users\qzg\Dropbox (ORNL)\Workshops\RAMS_2021\Day2\RAMS_2021_Final_QZhang\PG3_42704-2_10K_N.pcr]

File Edit Search

```
    268452.6562        15.4548          0.00
    270606.8125        13.7025          0.00
    278061.1250        10.5411          0.00
    280292.4062        20.9029          0.00
    285264.4375        16.8889          0.00
    291021.5938        11.3446          0.00
!
! Excluded regions (LowT  HighT) for Pattern#  1
   225000.00   300000.00
!
!
       1    !Number of refined parameters
!
!    Zero_i+SHF    Dtt1_i+SHF    Dtt2_i+SHF   Dtt_1overd_i+SHF  TwoThetaBank (Only shifts are refined)
!     -13.54186   22589.89258     -3.55042        0.24175      90.00000
!    Zero       Code       Dtt1       Code       Dtt2       Code  Dtt_1overd    Code  2ThetaBank  -> Patt#  1
!    1.58655    11.00     0.00000     0.00     0.00000     0.00     0.00000     0.00     90.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:  26.6028
!-------------------------------------------------------------------------------
# CRYSTAL DATA
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0   0   0   0   0   0       1642.504   0   9   0
!
!
P n m a                    <--Space group symbol
!Atom   Typ       X        Y        Z      Biso       Occ     In Fin N_t Spc /Codes
Sb      Sb      0.02910  0.25000  0.65879  0.31383   0.50000   0   0   0   0
                  0.00     0.00     0.00     0.00      0.00
Cr      Cr      0.15735  0.25000  0.04497  0.24748   0.50000   0   0   0   0
                  0.00     0.00     0.00     0.00      0.00
Se3     Se      0.17073  0.25000  0.48381  0.36833   0.50000   0   0   0   0
                  0.00     0.00     0.00     0.00      0.00
Se2     Se      0.28660  0.25000  0.21393  0.38468   0.50000   0   0   0   0
                  0.00     0.00     0.00     0.00      0.00
Se1     Se      0.50111  0.25000  0.60835  0.36807   0.50000   0   0   0   0
                  0.00     0.00     0.00     0.00      0.00
!-------> Profile Parameters for Pattern #   1  ----> Phase #   1
!  Scale        Extinc       Bov      Str1      Str2      Str3    Strain-Mode
  14.29689      0.0000    0.0000    0.0000    0.0000    0.0000         0
      0.00000      0.00      0.00      0.00      0.00      0.00
!     Sigma-2      Sigma-1      Sigma-0      Sigma-Q   Iso-GStrain    Iso-GSize    Ani-LSize   Size-Model
!  Instrumental Sigma interpolated from list in IRF file (Only sample contribution is refined)
        0.0000       0.0000       0.0000       0.0000       0.0000       0.0000       0.0000        0
          0.00         0.00         0.00         0.00         0.00         0.00         0.00
!     Gamma-2      Gamma-1      Gamma-0   Iso-LorStrain   Iso-LorSize
!  Instrumental Gamma interpolated from list in IRF file (Only sample contribution is refined)
       -4.4882      41.5199     -10.5597       0.0000       0.0000
          0.00         0.00         0.00         0.00         0.00
!     a         b         c       alpha      beta       gamma      #Cell Info
   9.149680   3.783304  13.330873  90.000000  90.000000  90.000000
    0.00000    0.00000    0.00000    0.00000    0.00000    0.00000
!     Pref1      Pref2      alph0      beta0      alph1      beta1      alphQ      betaQ
!  Instrumental alpha & beta interpolated from list in IRF file (Only shifts can be refined)
    0.000000   0.000000   0.000000   0.000000   0.000000   0.000000   0.000000   0.000000
        0.00       0.00       0.00       0.00       0.00       0.00       0.00       0.00
!Absorption correction parameters
   0.00000    0.00   0.00000    0.00            ABS: ABSCOR1  ABSCOR2
!  2Th1/TOF1    2Th2/TOF2  Pattern to plot
   14300.172  225000.000       1
```

External EdPCR Text Editor - [C:\Users\qzg\Dropbox (ORNL)\Workshops\MagneticStructure2022\FullProf\CrSbSe3_N_M_10K.pcr]

File Edit Search

```
!     -15.12841   22589.89258     -3.55042        0.24175      90.00000
!    Zero       Code       Dtt1       Code       Dtt2       Code  Dtt_1overd    Code  2ThetaBank  -> Patt#  1
!    0.00000    0.00     0.00000     0.00     0.00000     0.00     0.00000     0.00     90.000
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:   0.0000  Magnetic R-Factor:   0.0000
!-------------------------------------------------------------------------------
Nuclear and Magnetic Structure of: 62p447   VARY mxmymz
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ     Nvk Npr More
   5   0   0 0.0 0.0 1.0  10   0   2   0   0         0.000   0   9   0
!
Pnm'a'  number:62.447     <--Magnetic Space group symbol (BNS symbol & number)
Transform to standard: a,b,c;0,0,0
Parent Space Group: Pnma     IT_number:   62
Transform from Parent: a,b,c;0,0,0
! Nsym    Cen  N_Clat N_Ant
    4      2     0     0
!
! Symmetry operators
  1 x,y,z,+1
  2 -x,y+1/2,-z,-1
  3 x+1/2,y,-z+1/2,-1
  4 x+1/2,-y+1/2,-z+1/2,+1
!
!Atom   Typ     Mag Vek       X         Y         Z       Biso      Occ    N_type  Spc/Fftype  /Line below:Codes
!       Rx       Ry        Rz        Ix        Iy        Iz      MagPh  / Line below:Codes
!      beta11   beta22   beta33   beta12   beta13   beta23  / Line below:Codes
Sb      Sb        1  0   0.02910   0.25000   0.65879   0.00000   0.50000     0     0  #
                              0.00      0.00      0.00      0.00      0.00
Cr      MCR2      1  0   0.15735   0.25000   0.04497   0.00000   0.50000     1     0  #
                              0.00      0.00      0.00      0.00      0.00
     1.00000   0.00000   2.00000   0.00000   0.00000   0.00000   0.00000 <-MagPar
       21.00      0.00     11.00      0.00      0.00      0.00      0.00
Se3     Se        1  0   0.17073   0.25000   0.48381   0.00000   0.50000     0     0  #
                              0.00      0.00      0.00      0.00      0.00
Se2     Se        1  0   0.28660   0.25000   0.21393   0.00000   0.50000     0     0  #
                              0.00      0.00      0.00      0.00      0.00
Se1     Se        1  0   0.50111   0.25000   0.60835   0.00000   0.50000     0     0  #
                              0.00      0.00      0.00      0.00      0.00
!-------> Profile Parameters for Pattern #   1  ----> Phase #   1
!  Scale        Extinc       Bov      Str1      Str2      Str3    Strain-Mode
  1.000000      0.0000    0.0000    0.0000    0.0000    0.0000         0
      0.00000      0.00      0.00      0.00      0.00      0.00
!     Sigma-2      Sigma-1      Sigma-0      Sigma-Q   Iso-GStrain    Iso-GSize    Ani-LSize   Size-Model
!  Instrumental Sigma interpolated from list in IRF file (Only sample contribution is refined)
        0.1760      -0.1978       0.0915       0.0000       0.0301       0.0000       0.0000        0
          0.00         0.00         0.00         0.00         0.00         0.00         0.00
!     Gamma-2      Gamma-1      Gamma-0   Iso-LorStrain   Iso-LorSize
!  Instrumental Gamma interpolated from list in IRF file (Only sample contribution is refined)
        0.0000       0.0000       0.0000       0.0000       0.0000
          0.00         0.00         0.00         0.00         0.00
!     a         b         c       alpha      beta       gamma      #Cell Info
   9.149700   3.783300  13.330900  90.000000  90.000000  90.000000   #
    0.00000    0.00000    0.00000    0.00000    0.00000    0.00000
!     Pref1      Pref2      alph0      beta0      alph1      beta1      alphQ      betaQ
!  Instrumental alpha & beta interpolated from list in IRF file (Only shifts can be refined)
    1.000000   0.000000   0.000000   0.000000   0.000000   0.000000   0.000000   0.000000
        0.00       0.00       0.00       0.00       0.00       0.00       0.00       0.00
!Absorption correction parameters
   0.00000    0.00   0.00000    0.00            ABS: ABSCOR1  ABSCOR2
!  2Th1/TOF1    2Th2/TOF2  Pattern to plot
       2.000    130.000       1
```

Change MCR2 to MCR3

B parameters were reset to zero by Bilbao server when generating the mcif file!

Only refine scale factors, B factors and moment sizes

Atoms Information: Phase 1

List of Atoms

Number of Atoms: 5

	Label	Ntyp	Mag. Rot.	Prog. V...	X	Y	Z	B	Occ
Atom # 1	Sb	Sb	1	0	0.02910	0.25000	0.65879	0.00000	0.50000
Atom # 2	Cr	MCR3	1	0	0.15735	0.25000	0.04497	0.00000	0.50000
Atom # 3	Se3	Se	1	0	0.17073	0.25000	0.48381	0.00000	0.50000
Atom # 4	Se2	Se	1	0	0.28660	0.25000	0.21393	0.00000	0.50000

	Re[x]	Re[y]	Re[z]	Im[x]	Im[y]	Im[z]	MPhase
Atom #2	1.00000	0.00000	2.00000	0.00000	0.00000	0.00000	0.00000

Refine Positions

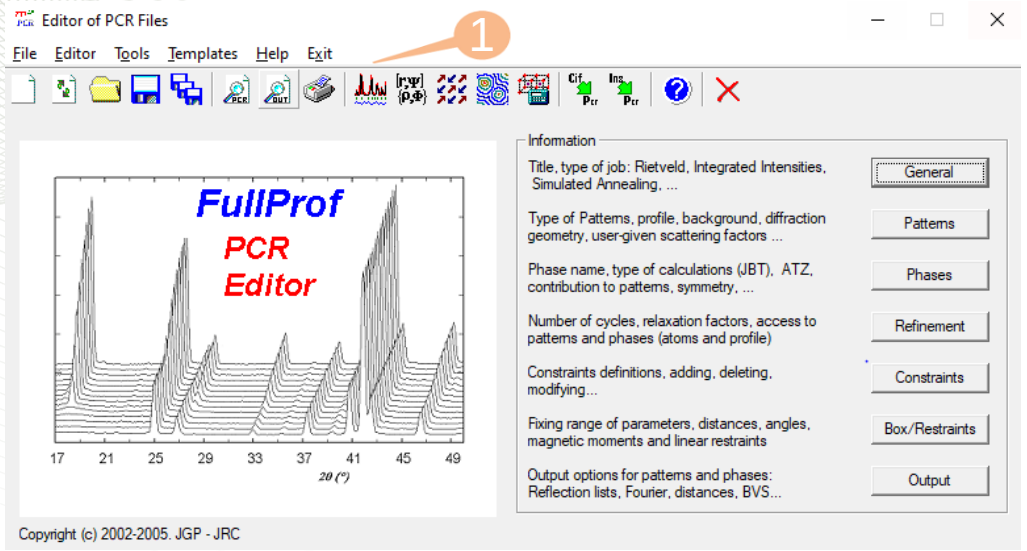
Refine B_iso

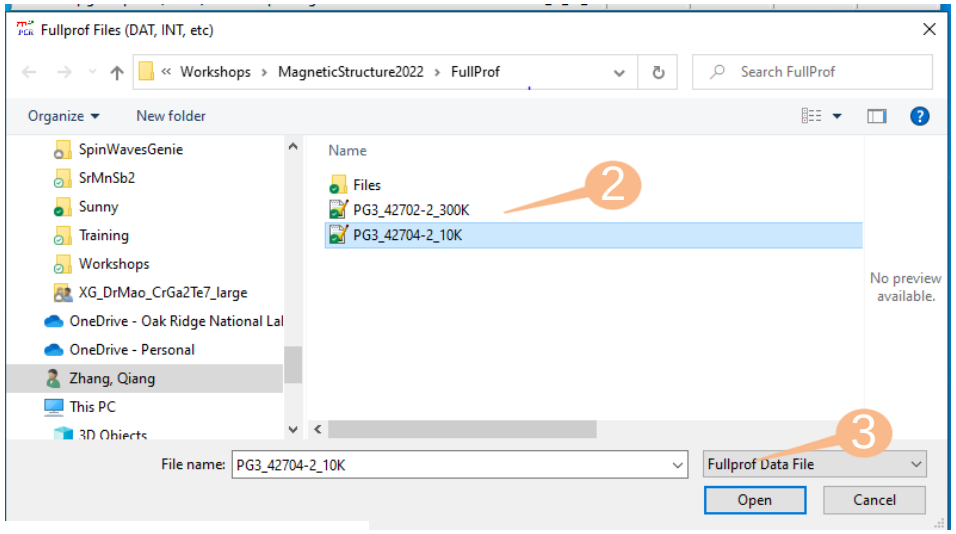
Refine B_aniso

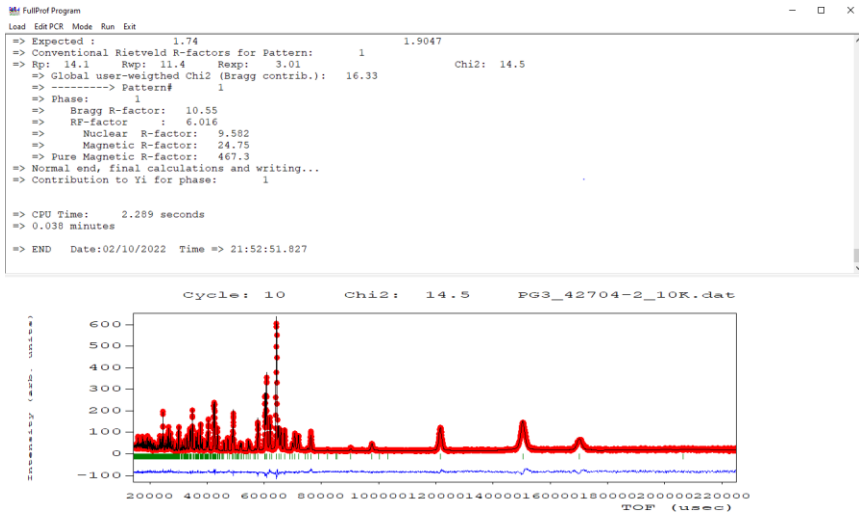
Fix All

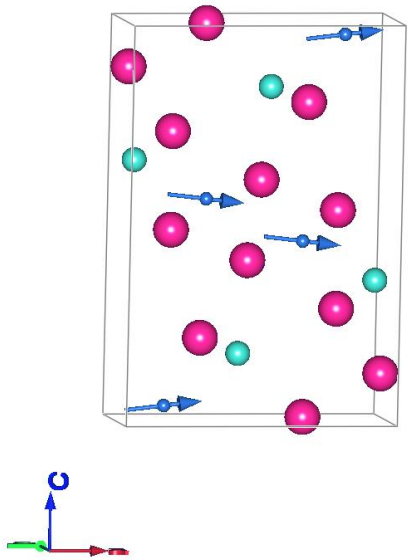
Cancel

OK









Canted order:

FM alignment along the *a* axis,
AFM alignment along the *c* axis.

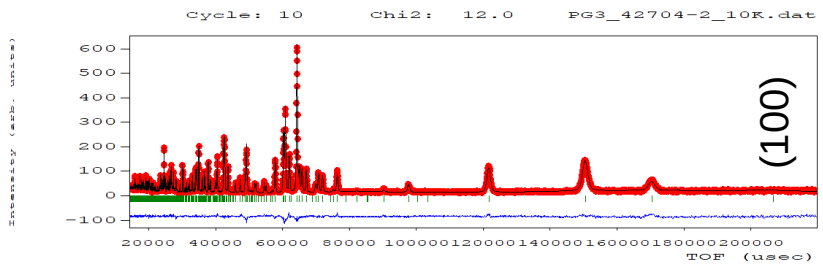
Add the refinement on lattice constants, Gam_2, Gam_1 and Gam_0, to optimize the refinement

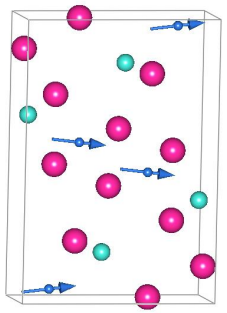
	Re[x]		Re[y]		Re[z]	
Atom #2	2.61694	☑	0.00000	☐	0.27388	☑

VIII. Check if small Mz (AFM component) is real

	Re[x]		Re[y]		Re[z]	
Atom #2	2.61694	✔	0.00000	☐	0.27388	✔

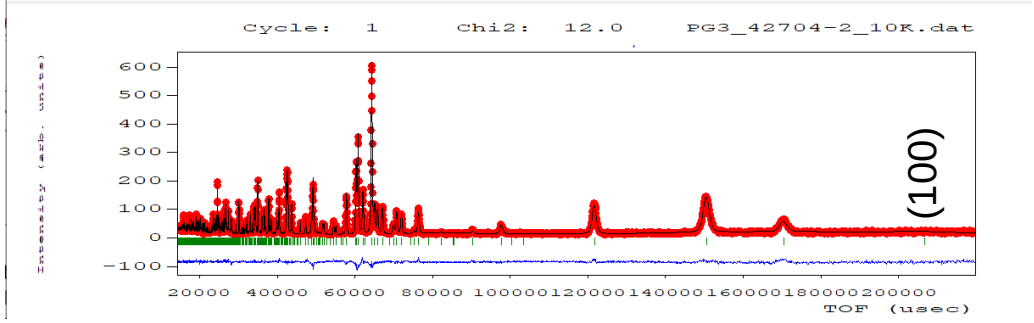
```
=> Rp:  12.3     Rwp:  9.62     Rexp:    2.78                    Chi2:  12.0
   => Global user-weigthed Chi2 (Bragg contrib.):   11.96
   => ---------> Pattern#        1
   => Phase:        1
   =>    Bragg R-factor:   8.718
   =>    RF-factor      :   3.284
   =>      Nuclear  R-factor:   8.718
   =>      Magnetic R-factor:   16.54
   => Pure Magnetic R-factor:  0.2833E-06
=> Normal end, final calculations and writing...
=> Contribution to Yi for phase:        1

=> CPU Time:     2.316 seconds
=> 0.039 minutes

=> END   Date:30/09/2022  Time => 11:41:27.740
```





	Re[x]		Re[y]		Re[z]	
Atom #2	2.63514	✔	0.00000	☐	0.00000	☐

```
   => Phase:        1
   =>    Bragg R-factor:   8.716
   =>    RF-factor      :   3.284
   =>      Nuclear  R-factor:   8.716
   =>      Magnetic R-factor:   16.53
   => Pure Magnetic R-factor:  0.2832E-06
=> Normal end, final calculations and writing...
=> Contribution to Yi for phase:        1

=> CPU Time:     2.336 seconds
=> 0.039 minutes

=> END   Date:30/09/2022  Time => 11:52:13.236
```

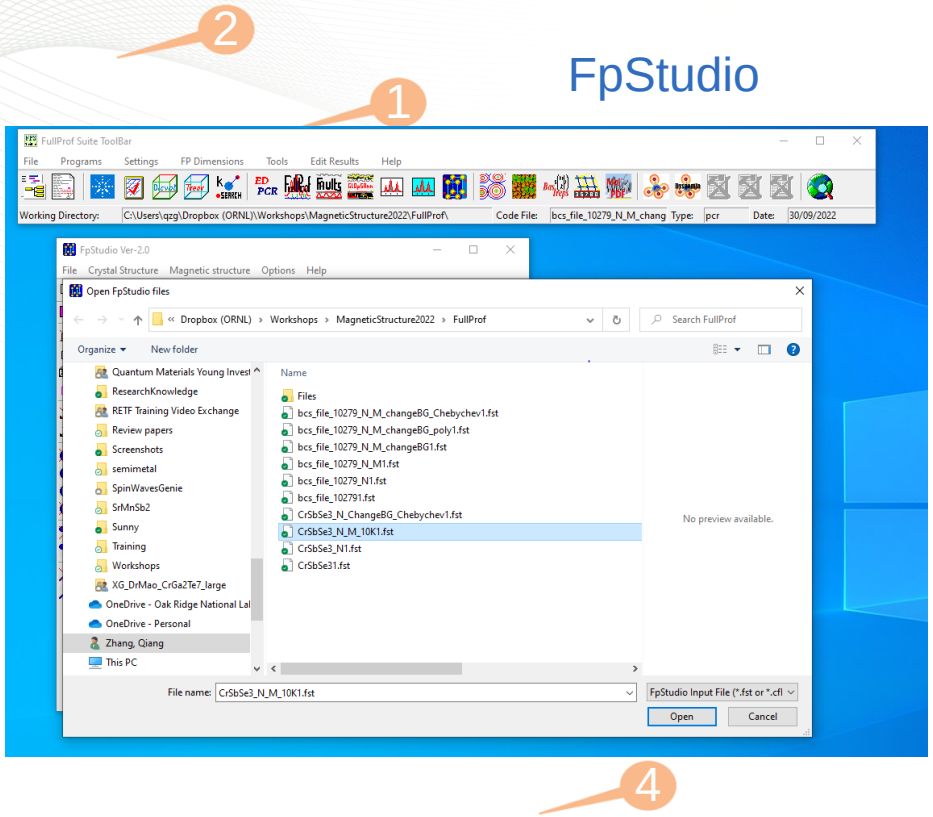


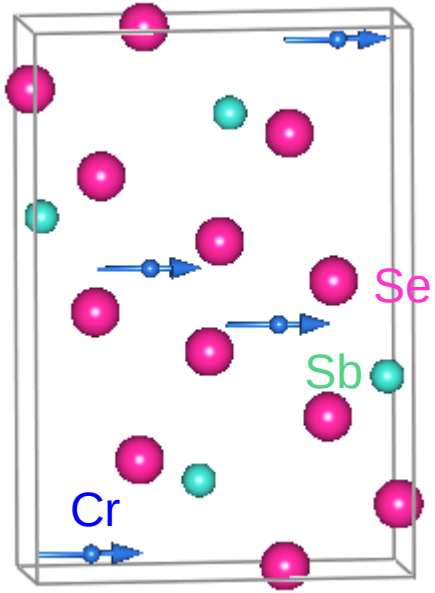
Two reasons why the small AFM coefficient C2 should be zero:

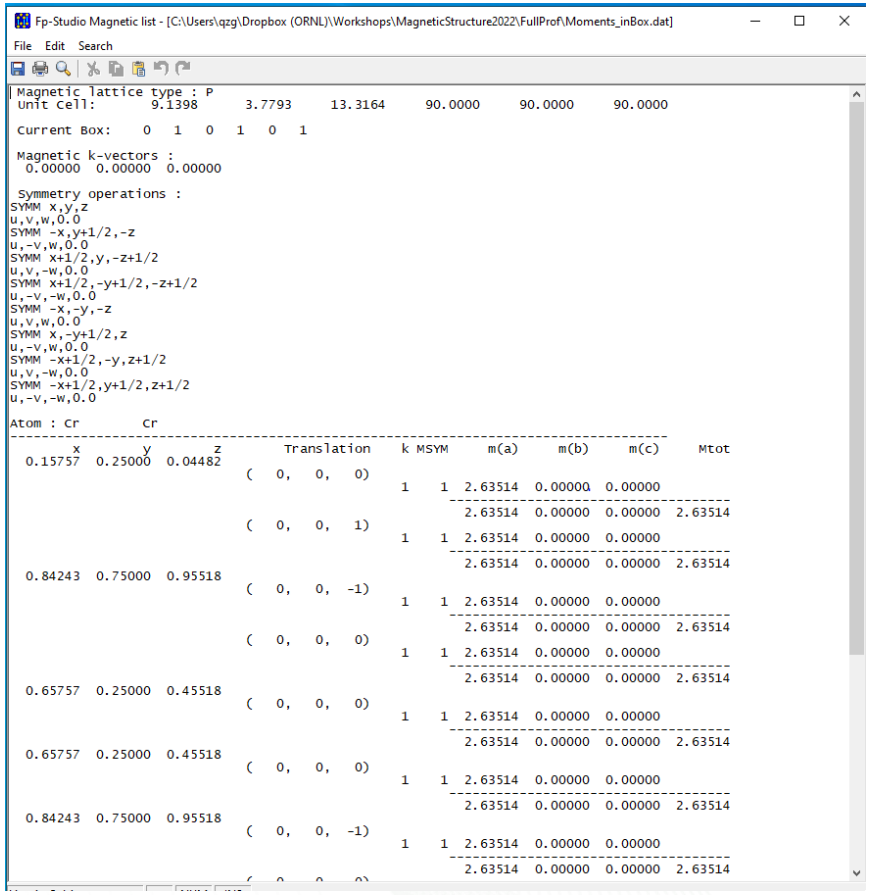
1). The refinement quality is similar using small C2 or zero C2;
2). The nonzero AFM coefficient C2 produces pure magnetic peak (100). The POWGN data shows there is no detectable (100) intensity, indicating that C2 should be zero.

IX. Display the magnetic structure using FpStudio or Vesta.

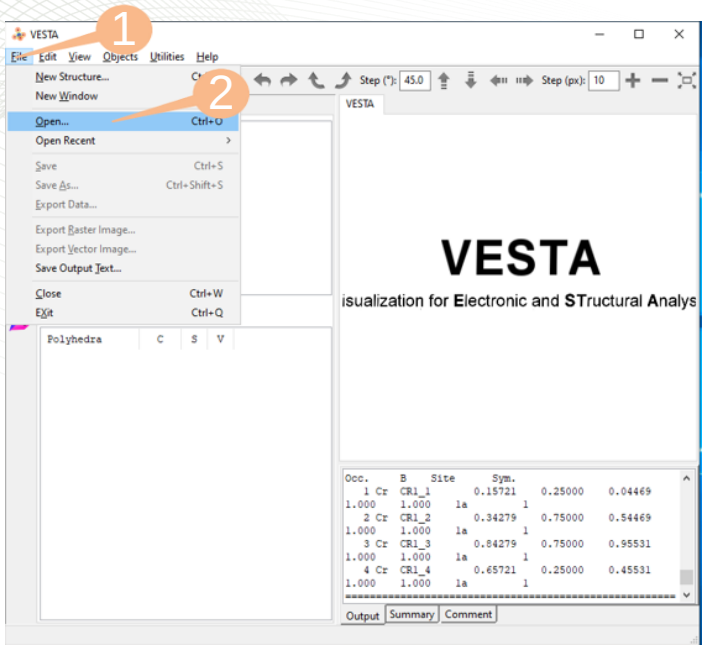
FpStudio

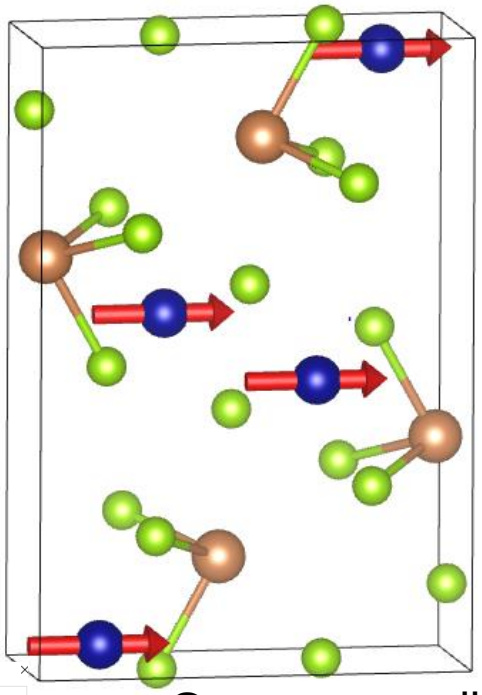


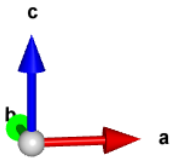


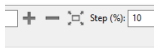


Vesta to open *.mcif file

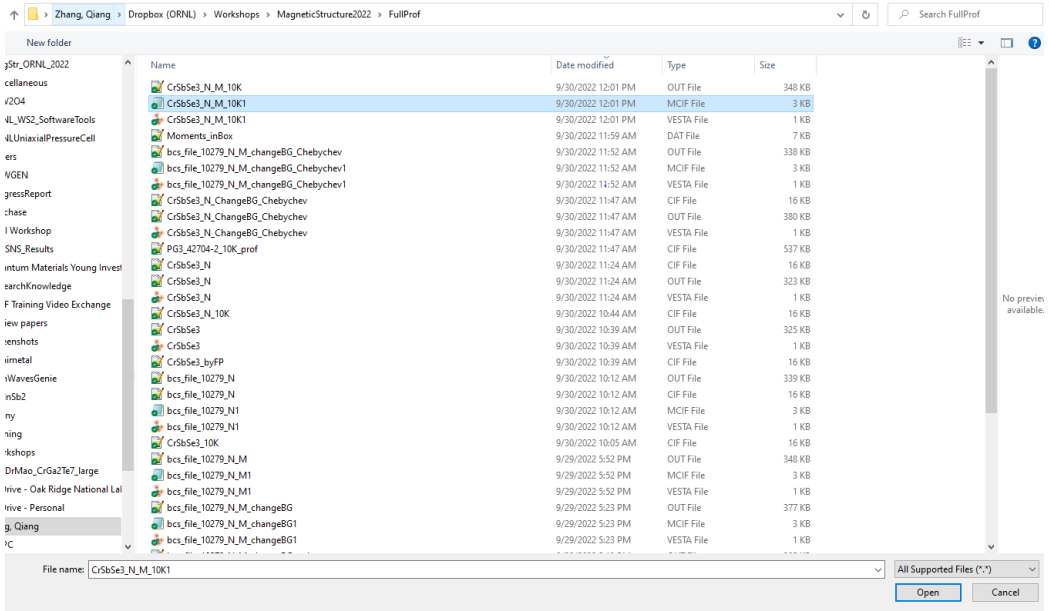


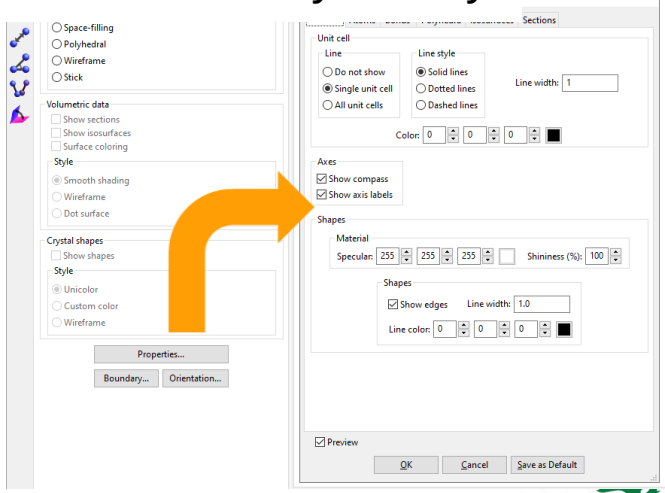






One may modify all these tabs to improve the figure





Magnetic Structure Determination from Neutron Diffraction Data (MagStr), October 3-7, 2022

Tutorial example for FullProf refinement of commensurate structure from TOF data

Please contact me if you have any question or comments. Thanks!

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