FullProf tutorial on crystal structure and commensurate magnetic structure

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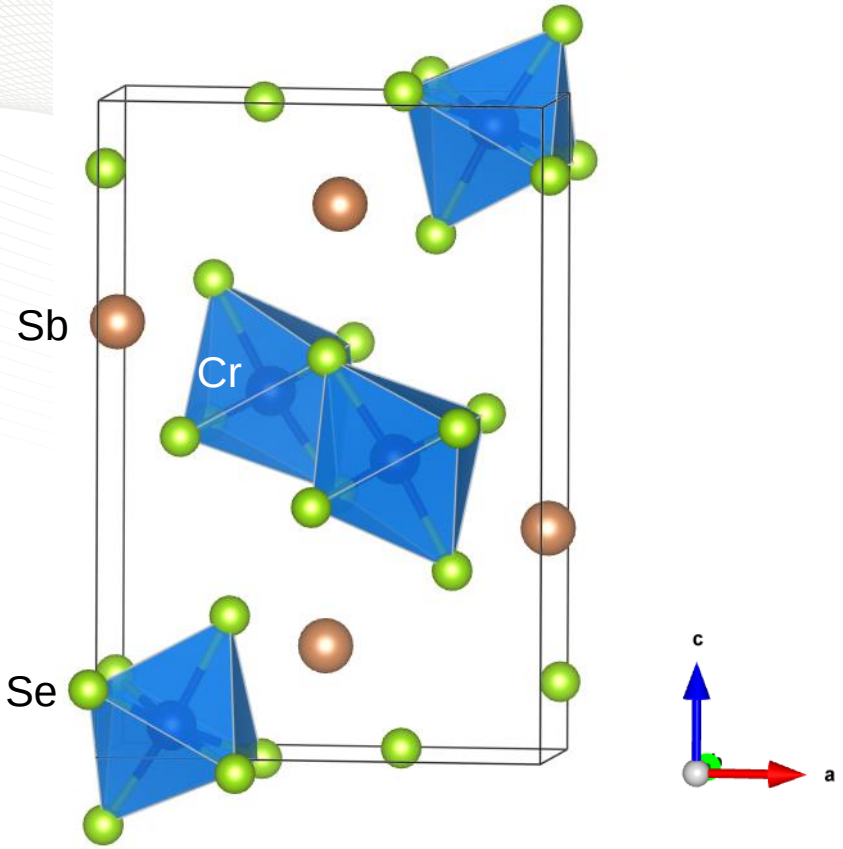
POWGEN workshop “Getting the Most from Your POWGEN Data”, June 15-17, 2023

$CrSbSe_3$



- 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
  PG3_42704-2_10K.pcr (representation analysis)

- Supporting information

FullProf_CM.PDF (step-by-step instructions)

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_1overD)/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:

$$\sigma^2 = {\sigma_0}^2 + {\sigma_1}^2 d^2 + {\sigma_2}^2 d^4 + \frac{{\sigma_q}^2}{d^2} \longrightarrow \sigma_q d \quad \text{GSASII used}$$

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

- 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  iresо: 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)

```
1   Instrumental Resolution Parameters for TOF (numerical look-up table) for POWGEN/SNS 2018 B cycle
2  ! To be used with functions NPROF=9 in FullProf (Res=5)
3  ! ------------------------------------------------  Bank 1
4  !  Type of profile function: back-to-back expon * pseudo-Voigt
5  NPROF   9
6  !       Tof-min(us)       step       Tof-max(us)
7  TOFRG   11308.69531       5  282062.50000
8  !             Dtt1              Dtt2       Dtt_1overD         Zero
9  D2TOF     22589.89258        -3.55042         0.24175       -15.12841
10 !     TOF-TWOTH of the bank
11 TWOTH      90.000
12 !       d-spacing       Sigma^2         Gamma           Alpha          Beta           Shift    for pattern # 1
13 LIST_SIG_GAM_ALF_BET_SHIFT  233
14          0.44348         1.21809         2.85332         0.39237        0.10474        0.00000
15          0.44515         1.21788         2.84730         0.39204        0.10578        0.00000
16          ...
17          0.89199         7.70333         3.42920         0.14657        0.10563        0.00000
18          ...
19          1.51101       757.12213        18.55641         0.11664        0.15918        0.00000
20          1.54497       844.20258        19.59661         0.11365        0.15412        0.00000
21          ...
22          2.56205      7234.60986        59.75310         0.06182        0.08379        0.00000
23          ...
24          5.12410    116786.14844       252.91258         0.02469        0.05172        0.00000
25          7.24657    462492.40625       465.01639         0.01463        0.04323        0.00000
26 END
27
```

TOF region and step

TOF to D

numerical coefficients

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

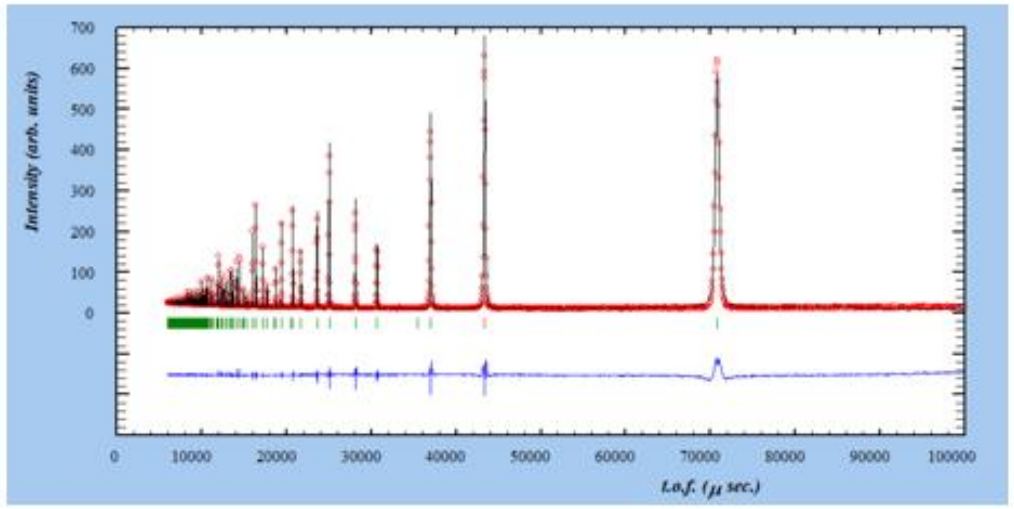
Allows a more accurate determination of instrumental resolution.

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

(a)



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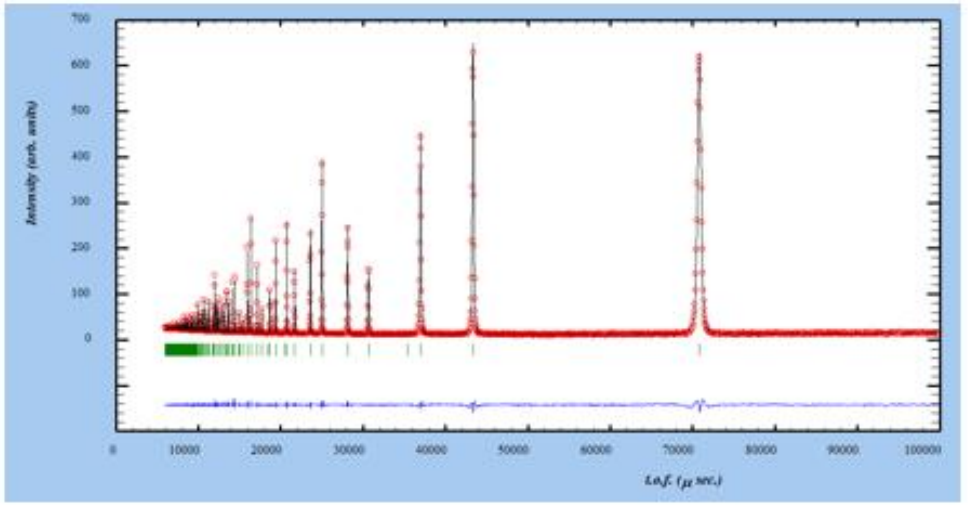

Same Si data at POWGEN

- 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 new version FullProf to refine POWGEN data for getting an improved refinement on the peakshape

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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 including the lattice constants, atomic positions, temperature factors and peak profile 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.

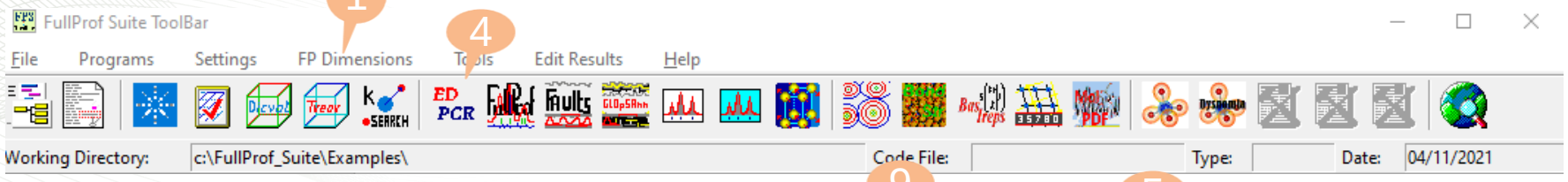
V. Symmetry analysis to obtain irreducible representations and Basic vectors using SARAh.

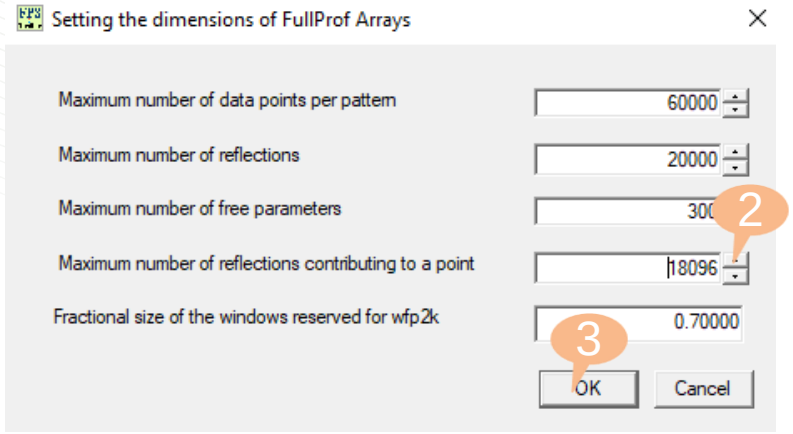
VI. Select a magnetic model and add it as the 2nd phase in the PCR.

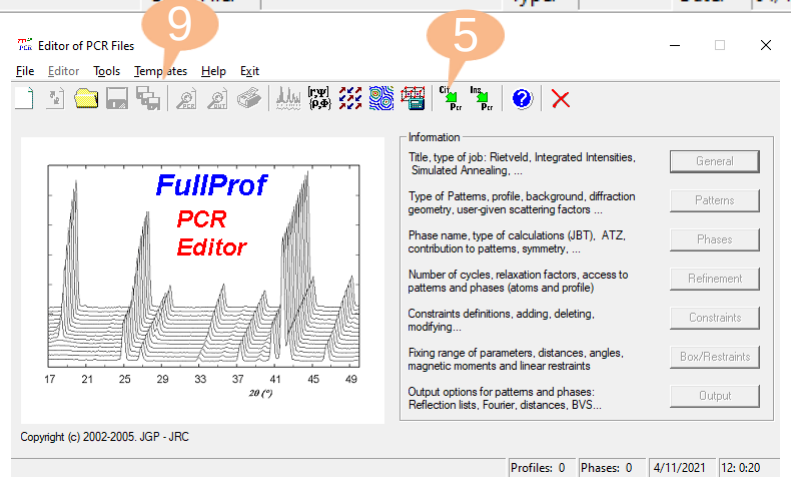
VII. Refine the magnetic phase to obtain the magnetic structure and ordered moment.

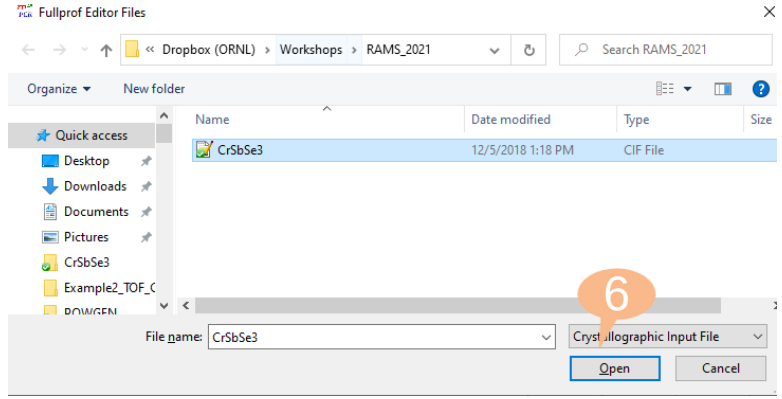
VIII. Display the magnetic structure using FpStudio and Vesta.

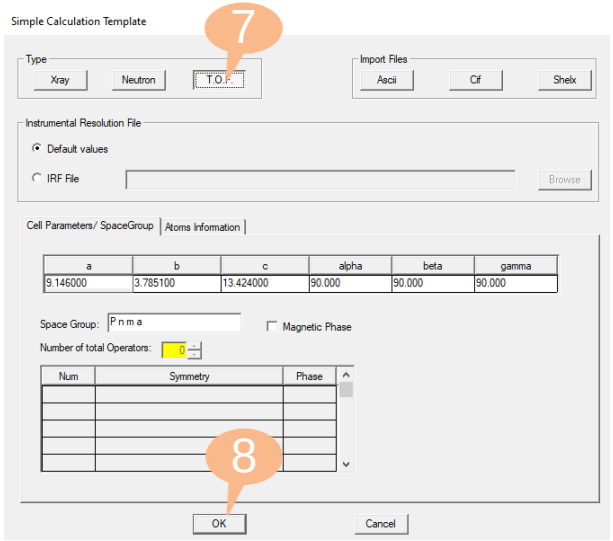
I. Import the cif file and create a PCR file for a single datafile at 300 K (T>Tm)

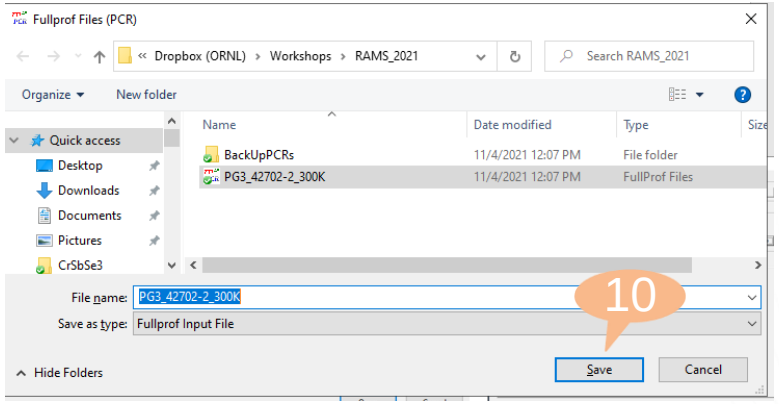










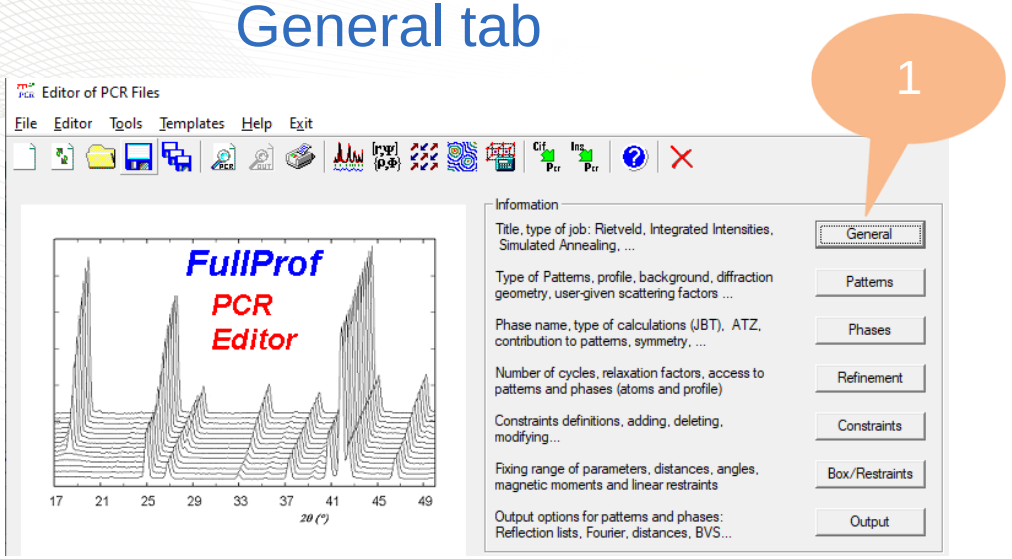


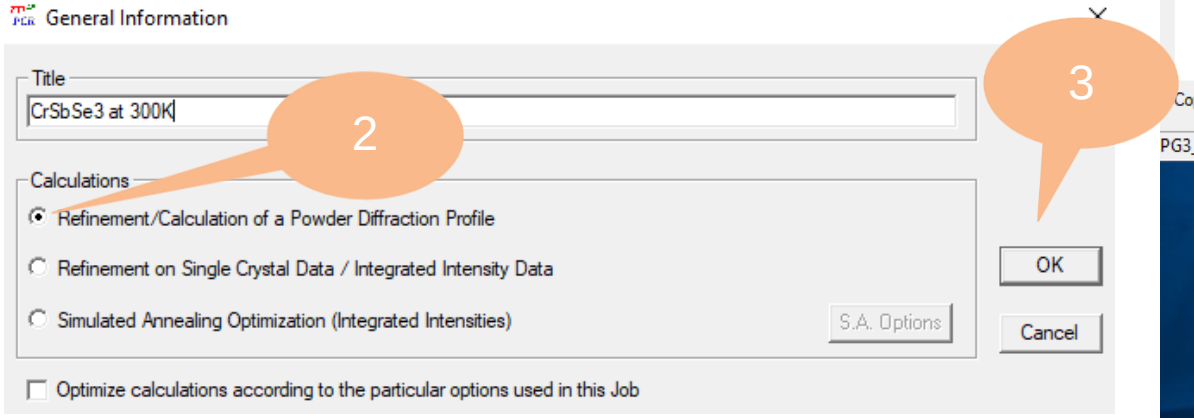
Important: check occ=
site multip/general multip

```
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
```

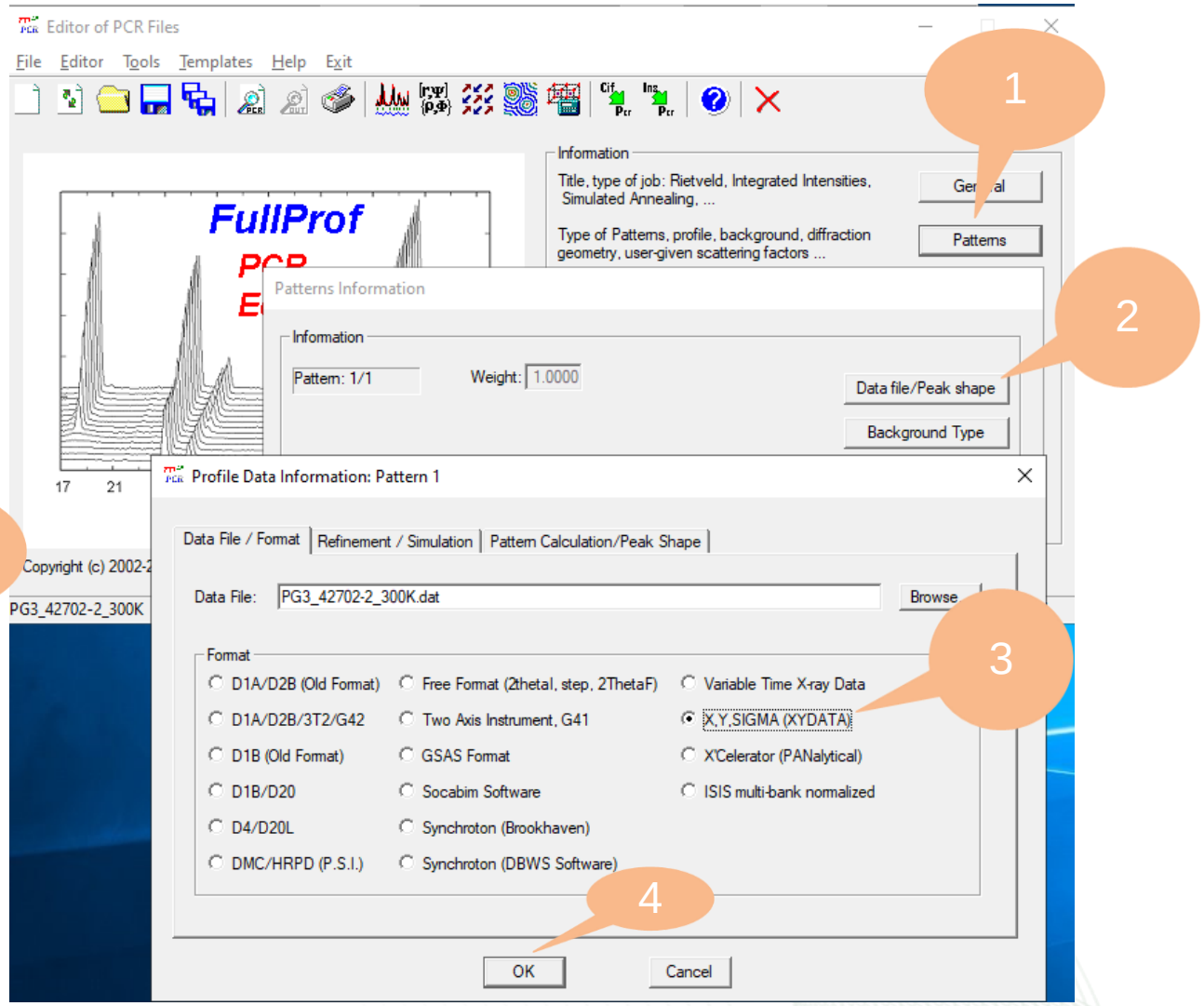
II. Refine the data to get accurate structural parameters at 300 K

General tab



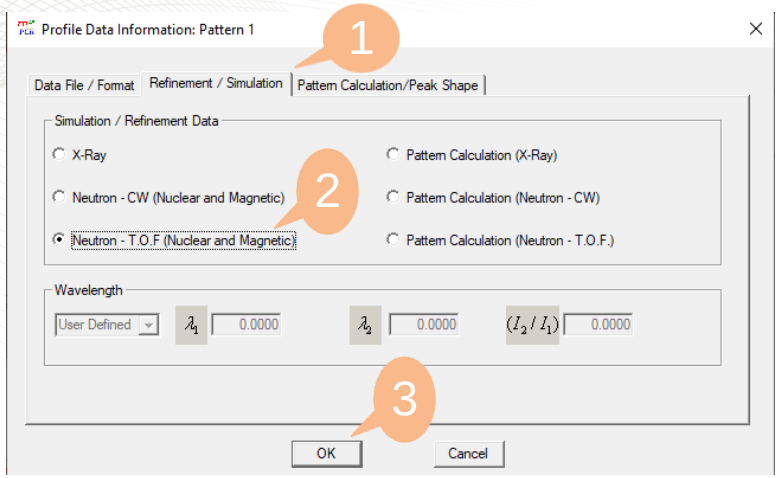


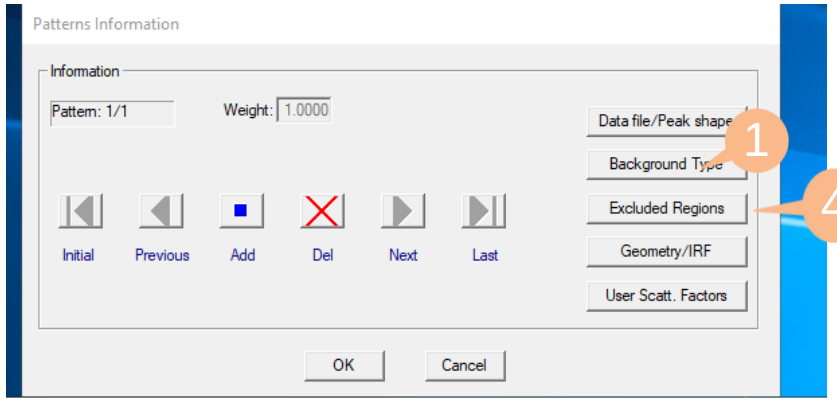
Pattern-Datafile/Peak shape tab

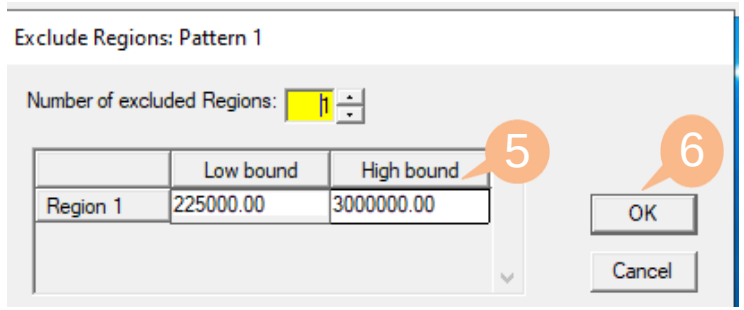


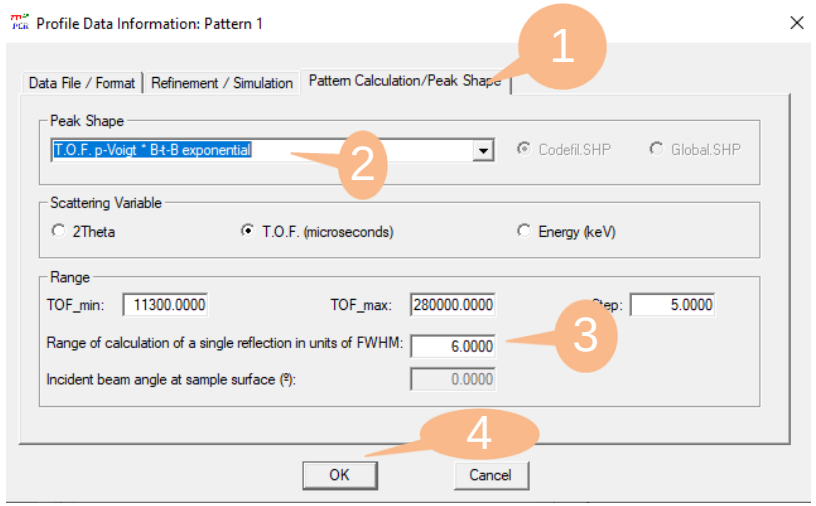
Pattern-Background Type

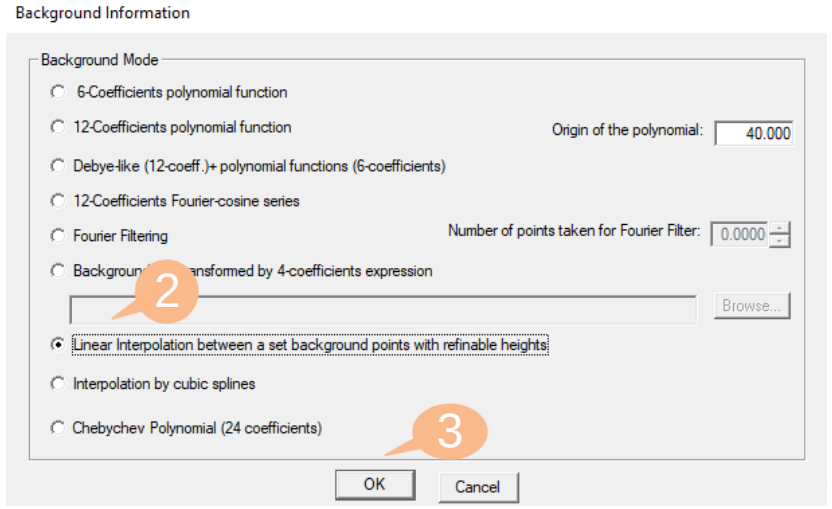
Pattern-Excluded Regions

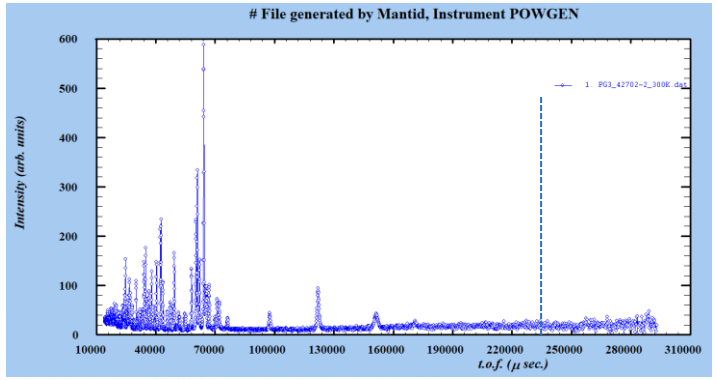




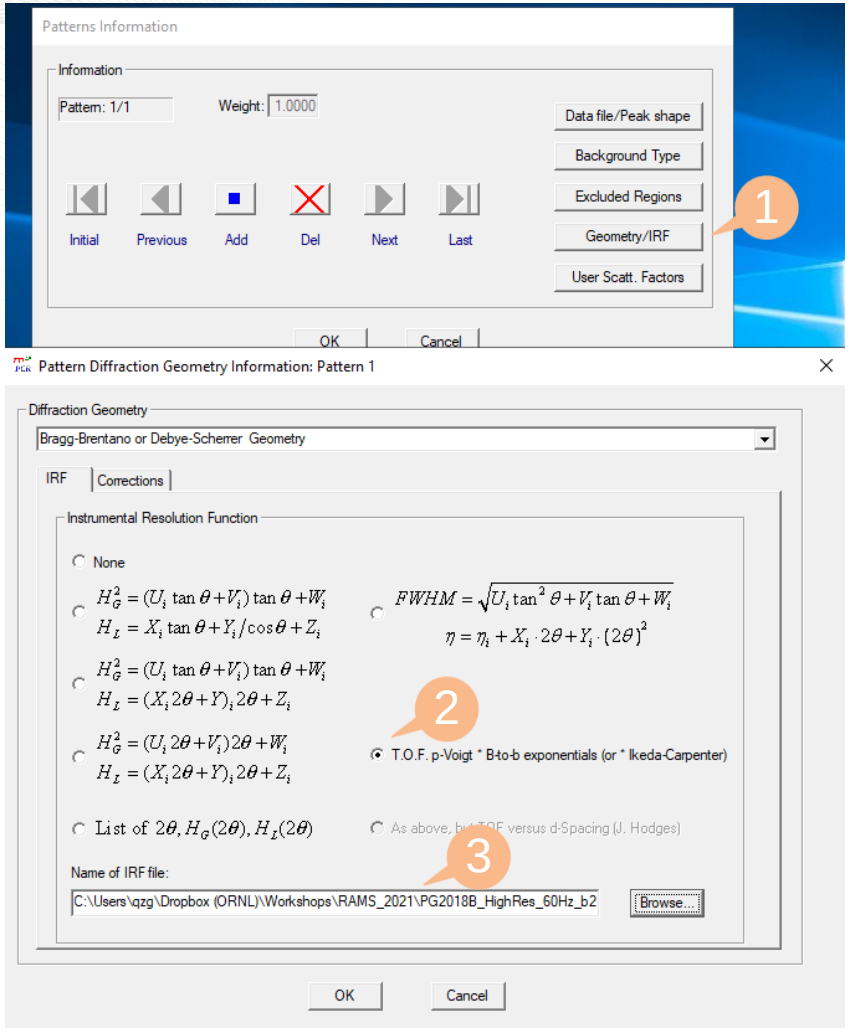




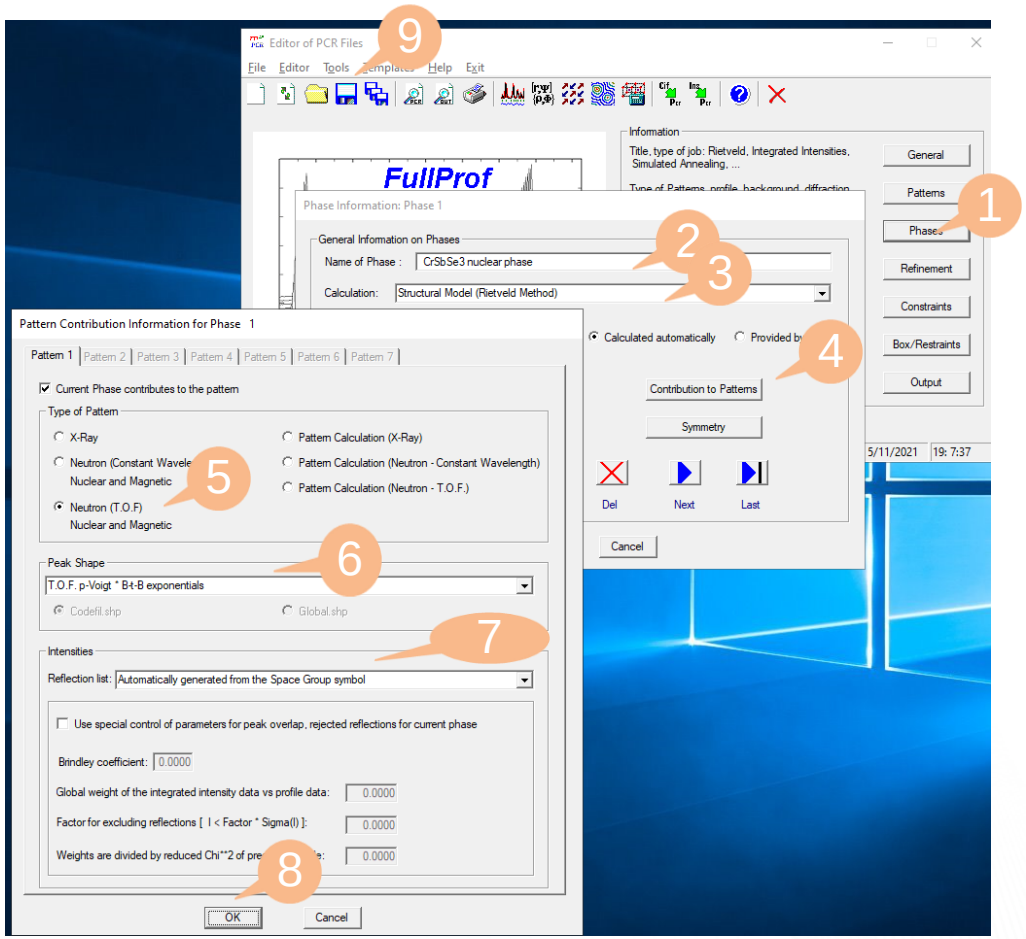




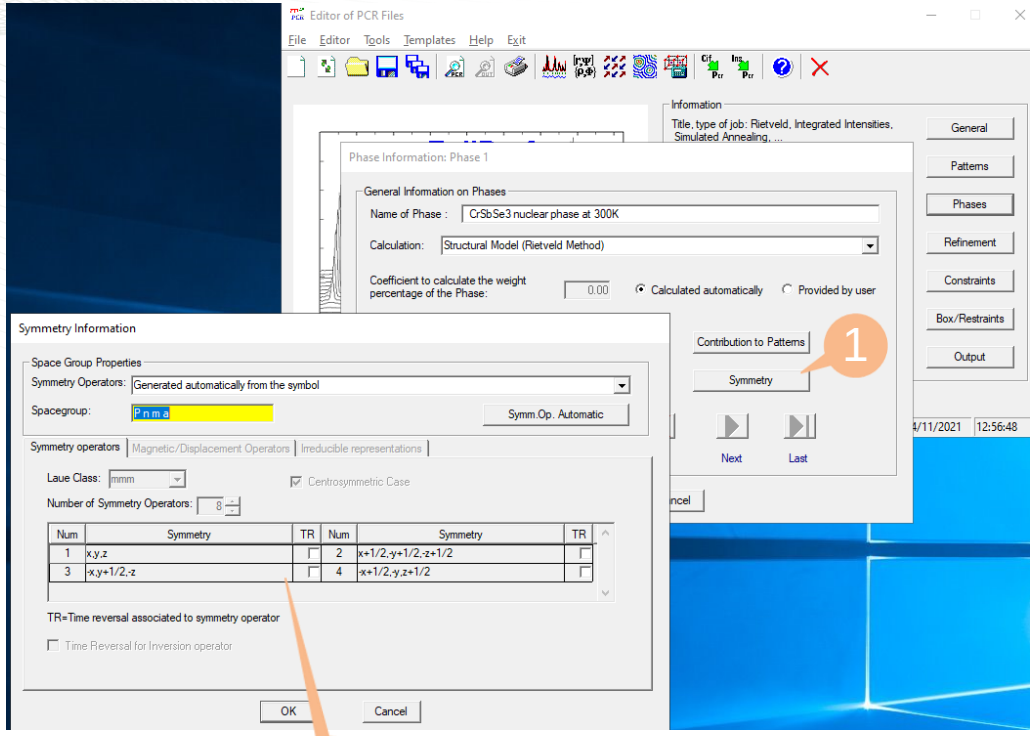
Pattern-Geometry/IRF tab



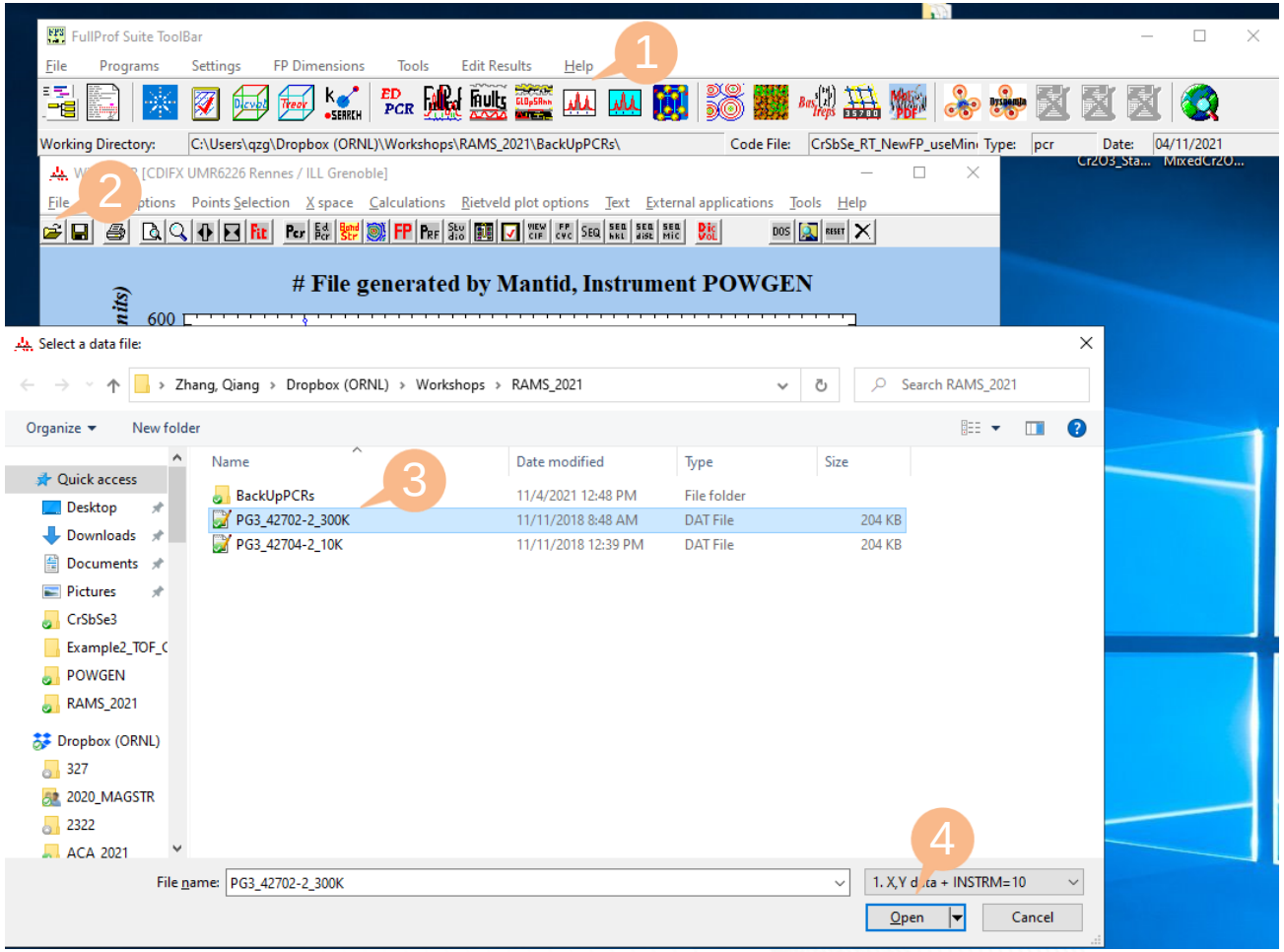
Phase-tab

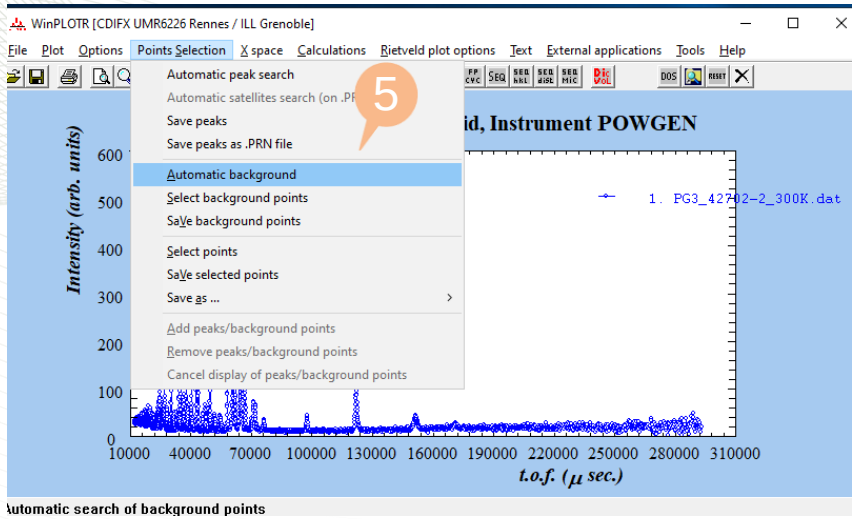


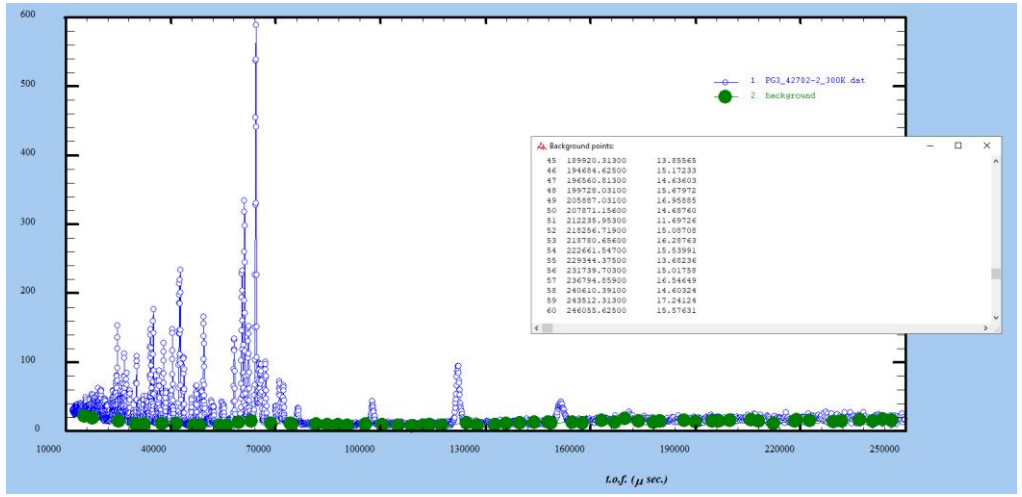
Use Winplotr to get the BG of the data



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 .PF
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.)
Automatic search of background points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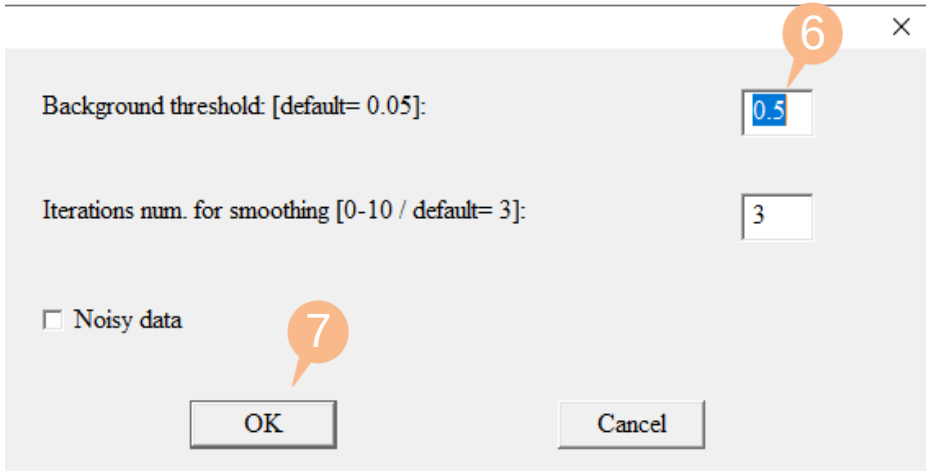
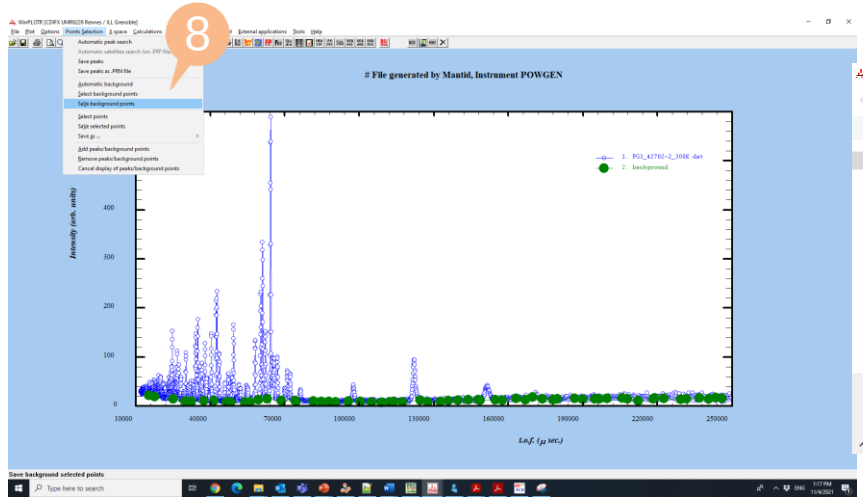
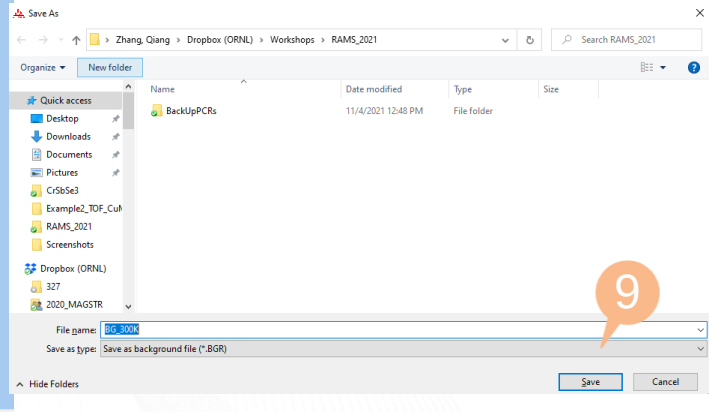
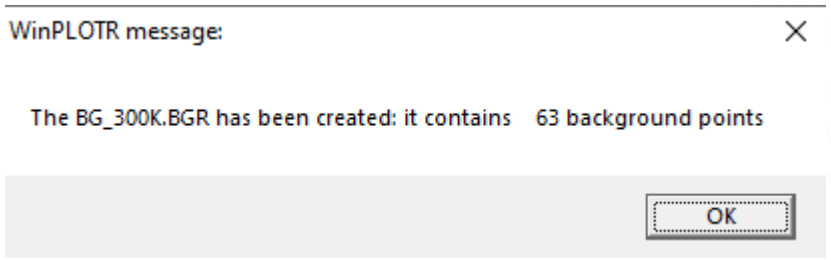
2. background
Background points:
t.o.f. (μ sec.)

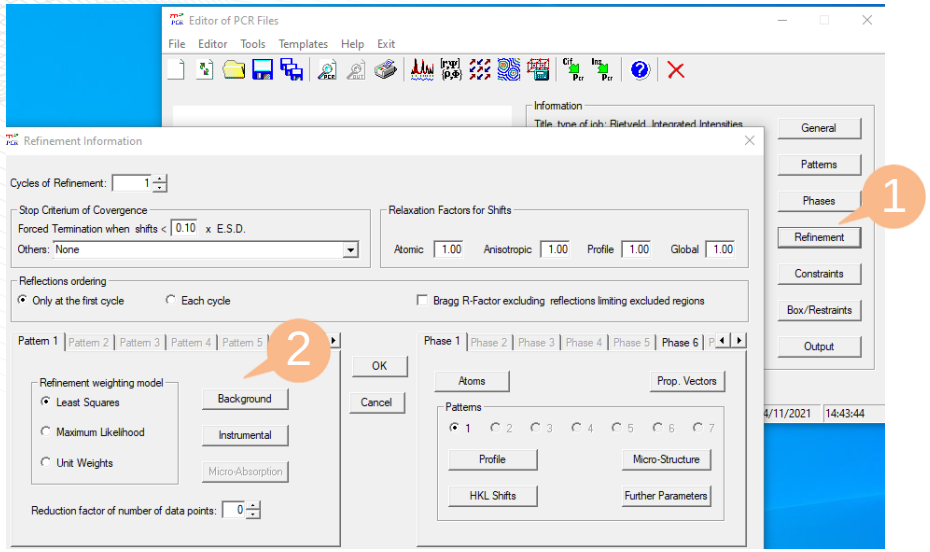

6
Background threshold: [default= 0.05]:
0.5
Iterations num. for smoothing [0-10 / default= 3]:
3
Noisy data
7
OK
Cancel

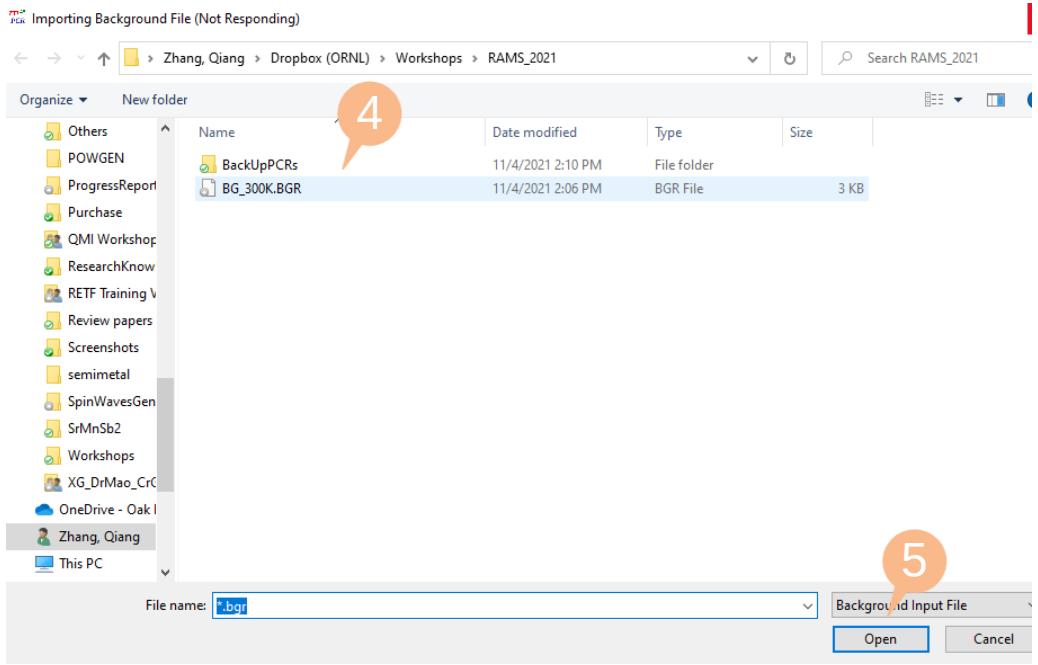

8
File generated by Mantid, Instrument POWGEN
t.o.f. (μ sec.)


Save As
Zhang, Qiang › Dropbox (ORNL) › Workshops › RAMS_2021
Search 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

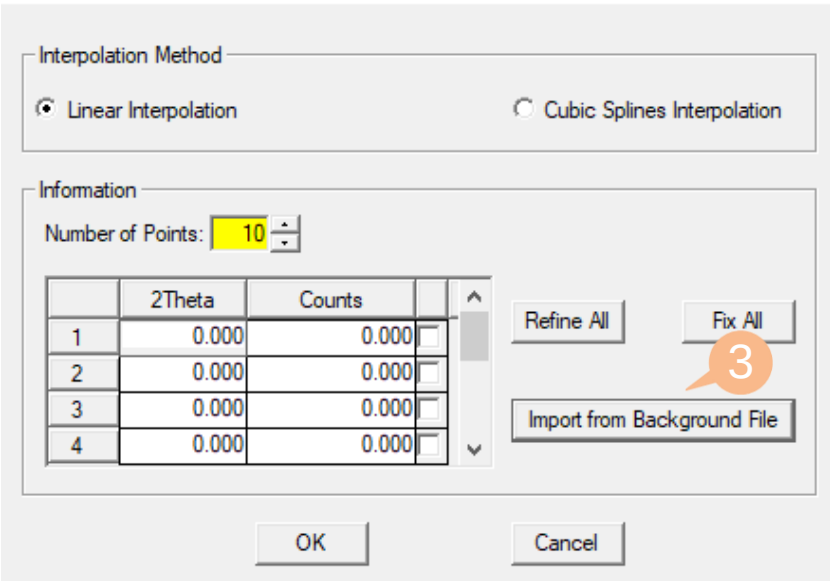

WinPLOTR message:
The BG_300K.BGR has been created: it contains 63 background points
OK

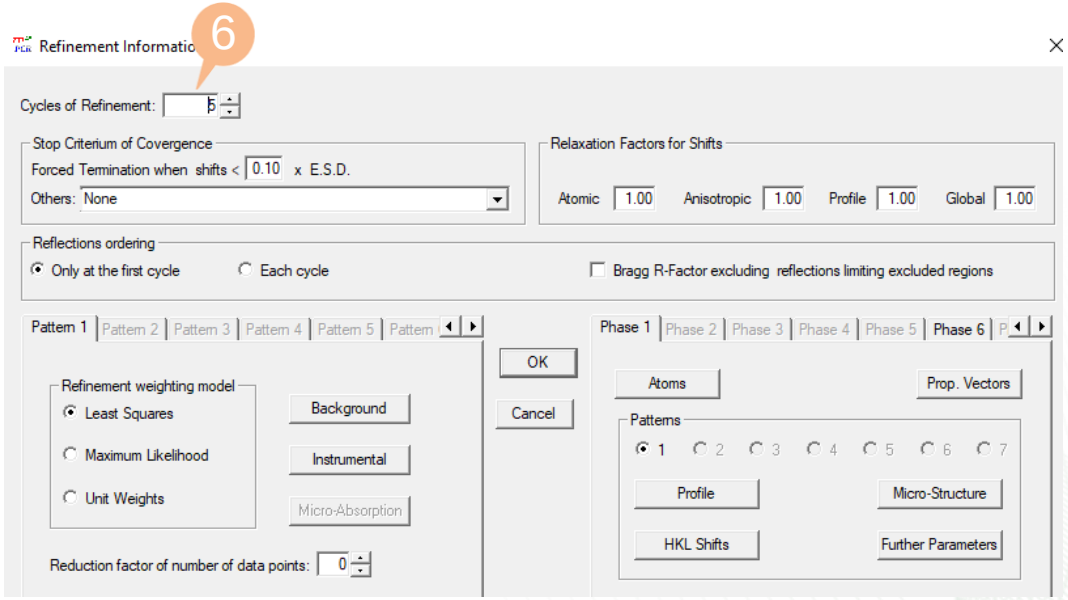
Refinement tab



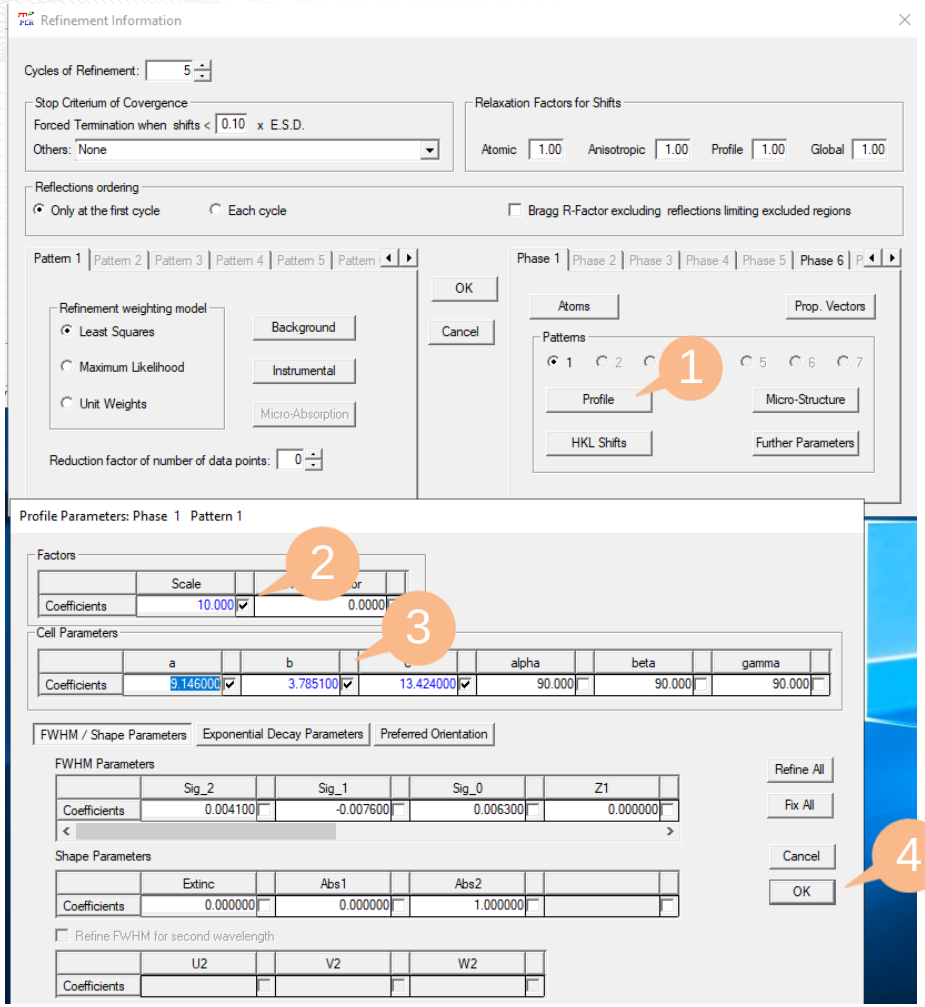


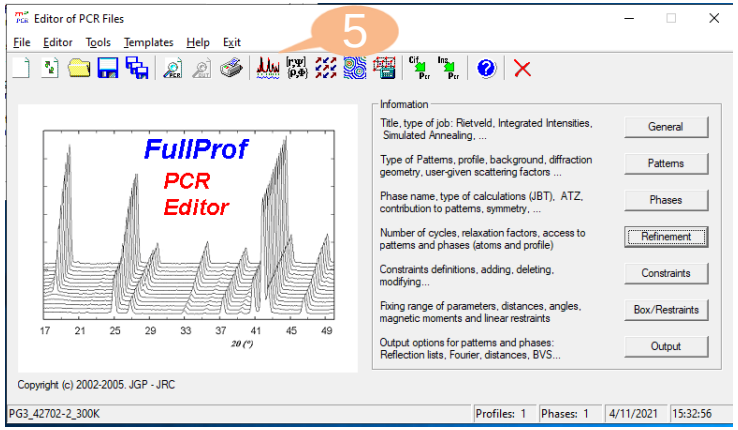
Linear interpolation between a set of Background Points: Pattern 1



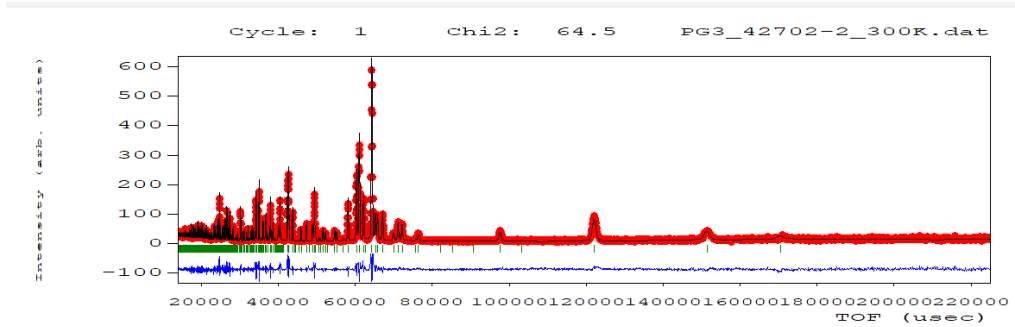


Refinement-Profile tab






```
   => 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

Profile Parameters: Phase 1 Pattern 1

Factors

	Scale	Overall B-factor
Coefficients	12.525	0.0000

Cell Parameters

	a	b	c	alpha	beta	gamma
Coefficients	9.144166	3.784407	13.416562	90.000	90.000	90.000

FWHM / Shape Parameters | Exponential Decay Parameters | Preferred Orientati

FWHM Parameters

	Gam_2	Gam_1	Gam_0	Gam_2
Coefficients	0.000	0.000	0.000	0.000000

Shape Parameters

	Extinc	Abs1	Abs2
Coefficients	0.000000	0.000000	1.000000

Refine FWHM for second wavelength

	U2	V2	W2
Coefficients			

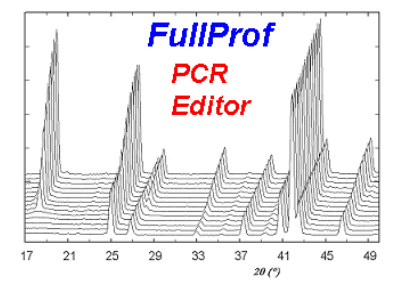
Refine All | Fix All | Cancel | OK

1 2

Editor of PCR Files

File Editor Tools Templates Help Exit

4



Information

Title, type of job: Rietveld, Integrated Intensities, Simulated Annealing, ... — General

Type of Patterns, profile, background, diffraction geometry, user-given scattering factors ... — Patterns

Phase name, type of calculations (JBT), ATZ, contribution to patterns, symmetry, ... — Phases

Number of cycles, relaxation factors, access to patterns and phases (atoms and profile) — Refinement

Constraints definitions, adding, deleting, modifying... — Constraints

Fixing range of parameters, distances, angles, magnetic moments and linear restraints — Box/Restraints

Output options for patterns and phases: Reflection lists, Fourier, distances, BVS... — Output

Copyright (c) 2002-2005. JGP - JRC

PG3_42702-2_300K | Profiles: 1 | Phases: 1 | 4/11/2021 | 15:32:56

```
   => 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 Information

Cycles of Refinement: 5

Stop Criterium of Covergence

Forced Termination when shifts < 0.10 x E.S.D.

Others: None

Relaxation Factors for Shifts

Atomic 1.00 | Anisotropic 1.00 | Profile 1.00 | Global 1.00

Reflections ordering

Only at the first cycle | Each cycle | Bragg R-Factor excluding reflections limiting excluded regions

Pattern 1 | Pattern 2 | Pattern 3 | Pattern 4 | Pattern 5 | Pattern

Refinement weighting model

Least Squares | Maximum Likelihood | Unit Weights

Background | Instrumental | Micro-Absorption

Reduction factor of number of data points: 0

OK | Cancel

3

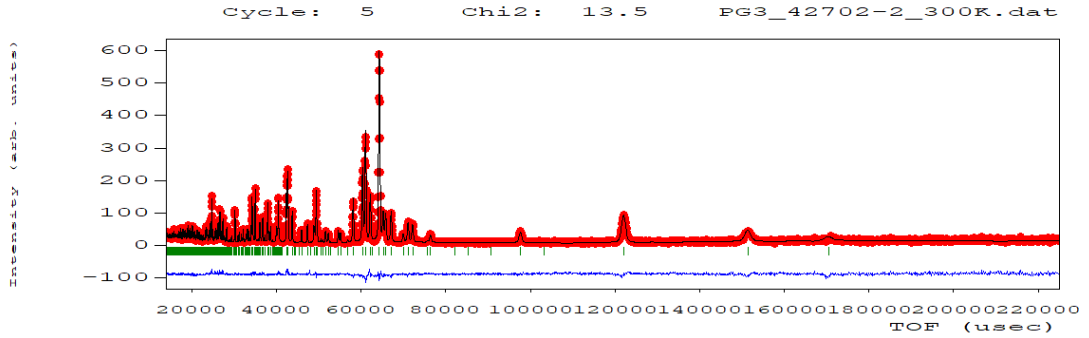
Phase | Phase 3 | Phase 4 | Phase 5 | Phase 6

Atoms | Prop. Vectors

Patterns

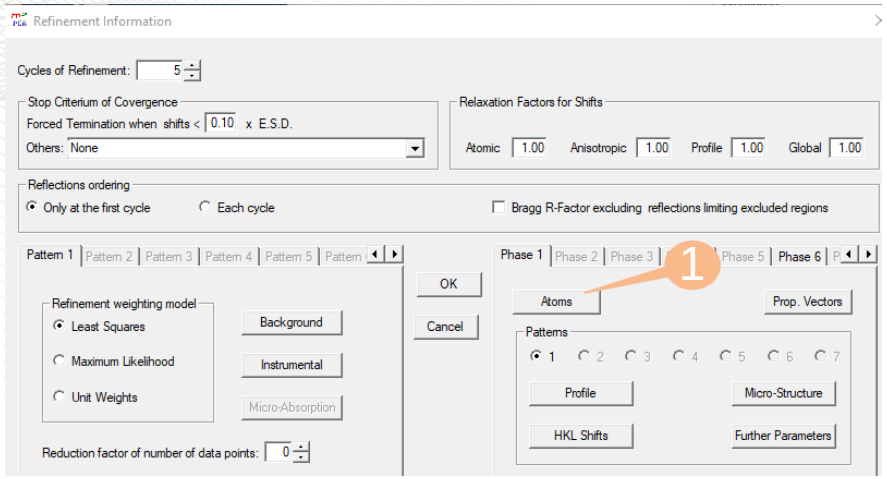
1 2 3 4 5 6 7

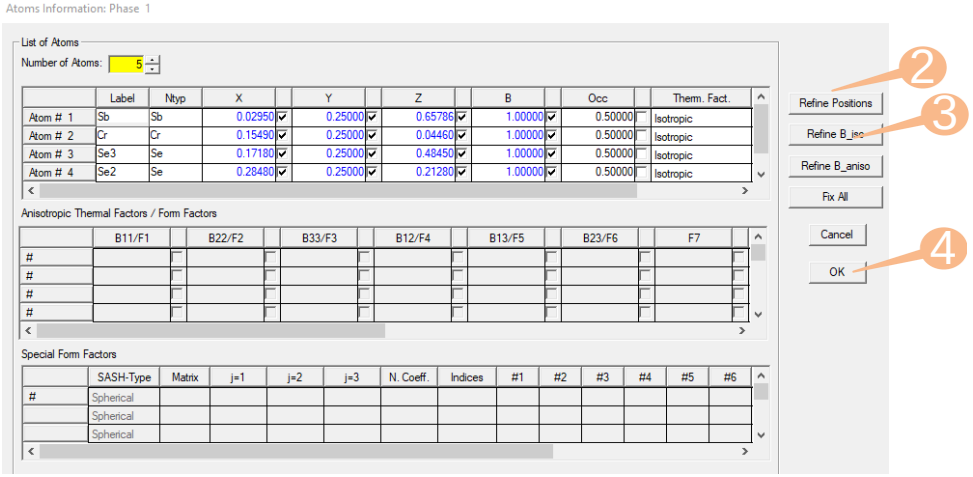
Profile | Micro-Structure | HKL Shifts | Further Parameters

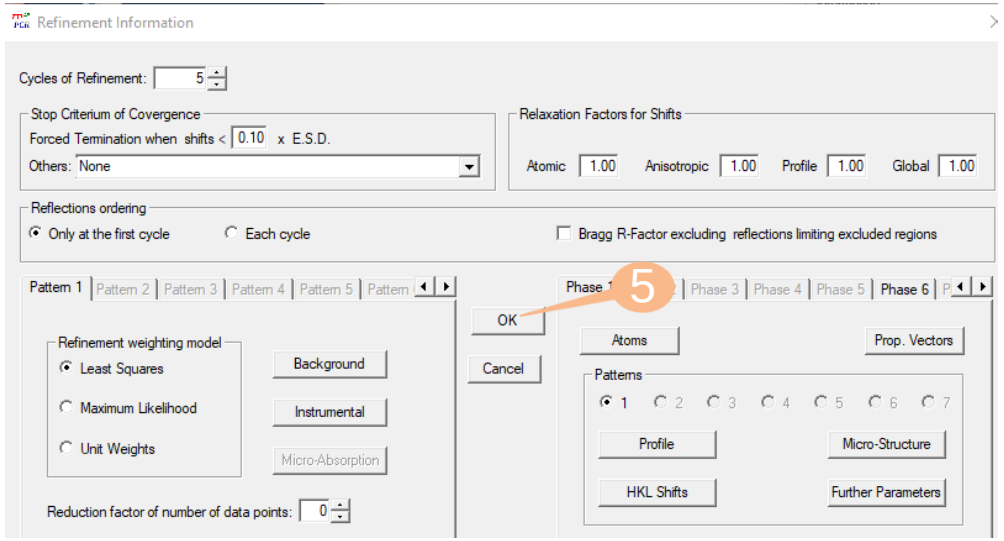


OAK RIDGE National Laboratory | HIGH FLUX ISOTOPE REACTOR | SPALLATION NEUTRON SOURCE

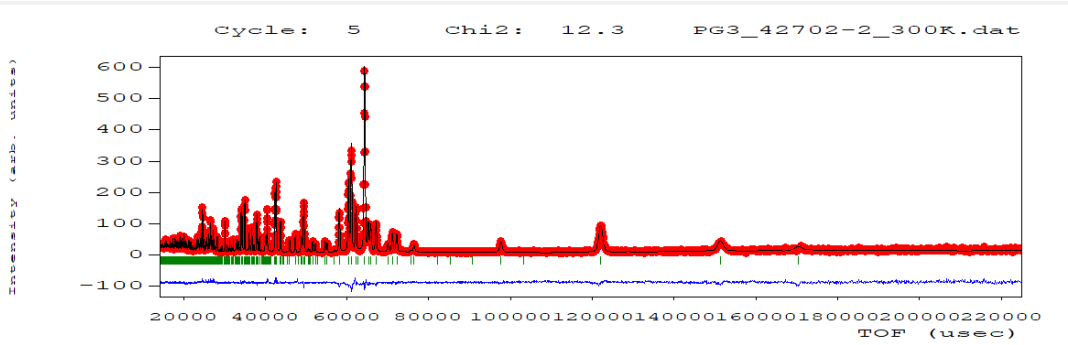
Refinement-Atoms tab (to refine atomic positions and temperature factors B)



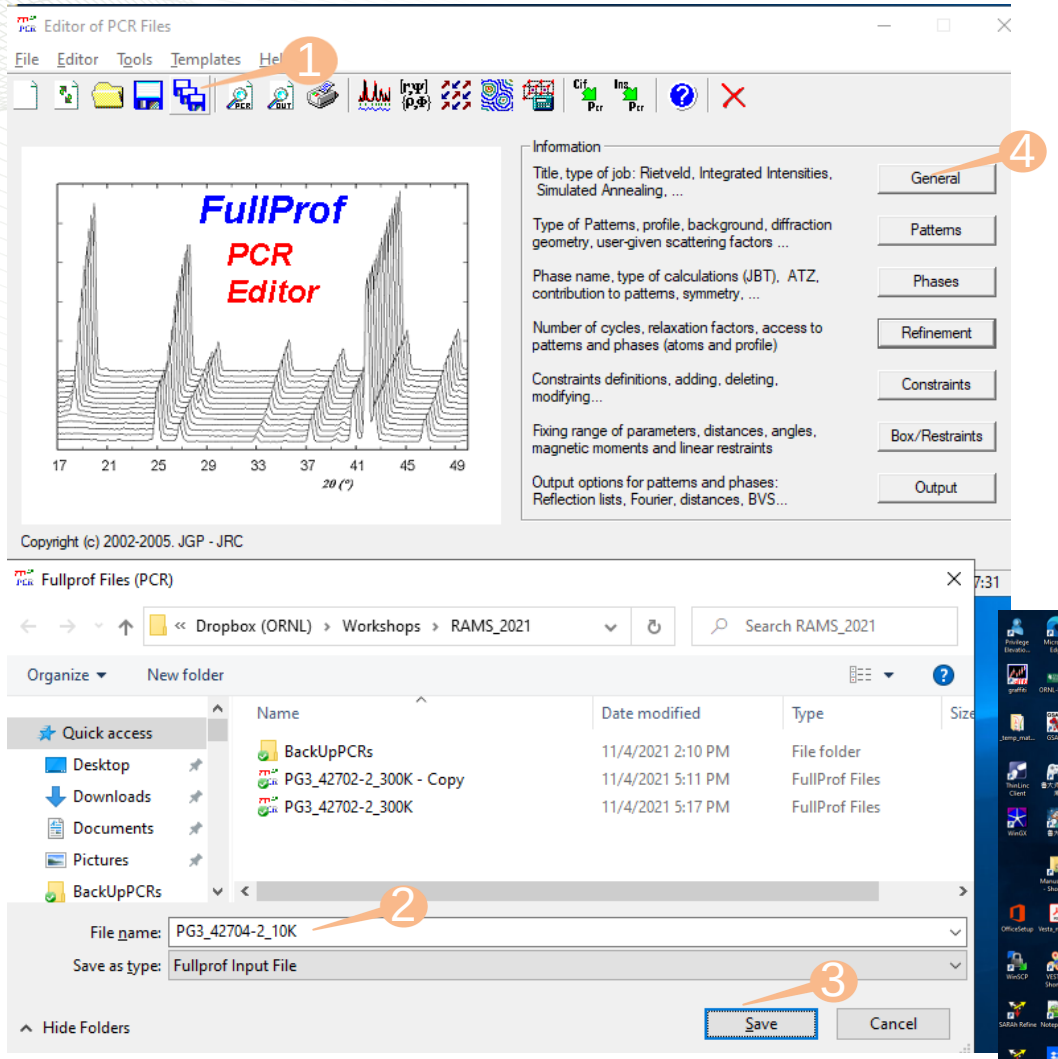


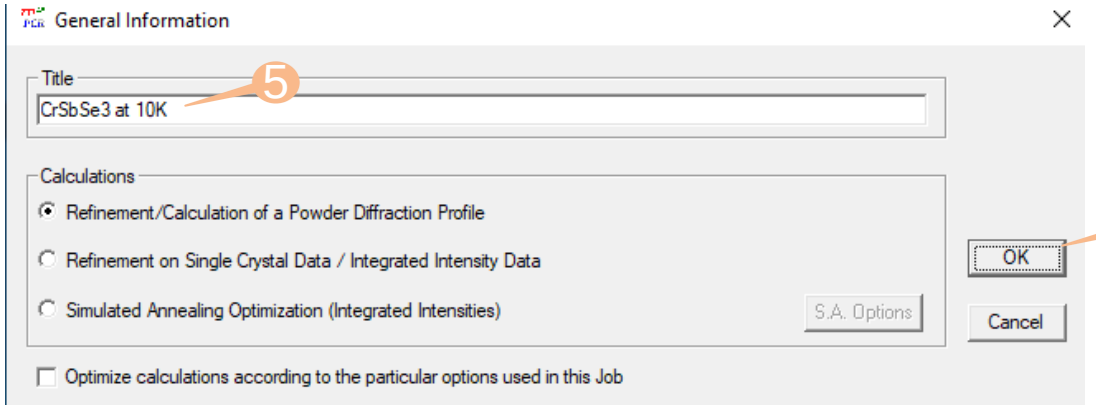



```
   =>    Bragg R-factor:    8.640
   =>    RF-factor     :    6.833
=> Conv. not yet reached -> [Max] Shift(Biso_Cr_ph1)/(eps*Sigma)=   -1.08 abs> 1
=> Normal end, final calculations and writing...


=> CPU Time:     3.750 seconds
=> 0.062 minutes

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

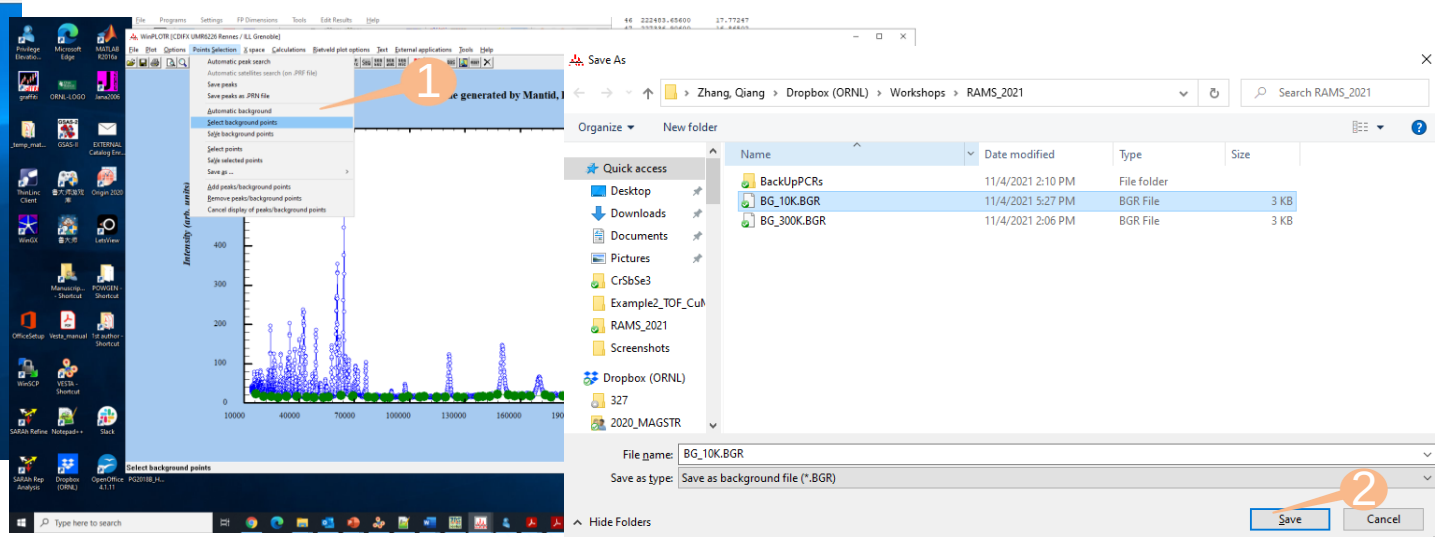


III. Save the PCR file at 300 K as a new PCR to refine the structural parameters at 10 K (T<Tm).

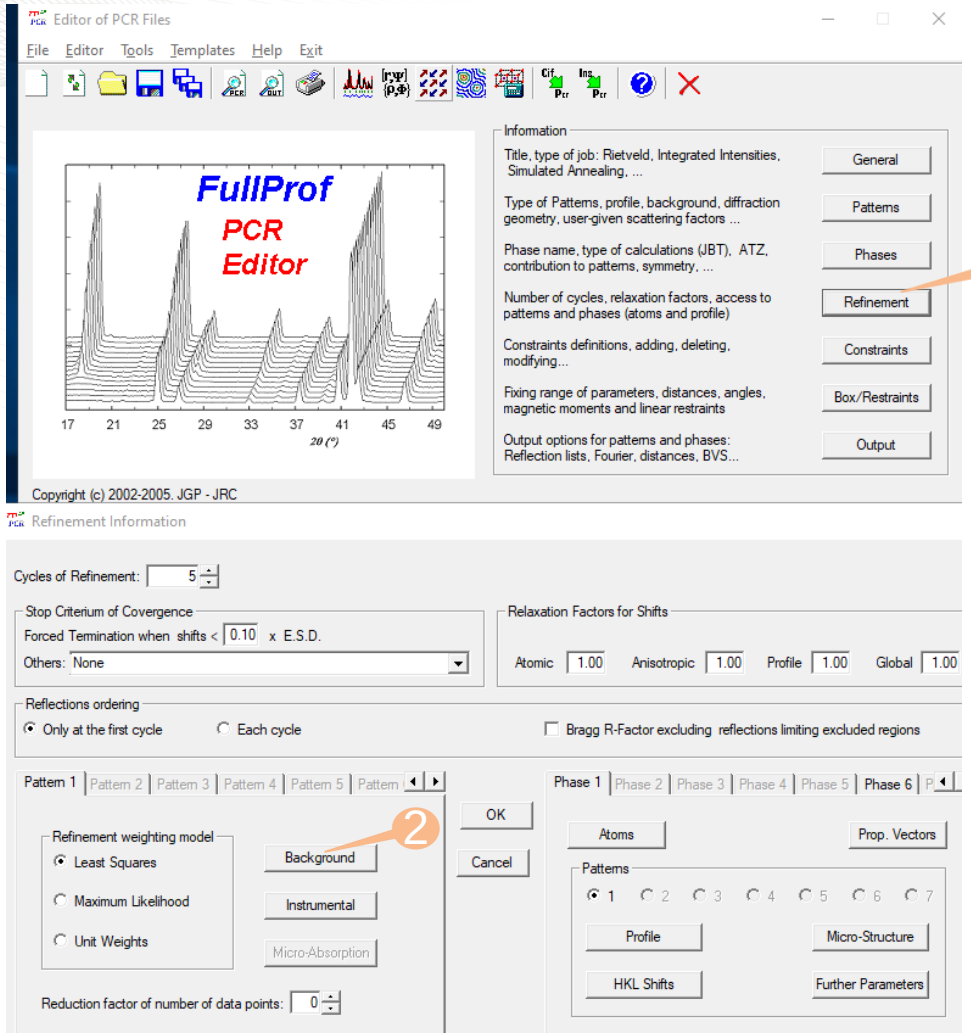


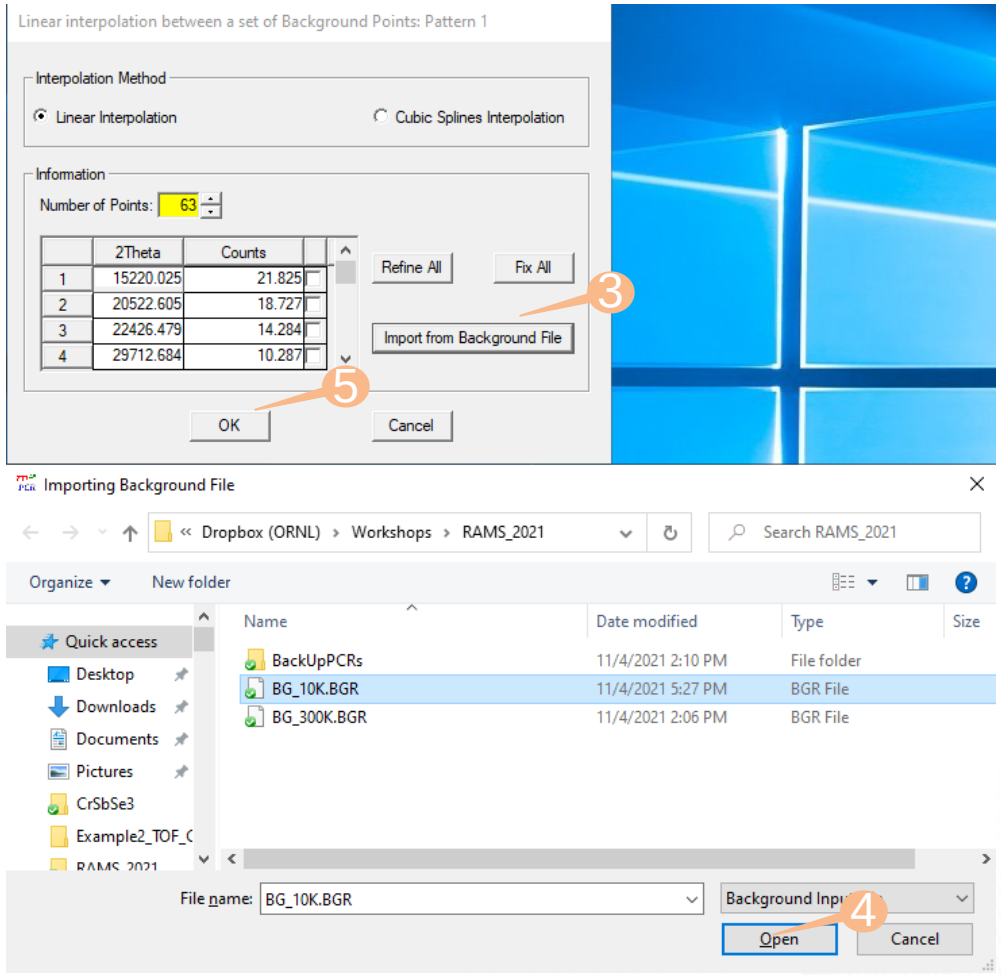


Use Winplotr to get the BG for PG3_42704-2_10K.dat (see slide 12-13)

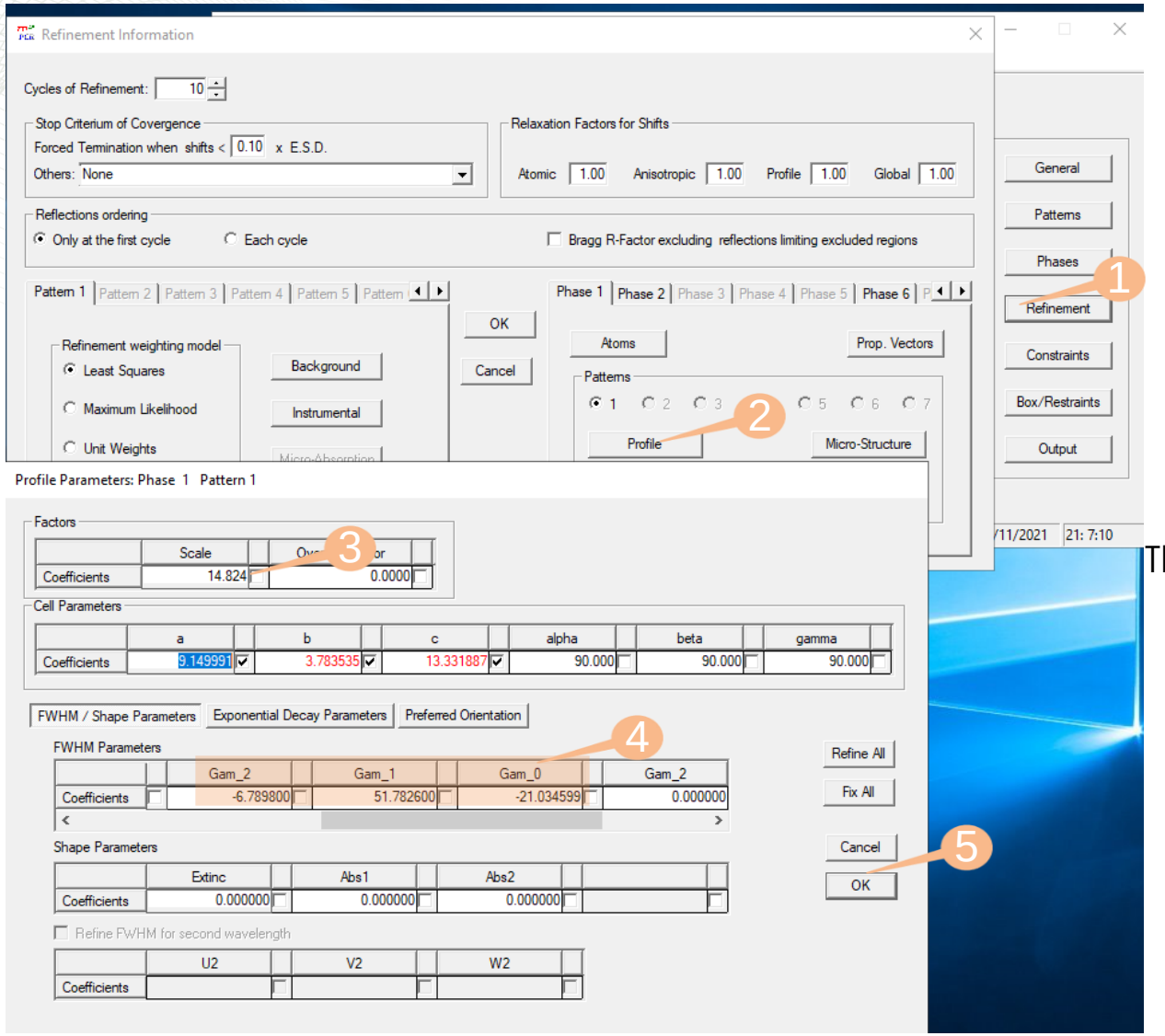


Use the BG for PG3_42704-2_10K.dat

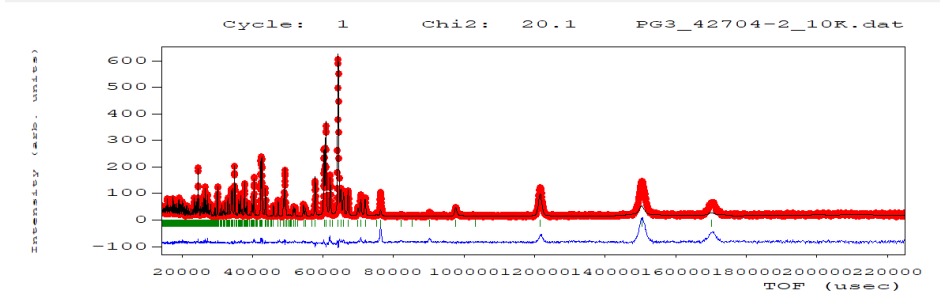




Refinement-Profile tab: unselect scale factor, Gam_2, Gam_1 and Gam_0 to refine lattice constants, the atomic positions and B factors



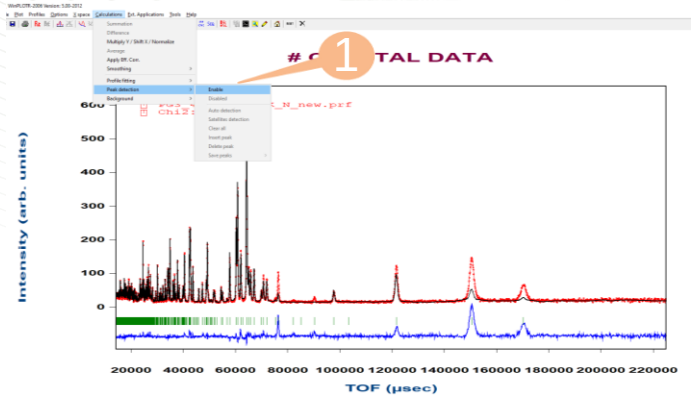

```
 => Phase:       1
 =>     Bragg R-factor:   25.46
 =>     RF-factor     :   6.479
=> Normal end, final calculations and writing...

=> CPU Time:      1.219 seconds
=> 0.020 minutes

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

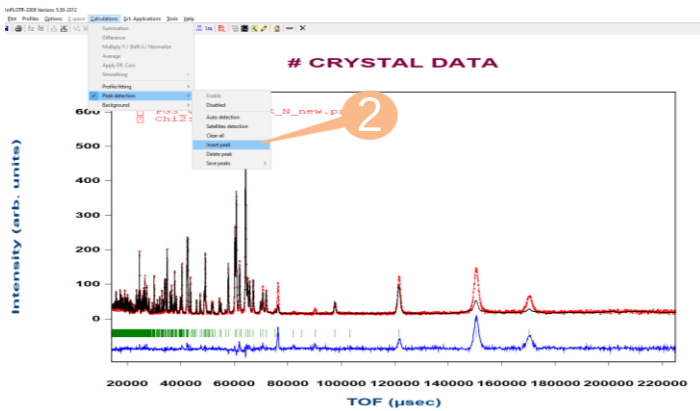


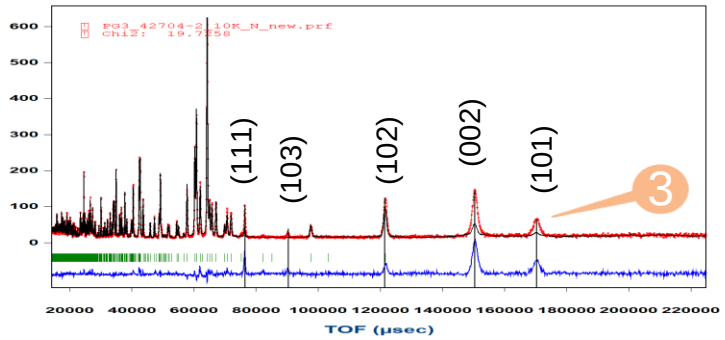
The B factors at 10 K are lower than those at 300 K as expected

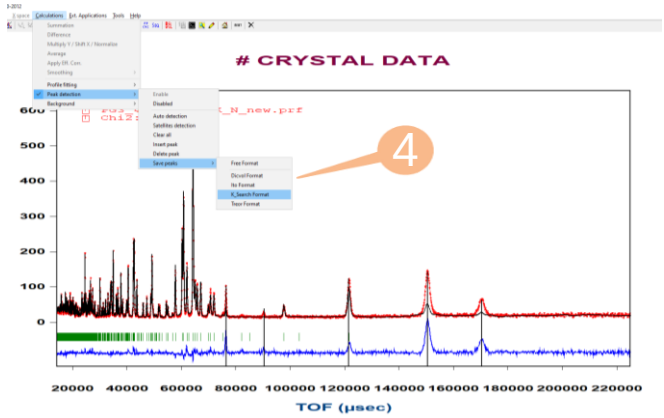
!Atom	Typ	X	Y	Z	Biso
Sb	Sb	0.02909	0.25000	0.65861	0.41769
		61.00	0.00	81.00	71.00
Cr	Cr	0.15737	0.25000	0.04474	0.34746
		91.00	0.00	111.00	101.00
Se3	Se	0.17096	0.25000	0.48400	0.41703
		121.00	0.00	141.00	131.00
Se2	Se	0.28632	0.25000	0.21412	0.38427
		151.00	0.00	41.00	51.00
Se1	Se	0.50108	0.25000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

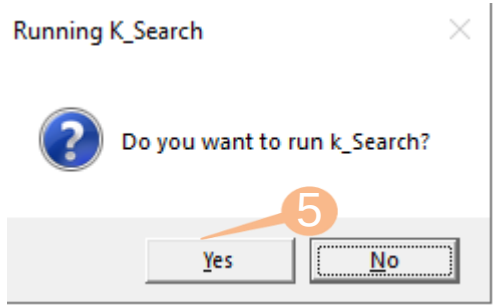
III. Identify magnetic peaks/contributions and determine the propagation vector











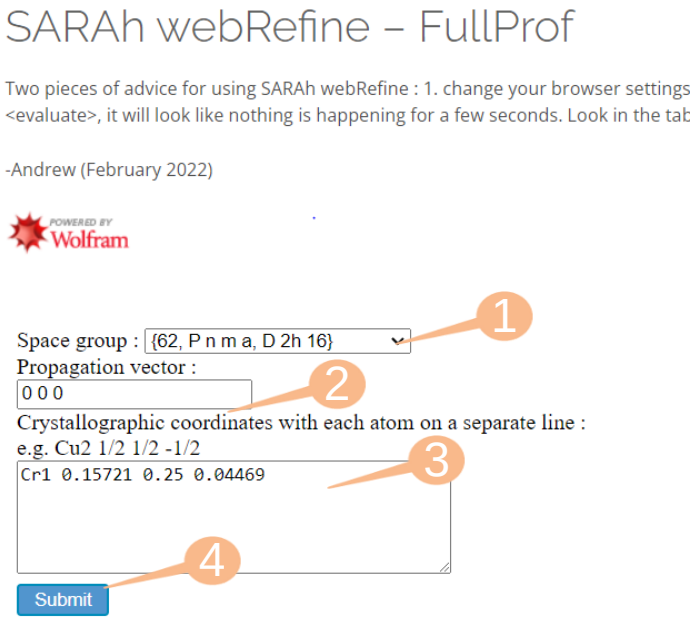

```
=> Testing 90 internal k-vectors
    Solution:       1 k =( 0.0000 0.0000 0.0000)  R-F:   0.5342
    Solution:       2 k =( 0.0000 0.3333 0.3333)  R-F:   3.5499
    Solution:       3 k =( 0.1250 0.5000 0.1250)  R-F:   4.2302
=> Special k-vector solutions found!

=> 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
```

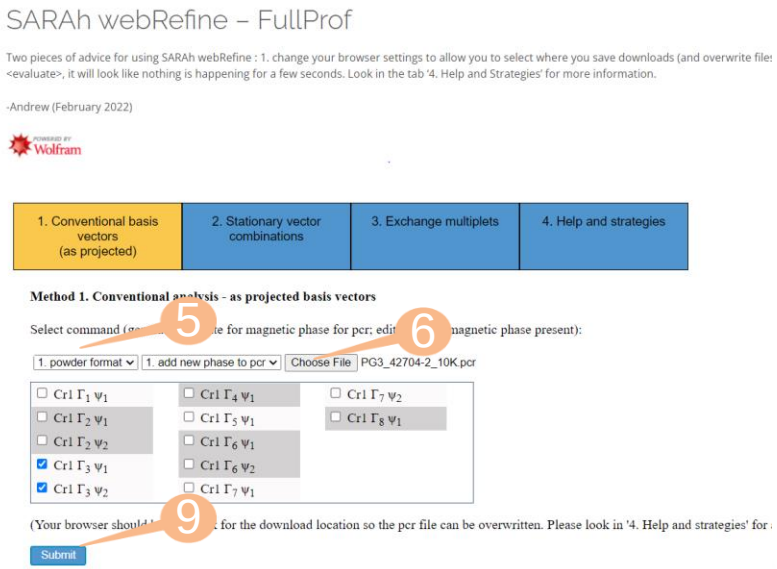
k=0!

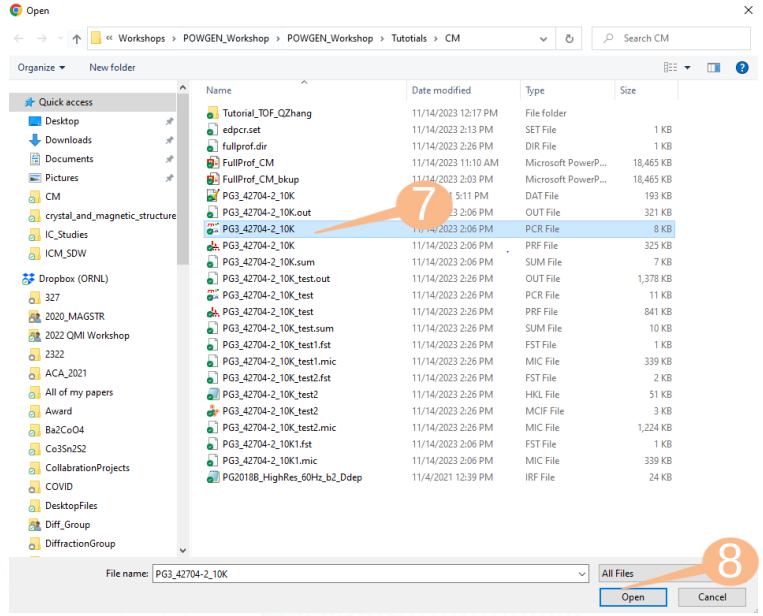
IV. Symmetry analysis to obtain irreducible representations and Basic vectors using SARAh

a). Use SARAh webRefine – FullProf:

http://fermat.chem.ucl.ac.uk/spaces/willsgroup/web-software/sarah-refine-fullprof/



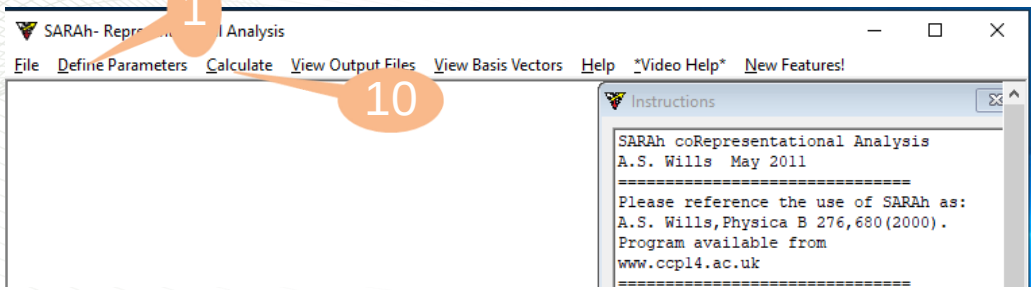


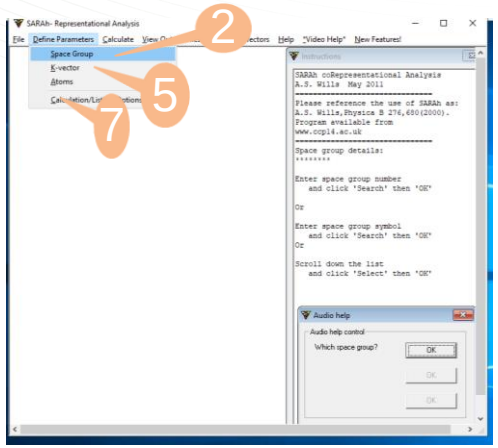


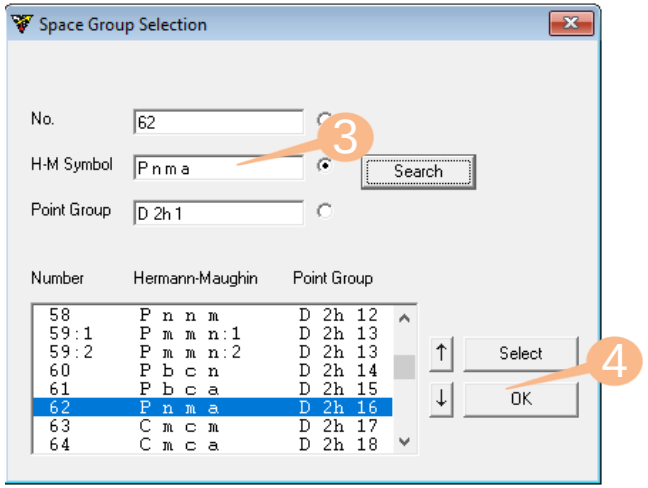
The generated PCR with the same name has been downloaded!

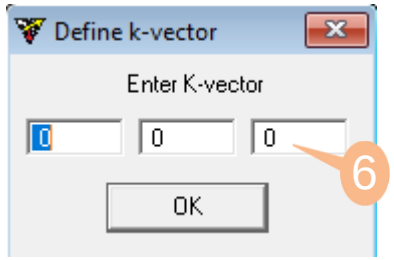
b): Use two softwares:

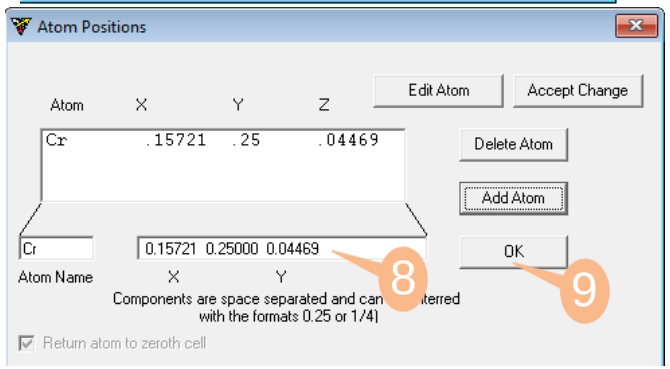
Fill in space group, K-vector and Atoms in **SARAh-Representational Analysis**












```
IR # 1, BASIS VECTOR: #   1 (ABSOLUTE NUMBER:# 1)
ATOM  1:   (     0     2     0)  +  i(     0     0     0)
ATOM  2:   (     0    -2     0)  +  i(     0     0     0)
ATOM  3:   (     0     2     0)  +  i(     0     0     0)
ATOM  4:   (     0    -2     0)  +  i(     0     0     0)
******


IR # 2, BASIS VECTOR: #   1 (ABSOLUTE NUMBER:# 2)
ATOM  1:   (     2     0     0)  +  i(     0     0     0)
ATOM  2:   (     2     0     0)  +  i(     0     0     0)
ATOM  3:   (    -2     0     0)  +  i(     0     0     0)
ATOM  4:   (    -2     0     0)  +  i(     0     0     0)

IR # 2, BASIS VECTOR: #   2 (ABSOLUTE NUMBER:# 3)
ATOM  1:   (     0     0     2)  +  i(     0     0     0)
ATOM  2:   (     0     0    -2)  +  i(     0     0     0)
ATOM  3:   (     0     0    -2)  +  i(     0     0     0)
ATOM  4:   (     0     0     2)  +  i(     0     0     0)
******


IR # 3, BASIS VECTOR: #   1 (ABSOLUTE NUMBER:# 4)
ATOM  1:   (     2     0     0)  +  i(     0     0     0)
ATOM  2:   (     2     0     0)  +  i(     0     0     0)
ATOM  3:   (     2     0     0)  +  i(     0     0     0)
ATOM  4:   (     2     0     0)  +  i(     0     0     0)

IR # 3, BASIS VECTOR: #   2 (ABSOLUTE NUMBER:# 5)
ATOM  1:   (     0     0     2)  +  i(     0     0     0)
ATOM  2:   (     0     0    -2)  +  i(     0     0     0)
ATOM  3:   (     0     0     2)  +  i(     0     0     0)
ATOM  4:   (     0     0    -2)  +  i(     0     0     0)
******


IR # 4, BASIS VECTOR: #   1 (ABSOLUTE NUMBER:# 6)
ATOM  1:   (     0     2     0)  +  i(     0     0     0)
ATOM  2:   (     0    -2     0)  +  i(     0     0     0)
ATOM  3:   (     0    -2     0)  +  i(     0     0     0)
ATOM  4:   (     0     2     0)  +  i(     0     0     0)
******
```

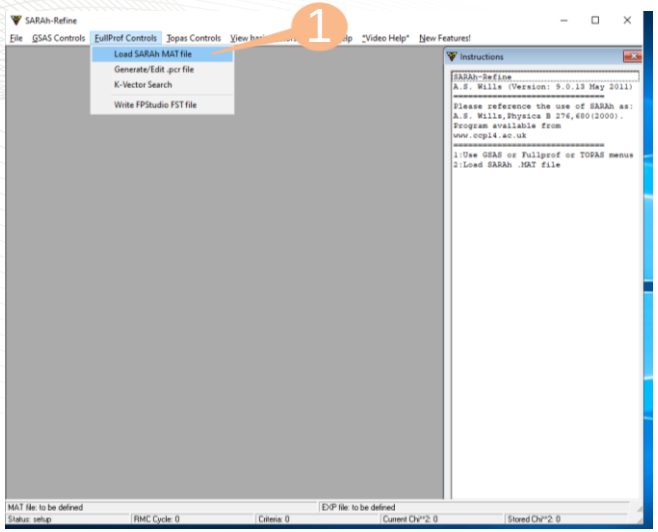
Magnetic space group

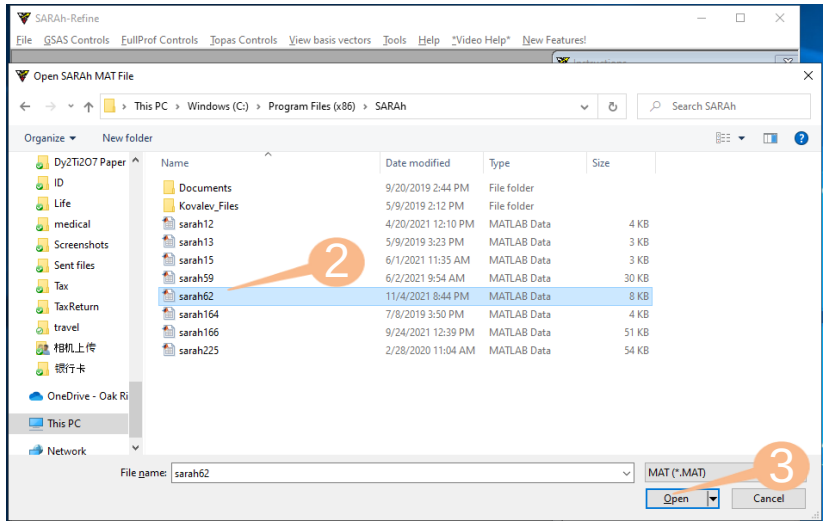
```
MAGNETIC SPACE GROUPS (THE BLACK AND WHITE SHUBNIKOV GROUPS)

 BV#        Shubnikov Group Group number
  1   IR# 1      Pnma          62.441
  2   IR# 2   Pn'm'a'          62.449
  3   IR# 2   Pn'm'a'          62.449
  4   IR# 3    Pnm'a'          62.447
  5   IR# 3    Pnm'a'          62.447
  6   IR# 4     Pn'ma          62.443
  7   IR# 5    Pn'ma'          62.448
  8   IR# 6     Pnm'a          62.444
  9   IR# 6     Pnm'a          62.444
 10   IR# 7    Pn'm'a          62.446
 11   IR# 7    Pn'm'a          62.446
 12   IR# 8     Pnma'          62.445
```

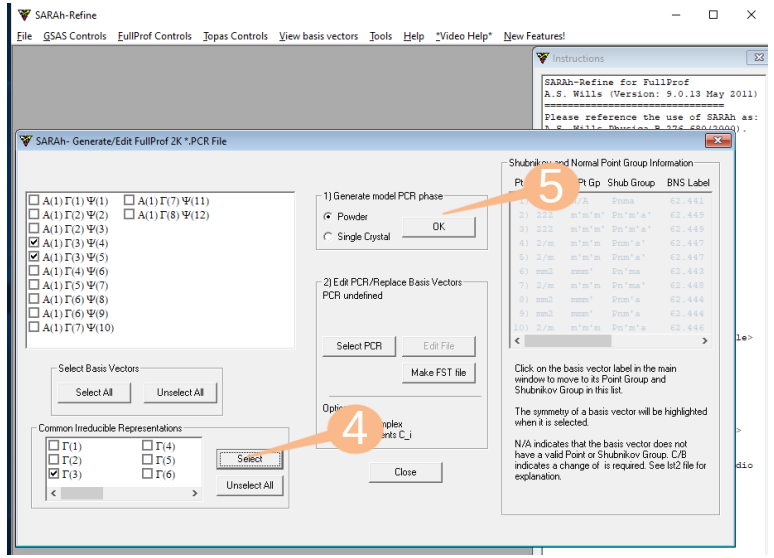
8 Irs in total

Select a magnetic model and add it as the 2nd phase in the PCR

Generate PCR of Γ_3 using **SARAh refine**. Γ_3 : moment in the *ac* plane



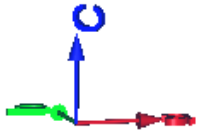


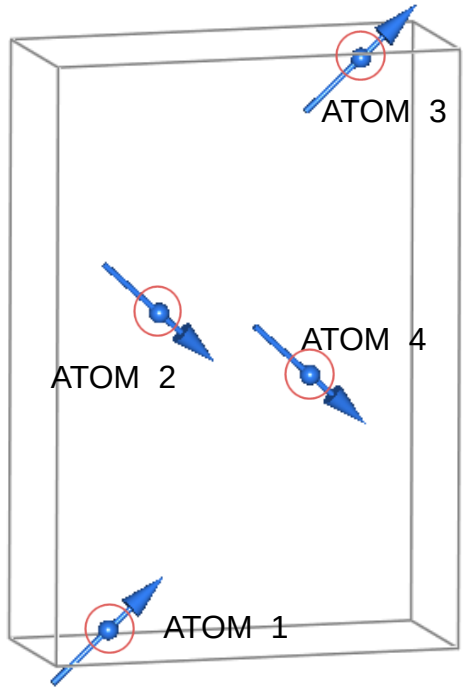


sarah62.pcr

```
!-------------------------------------------------------------------------------
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:    1.00
!-------------------------------------------------------------------------------
Magnetic Phase
!
!Nat Dis Mom Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth      ATZ    Nyk Npr More
1   0   0 0.0 0.0 1.0   1   0  -2   0   0         0.00   0   0   0
!
P -1                        <--Space group symbol
! Nsym   Cen  Laue Ireps N_Bas
    4     1     1    -1     2
! Real(0)-Imaginary(1) indicator for Ci
  0  0
!                    Basic vectors
SYMM X, Y, Z
BASR       2        0        0        0        0        2
BASI       0        0        0        0        0        0
SYMM -X+1/2, -Y+1, Z+1/2
BASR       2        0        0        0        0       -2
BASI       0        0        0        0        0        0
SYMM -X+1, Y+1/2, -Z+1
BASR       2        0        0        0        0        2
BASI       0        0        0        0        0        0
SYMM X+1/2, -Y+1/2, -Z+1/2
BASR       2        0        0        0        0       -2
BASI       0        0        0        0        0        0
!Atom Typ  Mag Vek     X        Y        Z       Biso    Occ      C1       C2       C3
!     C4       C5       C6       C7       C8       C9      MagPh
CR1  MCR3  1    0    .15721   .25000   .04469  .30000 1.00000    0.000    0.000    0.000
                     0.00     0.00     0.00     0.00     0.00     0.00     0.00     0.00
    0.000    0.000    0.000    0.000    0.000    0.000    .00000
    0.00     0.00     0.00     0.00     0.00     0.00     0.00
!-------> Profile Parameters for Pattern #  1
!  Scale       Shape1       Bov      Str1      Str2      Str3     Strain-Model
    10.0        0.0000    0.0000    0.0000    0.0000    0.0000        0
     0.00000      0.00      0.00      0.00      0.00      0.00
!     U         V         W         X         Y       GauSiz    LorSiz Size-Model
   1.08239   -0.23233    0.25618   0.00000   0.00000   0.00000   0.00000    0
     0.00      0.00      0.00      0.00      0.00      0.00      0.00
!      a         b         c        alpha      beta       gamma
273
     0.00000    0.00000    0.00000    0.00000    0.00000    0.00000
!  Pref1     Pref2      Asy1      Asy2      Asy3      Asy4
   0.00000   0.00000   0.00000   0.00000   0.00000   0.00000
     0.00      0.00      0.00      0.00      0.00      0.00
```

ATOM 1: (.15721, .25, .04469)
ATOM 2: (.34279, .75, .54469)
ATOM 3: (.84279, .75, .95531)
ATOM 4: (.65721, .25, .45531)





Between atom1 and 2 (or atom 3 and 4):

Component along *a is* FM (related to C1);
Component along *c is* AFM (related to C2);

Q: Is the magnetic structure of $CrSbSe_3$ FM along *a* axis or AFM along *c* aixs or canted AFM order?

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Fix all the structural parameters and initialize nonzero C1 and C2 coefficients to the two basic vectors

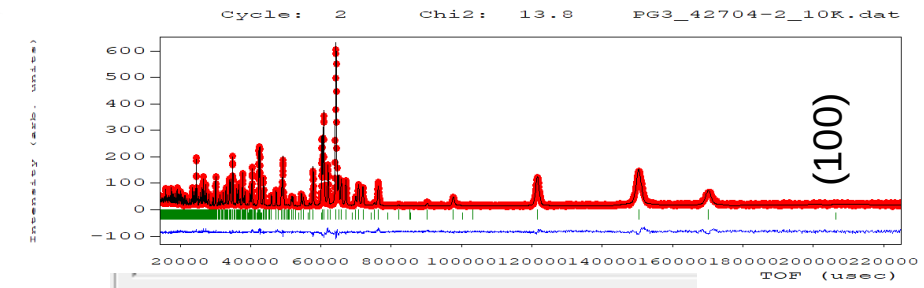
Basis Functions Coefficients

	C1	C2	C3	C4	C5	C6	C7
Atom # 1	1.00000	1.00000	0.00000	0.00000	0.00000	0.00000	0.00000

```
   => Phase:       1
   =>    Bragg R-factor:    8.241
   =>    RF-factor     :    6.263
   => Phase:       2
   => Magnetic R-factor:    18.79
=> Normal end, final calculations and writing...


=> CPU Time:     4.547 seconds
=> 0.076 minutes

=> END    Date:05/11/2021  Time => 00:55:25.340
```



Basis Functions Coefficients

	C1	C2
Atom # 1	1.36800	0.16000

Fix all the parameters and only initialize nonzero C1 to the first basic vector only

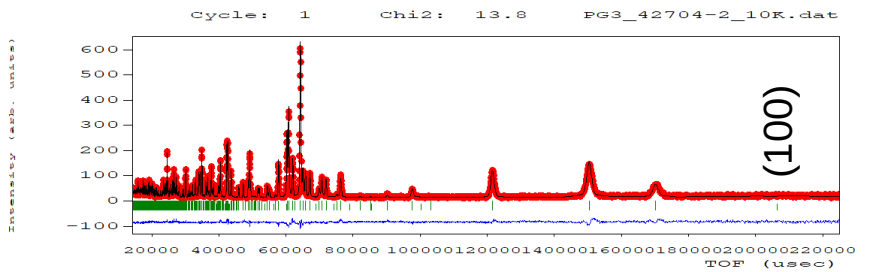
Basis Functions Coefficients

	C1	C2	C3	C4	C5	C6	C7
Atom # 1	1.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000

```
   => Phase:       1
   =>    Bragg R-factor:    8.480
   =>    RF-factor     :    6.259
   => Phase:       2
   => Magnetic R-factor:    16.89
=> Normal end, final calculations and writing...


=> CPU Time:     3.031 seconds
=> 0.051 minutes

=> END    Date:05/11/2021  Time => 00:56:38.862
```



Basis Functions Coefficients

	C1	C2
Atom # 1	1.38400	0.00000

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.

The fitting can be further improved by refining the scale factor, lattice parameters, profile parameters. Remember to constrain all of them to be the same for both nuclear and magnetic phases.

Modify PCR directly

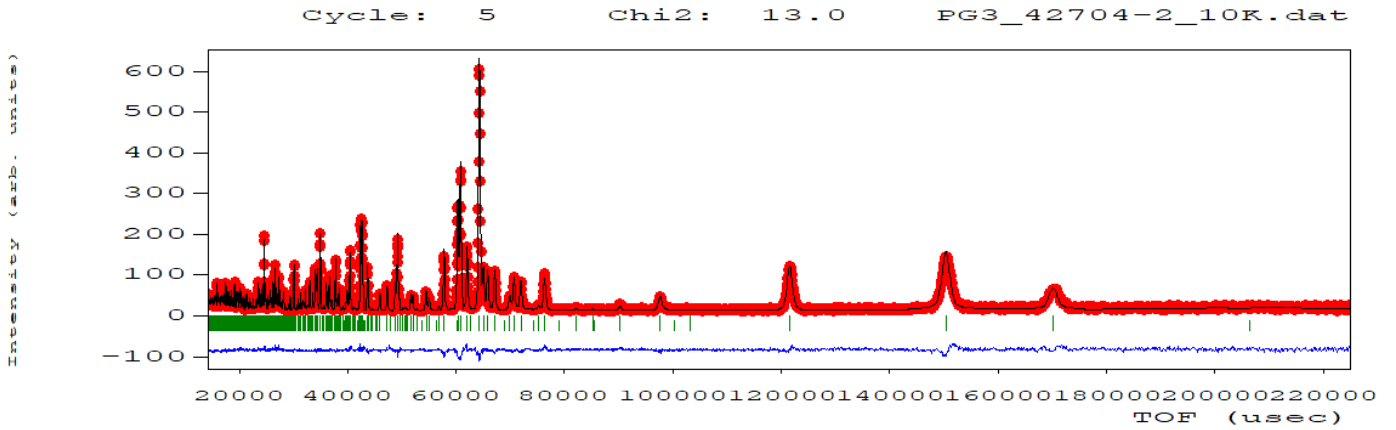
```
!-------> Profile Parameters for Pattern #   1  ----> Phase #   1
!  Scale          Extinc       Bov      Str1      Str2      Str3     Strain-Model
 15.61343       0.0000    0.0000    0.0000    0.0000    0.0000        0
    11     0.00      0.00      0.00      0.00      0.00
!       Sig-2       Sig-1       Sig-0       Sig-Q     G-Strain     G-Size        Z0  Size-Model
!  Instrumental Sigma interpolated from list in IRF file (Only sample contribution is refined)
     0.0000     -0.0076      0.0063      0.0190      0.0000      0.0000      0.0000   0
       0.00        0.00        0.00        0.00        0.00        0.00
!       Gam-2       Gam-1       Gam-0       LStr        LSiz
!  Instrumental Gamma interpolated from list in IRF file (Only sample contribution is refined)
    -7.0876     58.1942    -20.5226      0.0000      0.0000
       21          31          41          0.00        0.00
!     a          b          c          alpha       beta        gamma       #Cell Info
 9.150326   3.783540  13.331617  90.000000  90.000000  90.000000
  51  61  71    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   1.00000    0.00              ABS: ABSCOR1  ABSCOR2
!-------------------------------------------------------------------------------
!  Data for PHASE number:   2  ==> Current R_Bragg for Pattern#  1:   0.0000
!-------------------------------------------------------------------------------
MagneticPhaseIR3
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ    Nvk Npr More
  1   0   0 0.0 0.0 1.0   1   0  -2   0   0        207.984   0   9   0
!
!
P -1                    <--Space group symbol for hkl generation
! Nsym   Cen  Laue Ireps N_Bas
    4     1     1    -1     2
! Real(0)-Imaginary(1) indicator for Ci
  0  0
!
SYMM X, Y, Z
BASR        2        0        0        0        0        2
BASI        0        0        0        0        0        0
SYMM -X+1/2, -Y+1, Z+1/2
BASR        2        0        0        0        0       -2
BASI        0        0        0        0        0        0
SYMM -X+1, Y+1/2, -Z+1
BASR        2        0        0        0        0        2
BASI        0        0        0        0        0        0
SYMM X+1/2, -Y+1/2, -Z+1/2
BASR        2        0        0        0        0       -2
BASI        0        0        0        0        0        0
!
!Atom   Typ  Mag Vek    X       Y       Z       Biso    Occ      C1      C2      C3
!     C4      C5      C6      C7      C8      C9      MagPh
CR1    MCR3  1   0  0.15737 0.25000 0.04474 0.34746  1.00000   1.276   0.000   0.000
                     0.00    0.00    0.00    0.00     0.00    81    0.00    0.00
      0.000   0.000   0.000   0.000   0.000   0.000  0.00000
      0.00    0.00    0.00    0.00    0.00    0.00     0.00
!-------> Profile Parameters for Pattern #   1  ----> Phase #   2
!  Scale          Extinc       Bov      Str1      Str2      Str3     Strain-Model
 15.61343       0.0000    0.0000    0.0000    0.0000    0.0000        0
    11     0.00      0.00      0.00      0.00      0.00
!       Sig-2       Sig-1       Sig-0       Sig-Q     G-Strain     G-Size        Z0  Size-Model
!  Instrumental Sigma interpolated from list in IRF file (Only sample contribution is refined)
     0.0000     -0.0076      0.0063      0.0190      0.0000      0.0000      0.0000   0
       0.00        0.00        0.00        0.00        0.00        0.00
!       Gam-2       Gam-1       Gam-0       LStr        LSiz
!  Instrumental Gamma interpolated from list in IRF file (Only sample contribution is refined)
    -7.0876     58.1942    -20.5226      0.0000      0.0000
       21          31          41          0.00        0.00
!     a          b          c          alpha       beta        gamma       #Cell Info
 9.150326   3.783540  13.331617  90.000000  90.000000  90.000000
  51  61  71    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
```

Final refinement result:

```
   =>    Bragg R-factor:    7.513
   =>    RF-factor     :    5.997
   => Phase:       2
   => Magnetic R-factor:    9.653
=> Normal end, final calculations and writing...


=> CPU Time:      1.562 seconds
=> 0.026 minutes

=> END    Date:05/11/2021  Time => 00:01:57.213
```



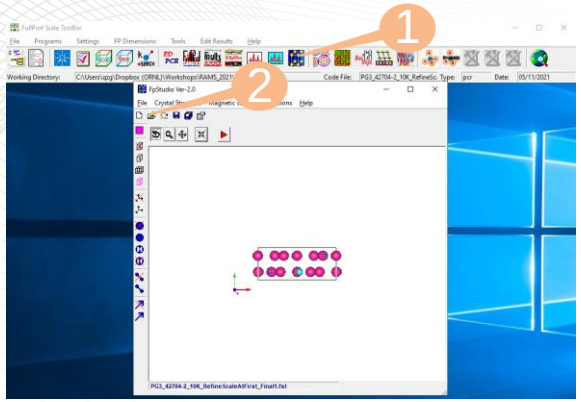
Moment size:

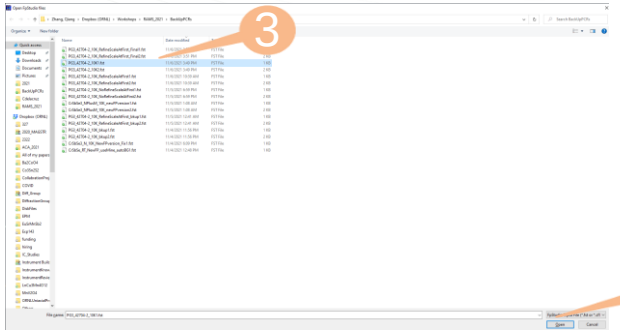
$$\overrightarrow{m_j} = \sum_{v,\vec{k}} S_v^{\vec{k}}\, e^{-2\pi i \vec{k}\cdot\overrightarrow{R_{ij}}} = \sum_{v,\vec{k}} c_v^{\vec{k}} \vec{\psi}_{i,v}^{\vec{k}}\, e^{-2\pi i \vec{k}\cdot\overrightarrow{R_{ij}}} = C1 * \begin{bmatrix}2\\0\\0\end{bmatrix} * \exp\left[-2\pi i \begin{bmatrix}0\\0\\0\end{bmatrix}\right] * \begin{bmatrix}0\\0\\0\end{bmatrix}$$

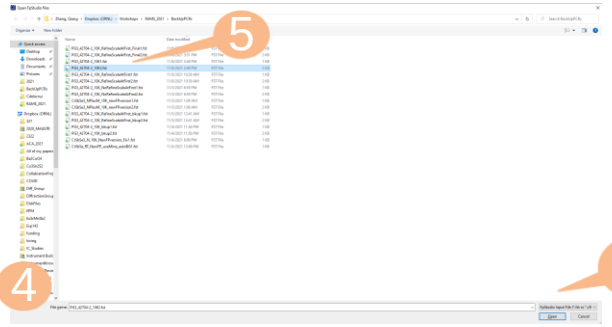
$= 1.39*2 = 2.78(5)\ \mu_B$

VIII. Display the magnetic structure using FpStudio or Vesta.

FpStudio





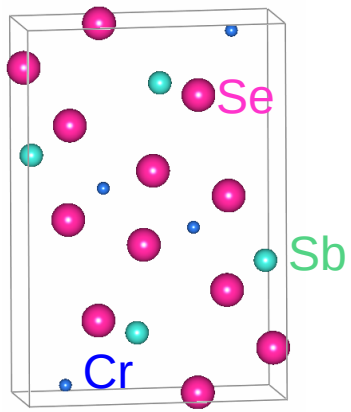


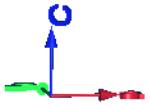
Combine two fst files

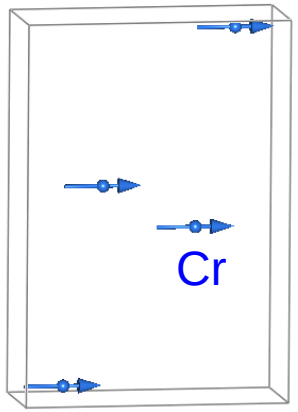
Combined fst file

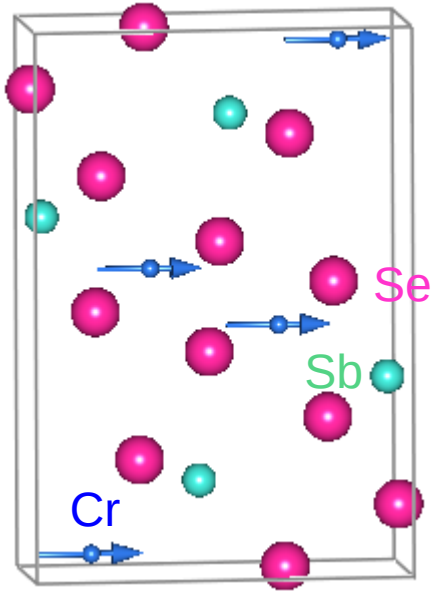
```
!   FILE for FullProf Studio: generated automatically by FullProf
!Title:   CrSbSe3 nuclear phase
SPACEG P n m a
CELL      9.150326    3.783540   13.331617  90.0000  90.0000  90.0000   DISPLAY MULTIPLE
BOX    -0.15  1.15   -0.15  1.15    -0.15  1.15
ATOM Sb       Sb        0.02909   0.25000   0.65861
ATOM Cr       Cr        0.15737   0.25000   0.04474
ATOM Se3      Se        0.17096   0.25000   0.48400
ATOM Se2      Se        0.28632   0.25000   0.21412
ATOM Se1      Se        0.50108   0.25000   0.60829

{
LATTICE P
K      0.00000   0.00000   0.00000
SYMM  x,y,z
MSYM  u,v,w,0.0
MATOM CR1_1             Cr      0.15737  0.25000  0.04474  SCALE 1.0 GROUP
SKP             1  1   2.56154   0.00000   0.00000     0.00000   0.00000   0.00000    0.00000
MATOM CR1_2             Cr      0.34263  0.75000  0.54474  SCALE 1.0 GROUP
SKP             1  1   2.56154   0.00000   0.00000     0.00000   0.00000   0.00000    0.00000
MATOM CR1_3             Cr      0.84263  0.75000  0.95526  SCALE 1.0 GROUP
SKP             1  1   2.56154   0.00000   0.00000     0.00000   0.00000   0.00000    0.00000
MATOM CR1_4             Cr      0.65737  0.25000  0.45526  SCALE 1.0 GROUP
SKP             1  1   2.56154   0.00000   0.00000     0.00000   0.00000   0.00000    0.00000
}
```

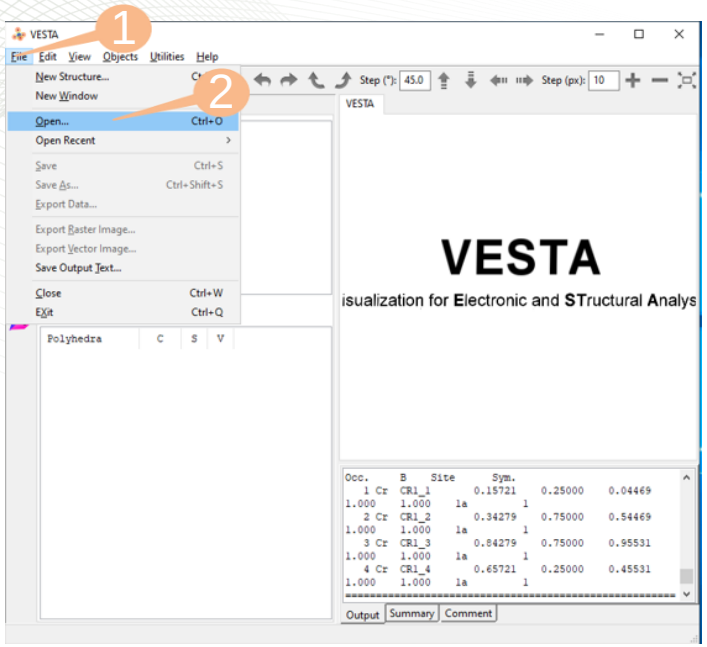


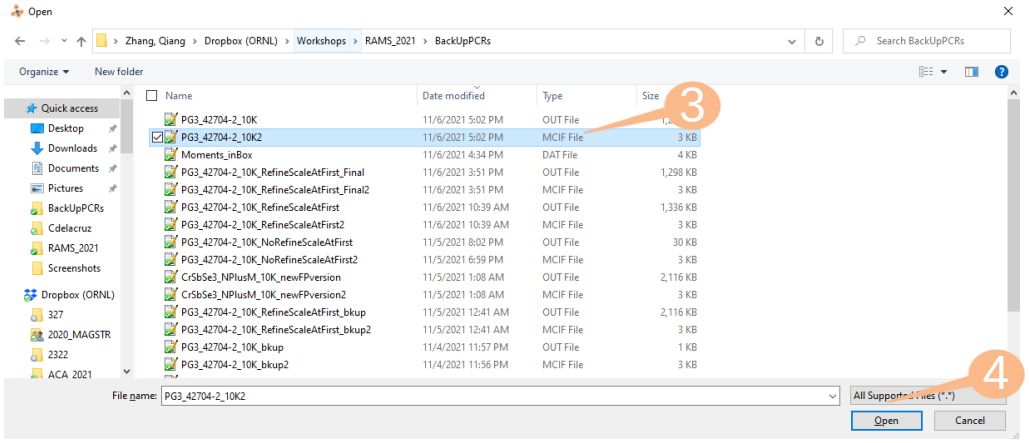


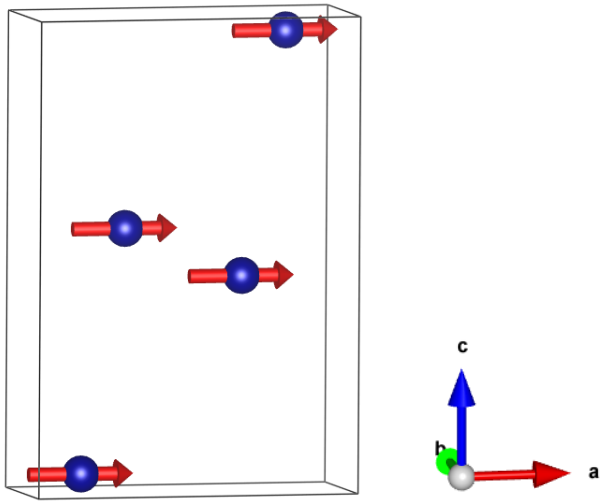




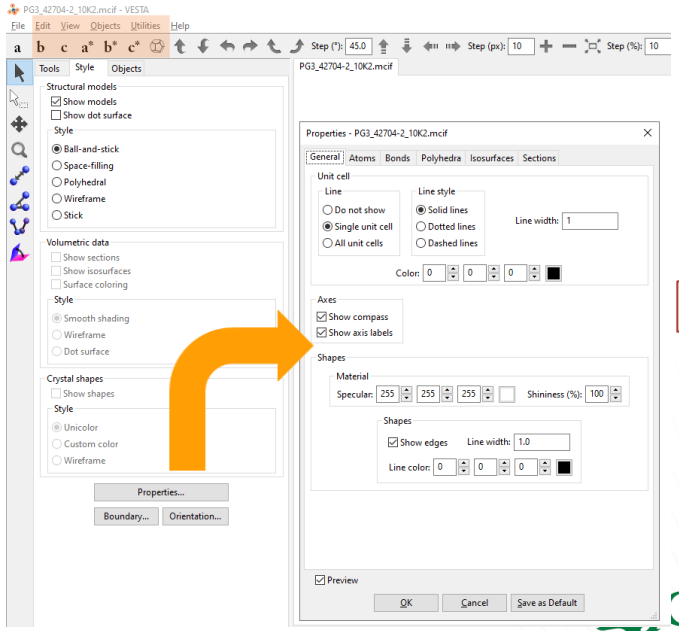
Vesta to open *.mcif file







One may modify all these tabs to improve the figure



Please remember to report the magnetic space group:

```
MAGNETIC SPACE GROUPS (THE BLACK AND WHITE SHUBNIKOV GROUPS)

 BV#        Shubnikov Group Group number
  1    IR# 1      Pnma          62.441
  2    IR# 2   Pn'm'a'          62.449
  3    IR# 2   Pn'm'a'          62.449
  4    IR# 3    Pnm'a'          62.447
  5    IR# 3    Pnm'a'          62.447
  6    IR# 4     Pn'ma          62.443
  7    IR# 5    Pn'ma'          62.448
  8    IR# 6     Pnm'a          62.444
  9    IR# 6     Pnm'a          62.444
 10    IR# 7    Pn'm'a          62.446
 11    IR# 7    Pn'm'a          62.446
 12    IR# 8     Pnma'          62.445
```

FullProf tutorial on crystal structure and commensurate magnetic structure

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

zhangq6@ornl.gov