

Magnetic Structure Workshop

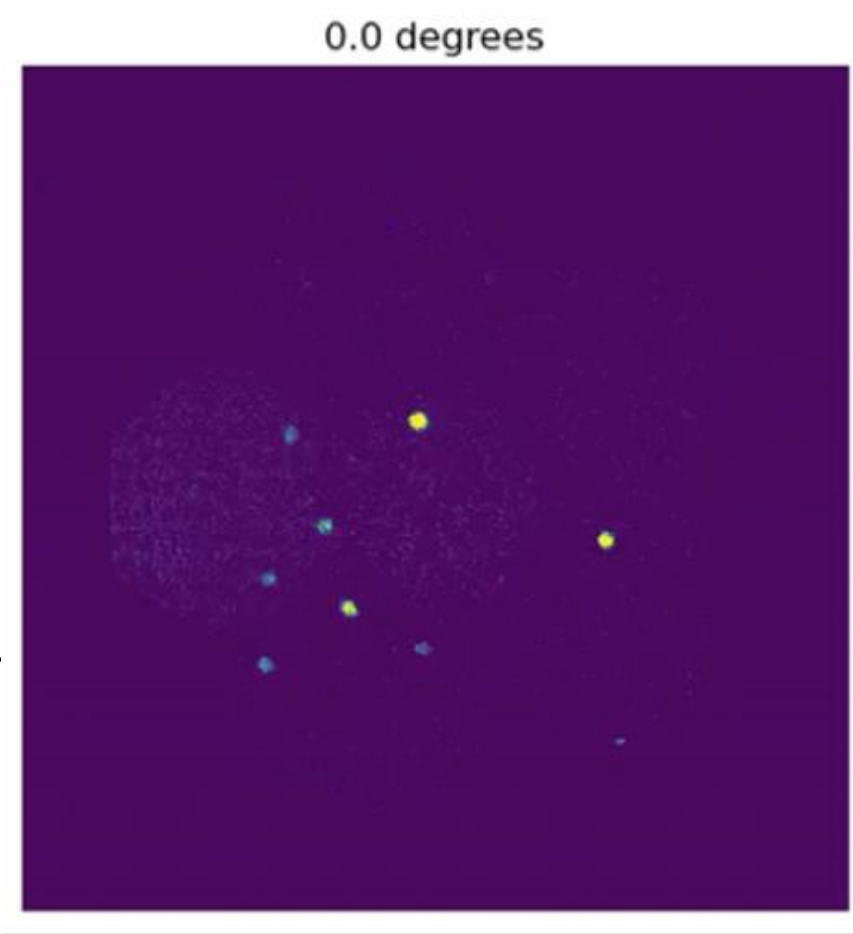
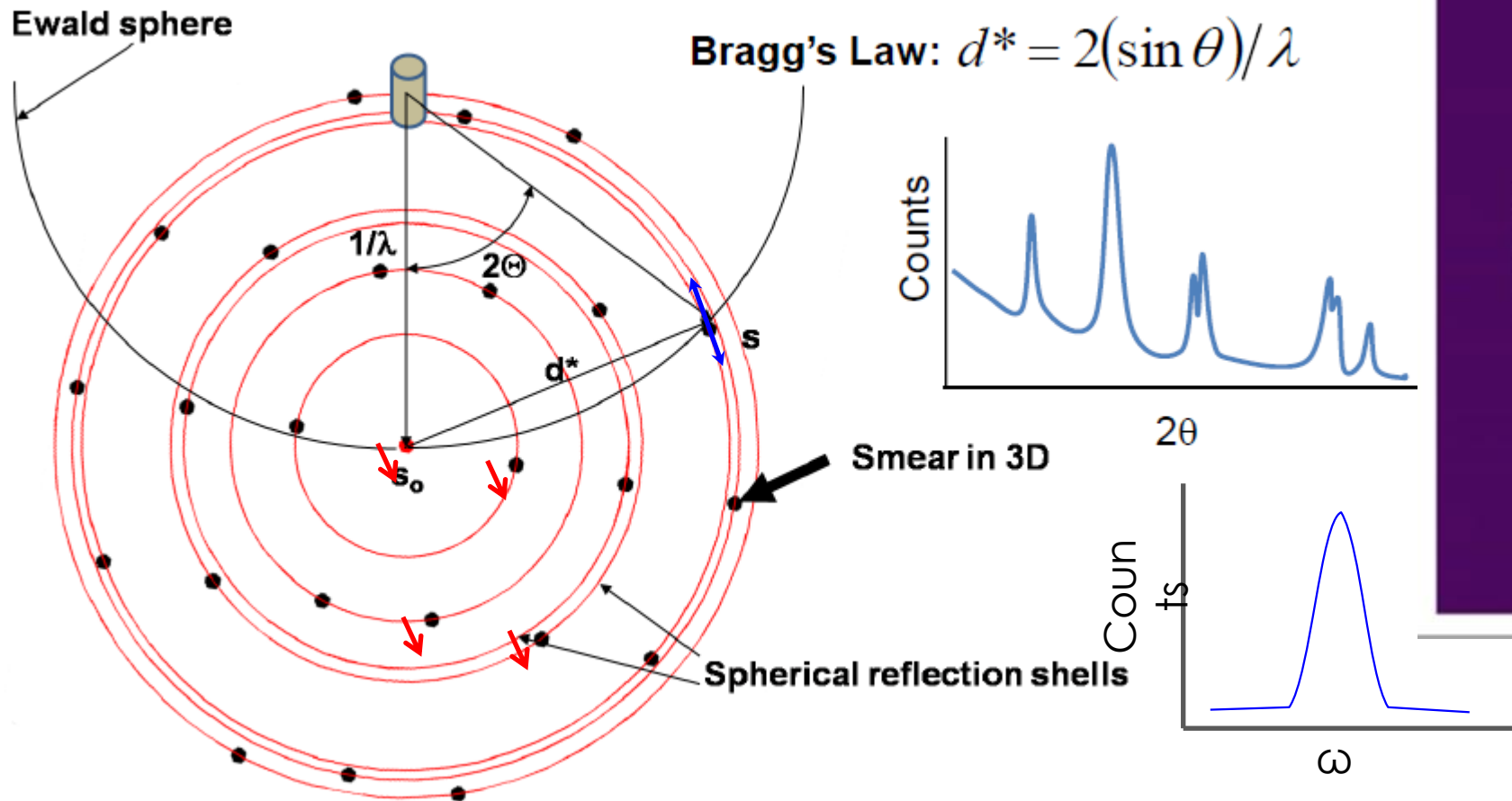
Single crystal neutron diffraction

Huibo Cao

Neutron Scattering Division, ORNL
Oak Ridge National Laboratory

ORNL is managed by UT-Battelle, LLC for the US Department of Energy

Single Crystal Diffraction



Propagation vector k ?

Single Crystal Diffraction

Advantage:

- ❖ More detailed information by measuring well separated Bragg peaks in 3D reciprocal space — **Magnetic form factors**
- ❖ **Less samples – mg VS g**
- ❖ Larger molecules and unit cells ---- **complex magnetic structures**
- ❖ **Less neutron absorption** due to small size, no isotope needed, such as for H, Li, even Eu, Sm, et. al. neutron “block” elements. Intensities corrected by applying the proper absorption correction, like the single crystal X-ray
- ❖ **Unambiguously determine the k-vector** –superlattice or satellite peaks (commensurate and incommensurate), dimensionality of scattering (rods, planes, etc.)

Disadvantage:

- ❖ Need to grow a single crystal
- ❖ Data collection can be more time consuming
- ❖ Extinction, domain, twinning

Always **GOOD to combine** both X-ray/neutron powder and single crystal diffraction!

DEMAND instrument (multi-modes)

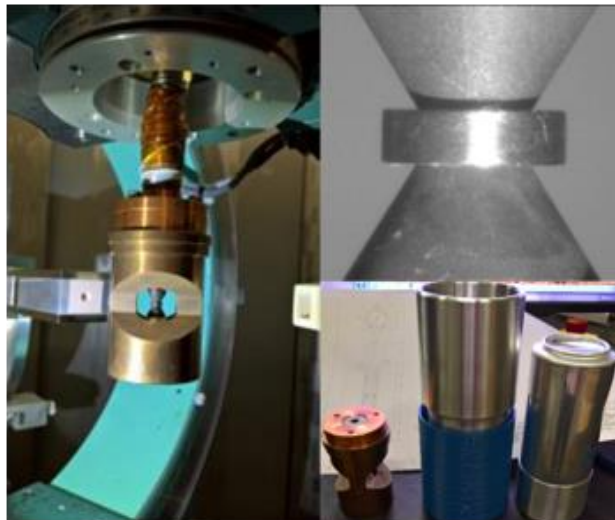
1) four-circle mode



2) two-axis mode



Extreme sample environment

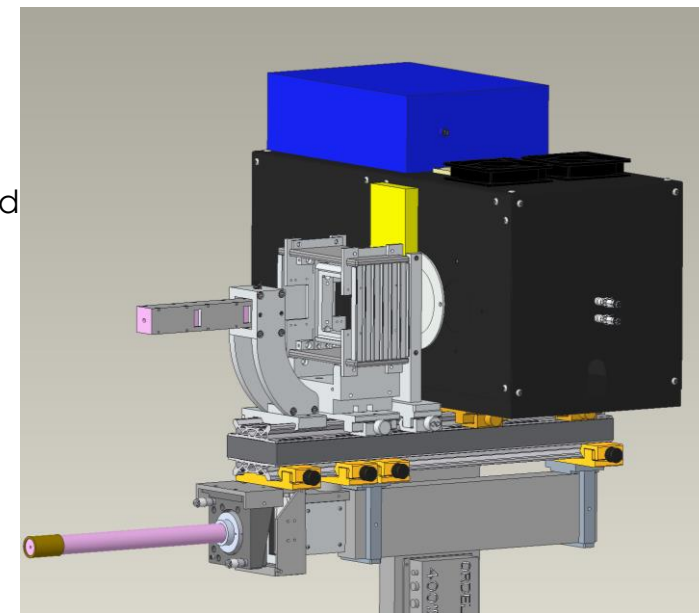


Ultra-low temperature
 $T \sim 0.05$ to 700 K

High magnetic field
 $H \sim -6$ to 6 T
(vertical)

High pressure
 $P \sim 0$ to 10 GPa

Electric field
 $E \sim 0$ - 10^4
voltage/cm



Large area detector 50° (hori) x 50° (vert), 3(1) columns position-sensitive but magnetic field insensitive

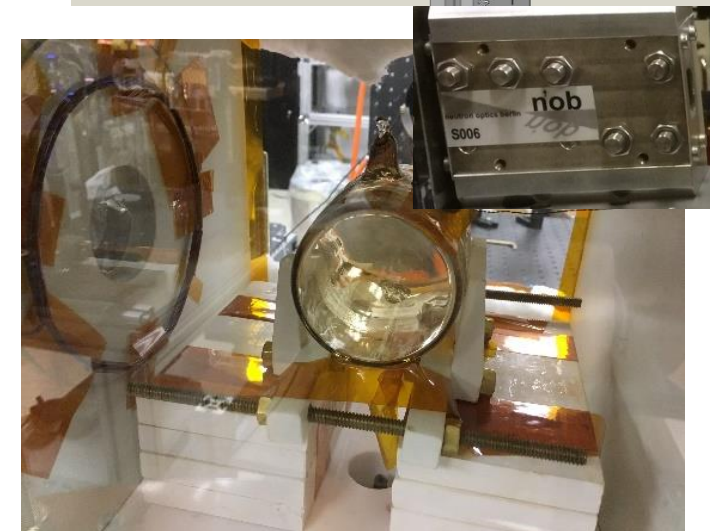
Beam:

a) High Flux: 2.2×10^7 n/cm²s

b) High Resolution: $\delta d/d = \delta Q/Q \sim 0.2\%$

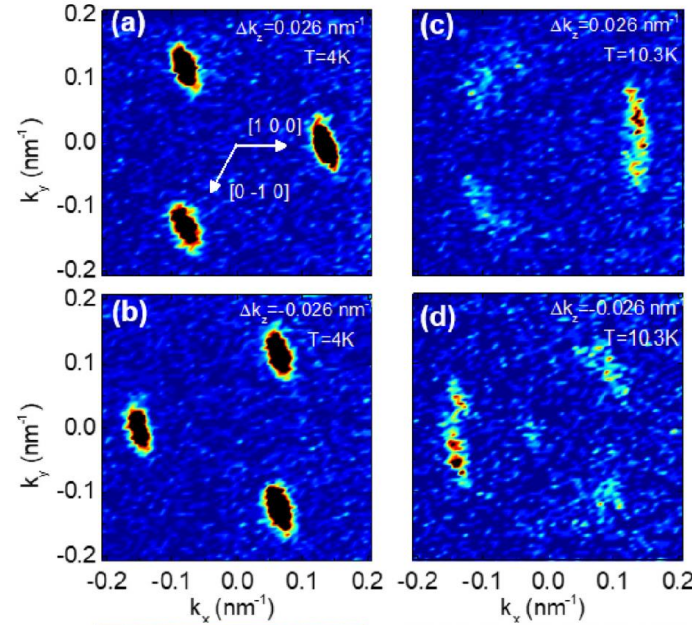
i) Unpolarized neutrons

ii) Polarized neutrons by S-bender SM and He-3 polarizer

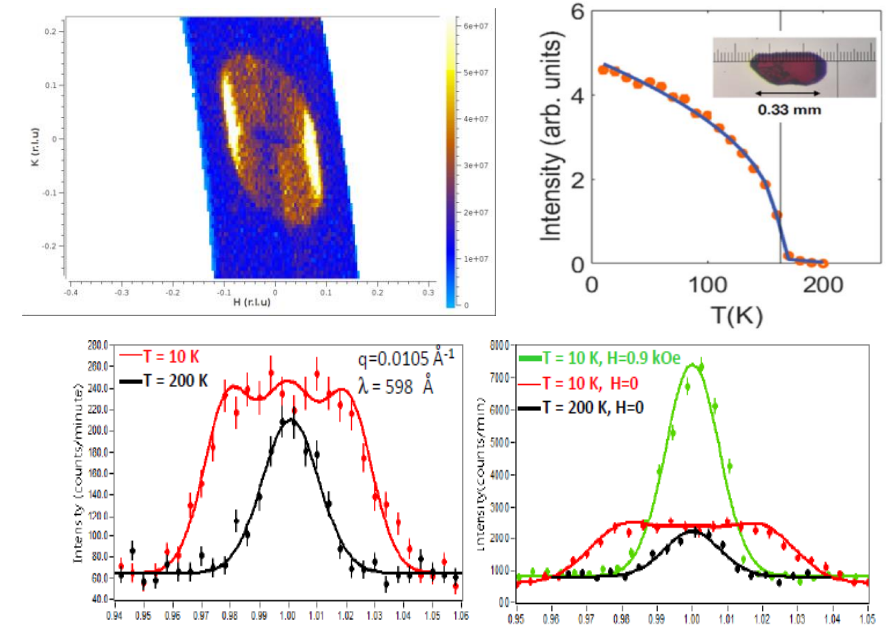


Capabilities of DEMAND at HB-3A HFIR

- ❖ Complex magnetism and structure
- ❖ New materials and phenomena
- ❖ Order parameter and phase transitions
- ❖ Long/short range magnetic order, local magnetic susceptibility, magnetic anisotropy
- ❖ Extreme condition tuning

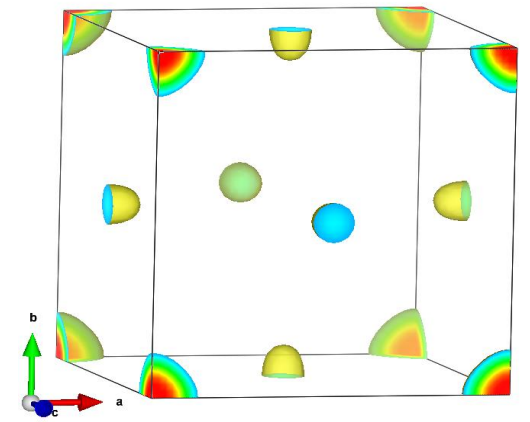
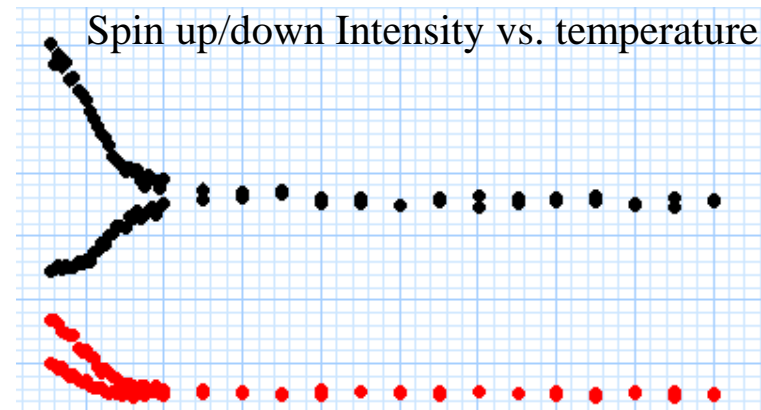
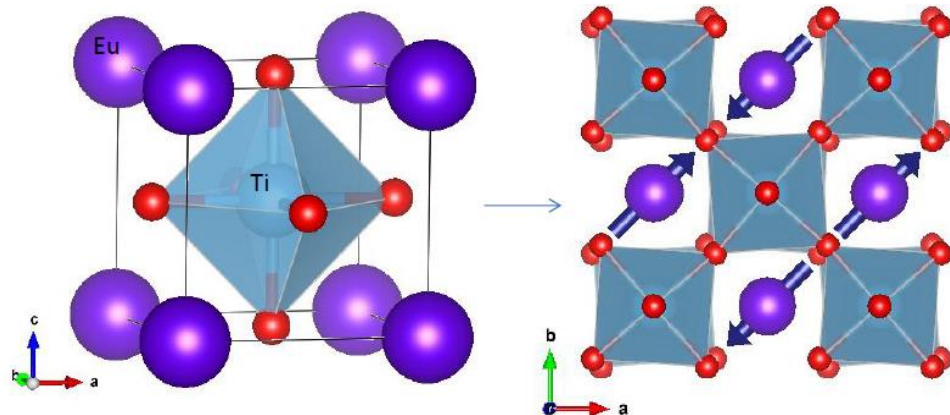


Physical Review Materials, **4**, 064419 (2020).



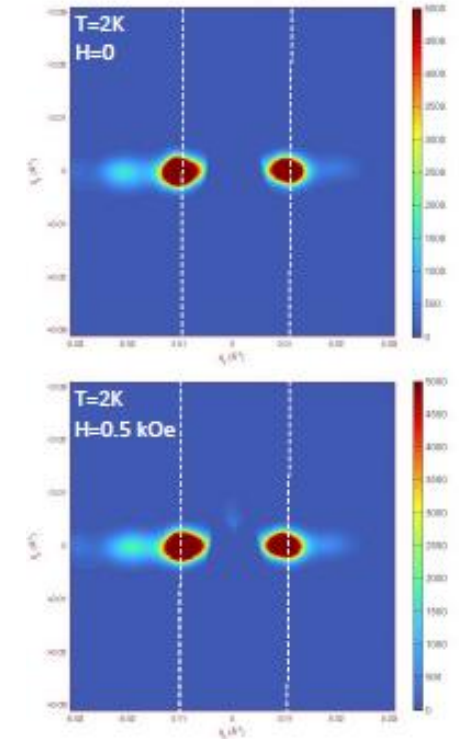
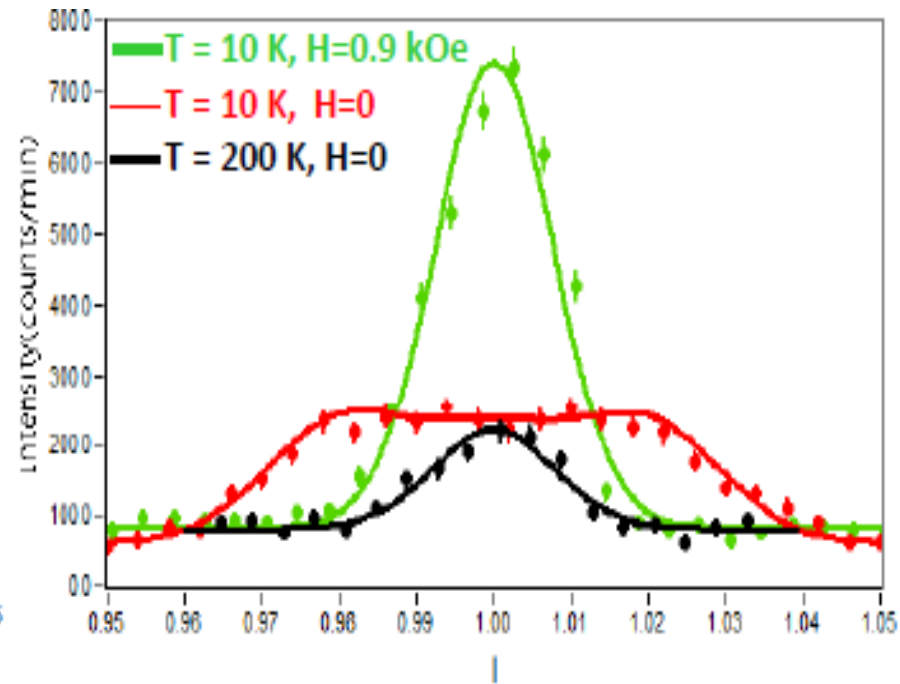
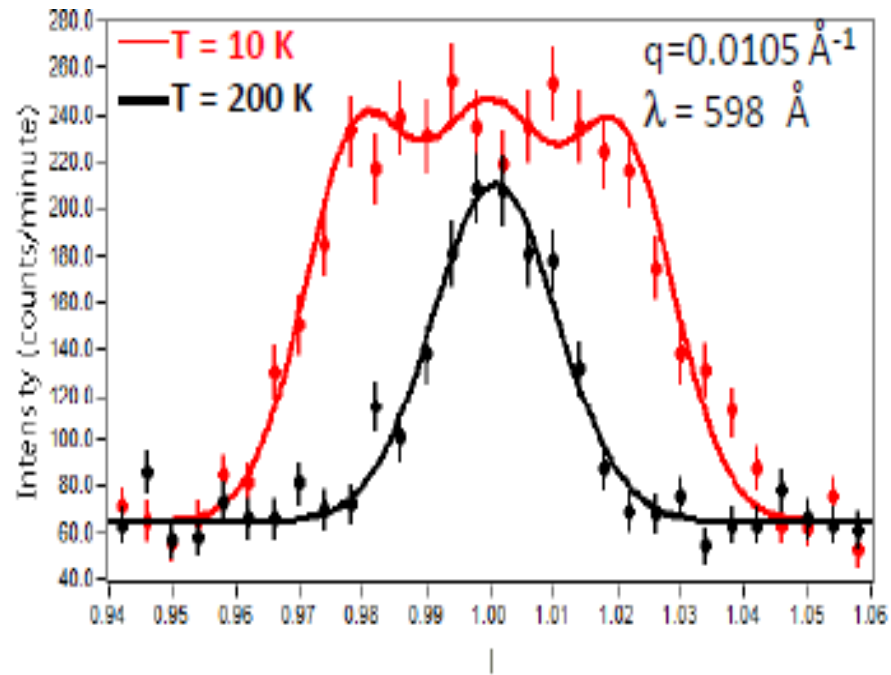
Physical Review B, **101**, 184417 (2020).

Polarized neutron diffraction



Single Crystal Diffraction

High Q-resolution



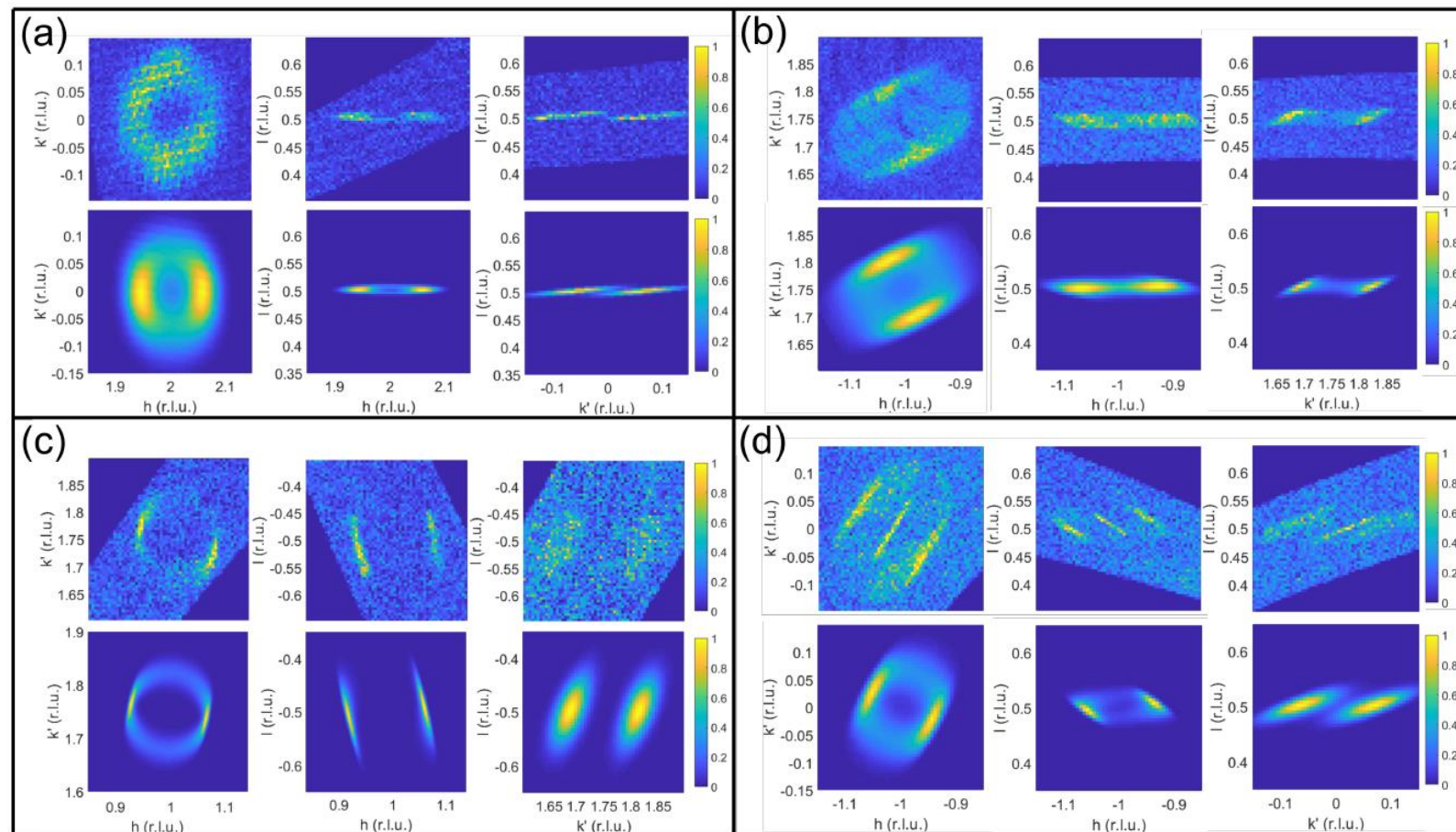
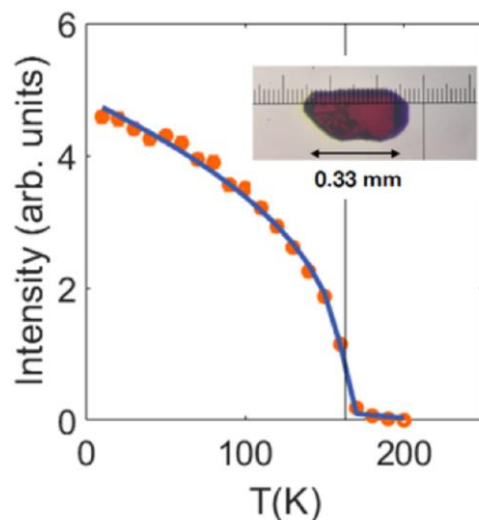
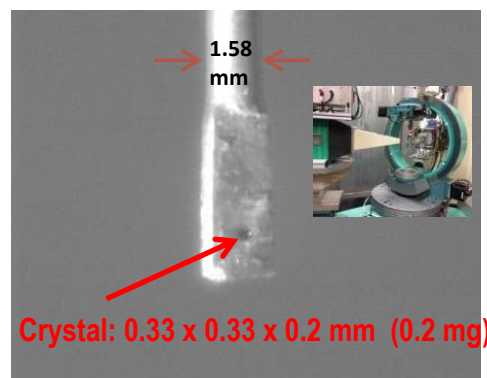
Thermal neutrons ===== Cold neutrons ===== \rightarrow SANS

Single Crystal Diffraction

High flux

Partial antiferromagnetic helical order in single-crystal $\text{Fe}_3\text{PO}_4\text{O}_3$

C. L. Sarkis, M. J. Tarne, J. R. Neilson, H. B. Cao, E. Coldren, M. P. Gelfand, and K. A. Ross
Phys. Rev. B **101**, 184417 – Published 18 May 2020



Single Crystal Diffraction

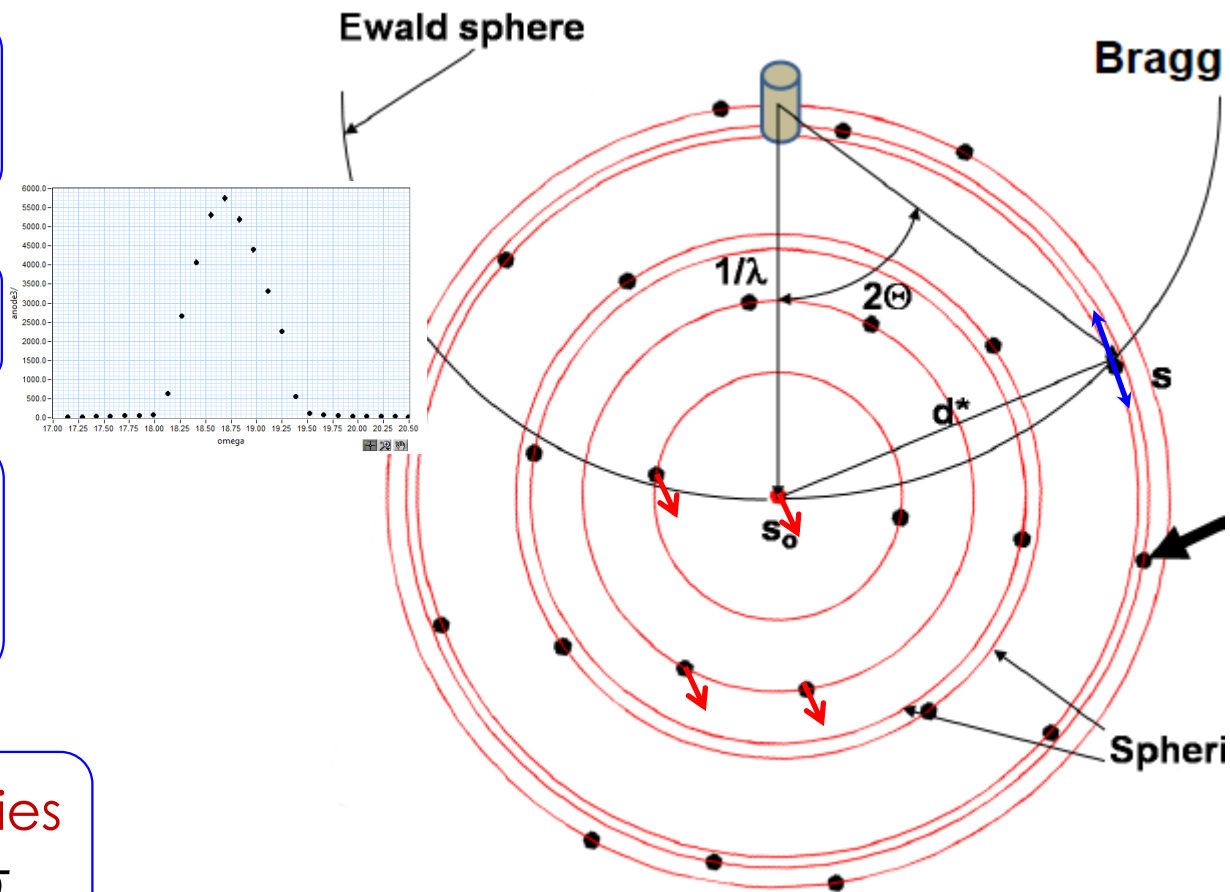
Lattice parameters
***k*-vector**

Data collection

Integration
Lorentz correction
Absorption correction

Corrected Integrated Intensities
 h k l $Int_{corr} (\propto F^2)$ σ

Structure solution



10 reflections for each refining parameter
Fixed lattice parameters used in data analysis

Single Crystal Diffraction

Use the magnetic symmetry with Bilbao crystallographic server



<https://www.youtube.com/watch?v=mBEiDP-Rz8s>

PhysRevB.100.144441.pdf

Structure

Orthorhombic

Space group

$Pbnm$

a (Å)

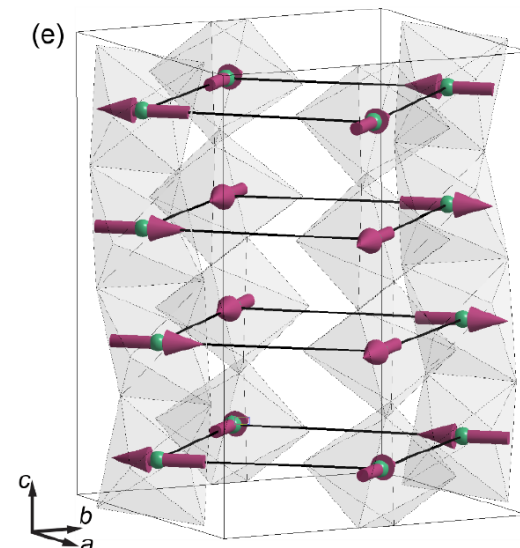
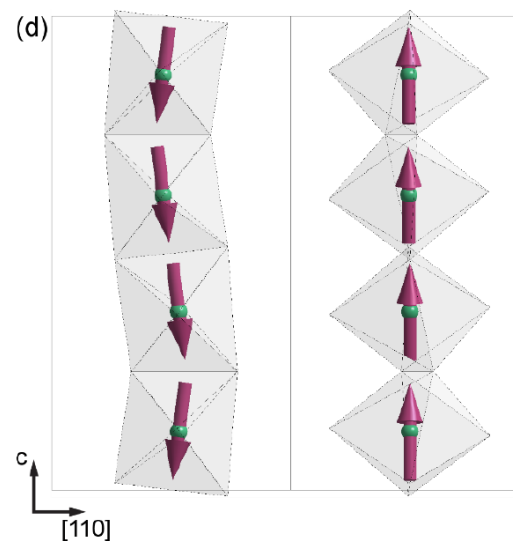
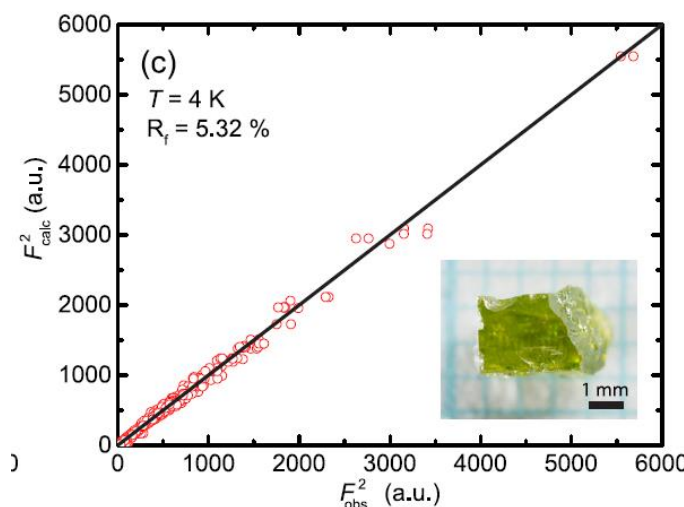
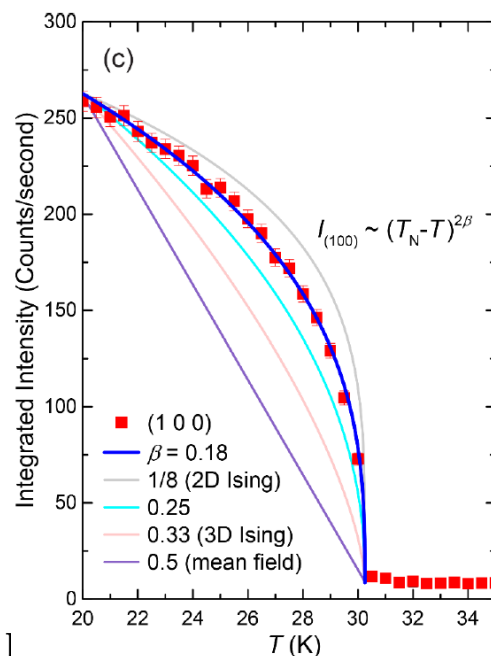
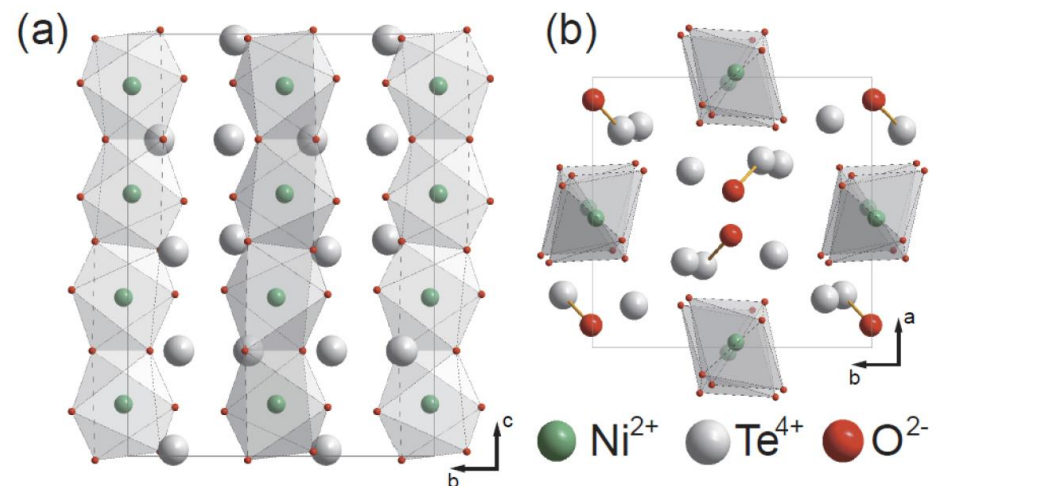
8.4418

b (Å)

8.8663

c (Å)

12.1203



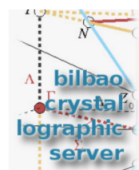
Single crystal diffraction example NiTe_2O_5

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bilbao crystallographic server

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workshops

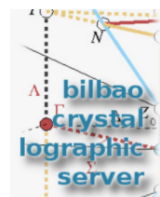
Space-group symmetry

Magnetic Symmetry and Applications

Group-Subgroup Relations of Space Groups

Representations and Applications

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in forthcoming schools and
workshops

News:

- **MAGNDATA reaches 1,000 entries**

06/2020: more than one thousand
published magnetic structures now

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Space-group symmetry

Magnetic Symmetry and Applications

[MGENPOS](#)

General Positions of Magnetic Space Groups

[MWYCKPOS](#)

Wyckoff Positions of Magnetic Space Groups

[MNORMALIZER](#)

Normalizers of Magnetic Space Groups

[IDENTIFY MAGNETIC GROUP](#)

Identification of a Magnetic Space Group from a set of generators in an arbitrary setting

[BNS2OG](#)

Transformation of symmetry operations between BNS and OG settings

[mCIF2PCR](#)

Transformation from mCIF to PCR format (FullProf).

[MPOINT](#)

Magnetic Point Group Tables

[MAGNEXT](#)

Extinction Rules of Magnetic Space Groups

[MAXMAGN](#)

Maximal magnetic space groups for a given space group and a propagation vector

[MAGMODE](#)

Magnetic structure models for any given magnetic symmetry

Single crystal diffraction example NiTe_2O_5

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 constraints 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 ICODISTORT, JANA2006, ShCrystal, VESTA, etc. These cif-like files

☒ 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)

1 *Pnma*

choose it

62

Please, enter the propagation vector k:

k_x

0

k_y

0

k_z

0

Submit

Parent paramagnetic structure cif file

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

1

Choose File

No file chosen

2

Upload the file

Note: The space group of the cif file will supersede any previous one.

Option 2: Specify structure data by hand:

Space Group: *Pnma* (No. 62)

Lattice parameters (Angstroms and degrees):

a= b= c= alpha=90 beta=90 gamma=90

Number of unique atomic positions:

Submit

NiTe2O5_powder X-ray.cif

Single crystal diffraction example NiTe_2O_5

Parent phase structure data: Magnetic Atoms

Parent space group: *Pnma* (No. 62)

Lattice parameters (Angstroms and degrees): $a=8.86640$, $b=12.12040$, $c=8.44180$, $\alpha=90.000$, $\beta=90.000$, $\gamma=90.000$

Atoms: Please select the magnetic ones

N	Atom name	Atom type	Wyckoff Position	Coordinates	Magnetic?
1	Te1	Te	8d	0.85150 0.48630 0.15960	<input type="checkbox"/>
2	Te2	Te	4c	0.10630 0.25000 0.19650	<input type="checkbox"/>
3	Te3	Te	4c	0.33470 0.25000 0.68170	<input type="checkbox"/>
4	Ni1	Ni	8d	0.51680 0.12270 0.98460	<input checked="" type="checkbox"/>
5	O1	O	4c	0.12570 0.25000 0.66770	<input type="checkbox"/>
6	O2	O	4c	0.45830 0.25000 0.12450	<input type="checkbox"/>
7	O3	O	8d	0.48600 0.13540 0.40880	<input type="checkbox"/>
8	O4	O	8d	0.68770 0.10950 0.14060	<input type="checkbox"/>
9	O5	O	8d	0.38430 0.48520 0.11530	<input type="checkbox"/>
10	O6	O	8d	0.34410 0.12770 0.82250	<input type="checkbox"/>



Submit

2

Single crystal diffraction example NiTe_2O_5

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

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	<i>Pn'm'a'</i> (#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	<i>Pn'ma'</i> (#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	<i>Pnm'a'</i> (#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	<i>Pn'm'a</i> (#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	<i>Pnma'</i> (#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	<i>Pnm'a</i> (#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	<i>Pn'ma</i> (#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	<i>Pnma</i> (#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

Try all possible magnetic symmetries, sometimes need to go lower symmetries for a solution. Here I pick the "*Pnma*" as the right option since I have tried all of them.

1

Single crystal diffraction example NiTe₂O₅

Magnetic Structure

Selected magnetic space group: 8- *Pnma* (#62.441)
Setting of the parent group
Parent space group *Pnma* (No. 62)
Lattice parameters: a=8.86640, b=12.12040, c=8.44180, alpha=90.000, beta=90.000, gamma=90.000

[Go to setting standard (a, b, c ; 0, 0, 0)]
[Go to an alternative setting]

1

Export data to MCIF file/Visualize Go to a subgroup

Atomic positions, Wyckoff positions and Magnetic Moments

N	Atom	New WP	Multiplicity	Magnetic moment	Values of M _x , M _y , M _z
1	Te1 Te 0.85150 0.48630 0.15960	(x,y,z m _x m _y m _z) (-x+1/2,-y,z+1/2 -m _x -m _y m _z) (-x,y+1/2,-z -m _x m _y -m _z) (x+1/2,-y+1/2,-z+1/2 m _x -m _y -m _z) (-x,-y,-z m _x m _y m _z) (x+1/2,y,-z+1/2 -m _x -m _y m _z) (x,-y+1/2,z -m _x m _y -m _z) (-x+1/2,y+1/2,z+1/2 m _x -m _y -m _z)	8	-	-
2	Te2 Te 0.10630 0.25000 0.19650	(x,1/4,z 0,m _y ,0) (-x+1/2,3/4,z+1/2 0,-m _y ,0) (-x,3/4,-z 0,m _y ,0) (x+1/2,1/4,-z+1/2 0,-m _y ,0)	4	-	-
3	Te3 Te 0.33470 0.25000 0.68170	(x,1/4,z 0,m _y ,0) (-x+1/2,3/4,z+1/2 0,-m _y ,0) (-x,3/4,-z 0,m _y ,0) (x+1/2,1/4,-z+1/2 0,-m _y ,0)	4	-	-
4	Ni1 Ni 0.51680 0.12270 0.98460	(x,y,z m _x m _y m _z) (-x+1/2,-y,z+1/2 -m _x -m _y m _z) (-x,y+1/2,-z -m _x m _y -m _z) (x+1/2,-y+1/2,-z+1/2 m _x -m _y -m _z) (-x,-y,-z m _x m _y m _z) (x+1/2,y,-z+1/2 -m _x -m _y m _z) (x,-y+1/2,z -m _x m _y -m _z) (-x+1/2,y+1/2,z+1/2 m _x -m _y -m _z)	8	(M _x ,M _y ,M _z)	M _x = <input type="text"/> M _y = <input type="text"/> M _z = <input type="text"/>
5	O1 O 0.12570 0.25000 0.66770	(x,1/4,z 0,m _y ,0) (-x+1/2,3/4,z+1/2 0,-m _y ,0) (-x,3/4,-z 0,m _y ,0) (x+1/2,1/4,-z+1/2 0,-m _y ,0)	4	-	-
6	O2 O 0.45830 0.25000 0.12450	(x,1/4,z 0,m _y ,0) (-x+1/2,3/4,z+1/2 0,-m _y ,0) (-x,3/4,-z 0,m _y ,0) (x+1/2,1/4,-z+1/2 0,-m _y ,0)	4	-	-
7	O3 O 0.48600 0.13540 0.40880	(x,y,z m _x m _y m _z) (-x+1/2,-y,z+1/2 -m _x -m _y m _z) (-x,y+1/2,-z -m _x m _y -m _z) (x+1/2,-y+1/2,-z+1/2 m _x -m _y -m _z) (-x,-y,-z m _x m _y m _z) (x+1/2,y,-z+1/2 -m _x -m _y m _z) (x,-y+1/2,z -m _x m _y -m _z) (-x+1/2,y+1/2,z+1/2 m _x -m _y -m _z)	8	-	-
8	O4 O 0.68770 0.10950 0.14060	(x,y,z m _x m _y m _z) (-x+1/2,-y,z+1/2 -m _x -m _y m _z) (-x,y+1/2,-z -m _x m _y -m _z) (x+1/2,-y+1/2,-z+1/2 m _x -m _y -m _z) (-x,-y,-z m _x m _y m _z) (x+1/2,y,-z+1/2 -m _x -m _y m _z) (x,-y+1/2,z -m _x m _y -m _z) (-x+1/2,y+1/2,z+1/2 m _x -m _y -m _z)	8	-	-
9	O5 O 0.38430 0.48520 0.11530	(x,y,z m _x m _y m _z) (-x+1/2,-y,z+1/2 -m _x -m _y m _z) (-x,y+1/2,-z -m _x m _y -m _z) (x+1/2,-y+1/2,-z+1/2 m _x -m _y -m _z) (-x,-y,-z m _x m _y m _z) (x+1/2,y,-z+1/2 -m _x -m _y m _z) (x,-y+1/2,z -m _x m _y -m _z) (-x+1/2,y+1/2,z+1/2 m _x -m _y -m _z)	8	-	-
10	O6 O 0.34410 0.12770 0.82250	(x,y,z m _x m _y m _z) (-x+1/2,-y,z+1/2 -m _x -m _y m _z) (-x,y+1/2,-z -m _x m _y -m _z) (x+1/2,-y+1/2,-z+1/2 m _x -m _y -m _z) (-x,-y,-z m _x m _y m _z) (x+1/2,y,-z+1/2 -m _x -m _y m _z) (x,-y+1/2,z -m _x m _y -m _z) (-x+1/2,y+1/2,z+1/2 m _x -m _y -m _z)	8	-	-



mCIF file of the structure

Submit this mcif file to MVisualize for 3D visualization of the structure using Jmol:

Submit to MVisualize

Download mCIF file: bcs_file.mcif

[The preview text below is non-editable, only copy-allowed]

```
#CIF 2.0
# Created by the Bilbao Crystallographic Server
# http://www.cryst.ehu.es
# Date: 27/09/2020 07:02:10
# NiTe2O5_powder X-ray.cif

data_5yOhtAoR
_audit_creation_date 2020-09-27
_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
? ? ? ? ? ? ?

_exptl_crystal_magnetic_properties_details
;
;

_active_magnetic_irreps_details
;
k-maximal magnetic symmetry
;

_parent_space_group.name_H-M_alt 'P n m a'
_parent_space_group.IT_number 062
_parent_space_group.transform_Pp_abc 'a,b,c;0,0,0'
```

Single crystal diffraction example NiTe_2O_5

bcs_file_23269.mcif



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

Choose a structure file (mCIF format):

Choose File No file chosen

Convert

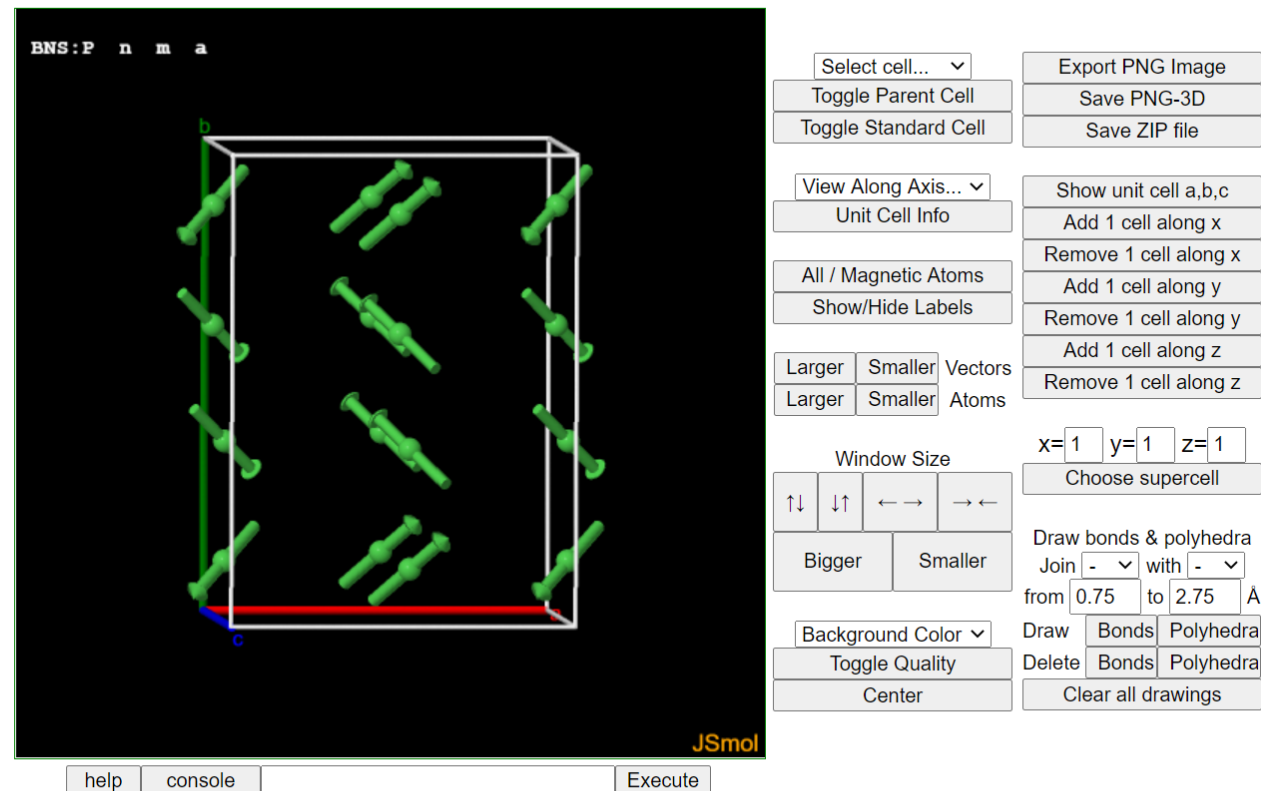


bcs_file_23269.pcr

MVISUALIZE Main Page

Show/Hide File

MVISUALIZE: 3D Visualization of magnetic structures with Jmol



Note: If the application stops working right or any malfunction is observed, it is probably a temporal problem due to the cache memory of your browser. Clear your web browser cache to solve it. If you still observe any malfunction, write an e-mail to cryst@wm.lc.edu explaining the problem in detail.

Single crystal diffraction example NiTe_2O_5

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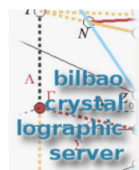
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Space-group symmetry

Magnetic Symmetry and Applications

Group-Subgroup Relations of Space Groups

Representations and Applications



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Space-group symmetry

Magnetic Symmetry and Applications

MGENPOS

General Positions of Magnetic Space Groups

MWYCKPOS

Wyckoff Positions of Magnetic Space Groups

MNORMALIZER

Normalizers of Magnetic Space Groups

IDENTIFY MAGNETIC GROUP

Identification of a Magnetic Space Group from a set of generators in an arbitrary setting

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Transformation of symmetry operations between BNS and OG settings

mCIF2PCR

Transformation from mCIF to PCR format (FullProf).

MPOINT

Magnetic Point Group Tables

MAGNEXT

Extinction Rules of Magnetic Space Groups

MAXMAGN

Maximal magnetic space groups for a given space group and a propagation vector

MAGMODELIZE

Magnetic structure models for any given magnetic symmetry

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workshops

News:

- **MAGNDATA reaches 1,000 entries**

06/2020: more than one thousand
published magnetic structures have been

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

Choose a structure file (mCIF format):

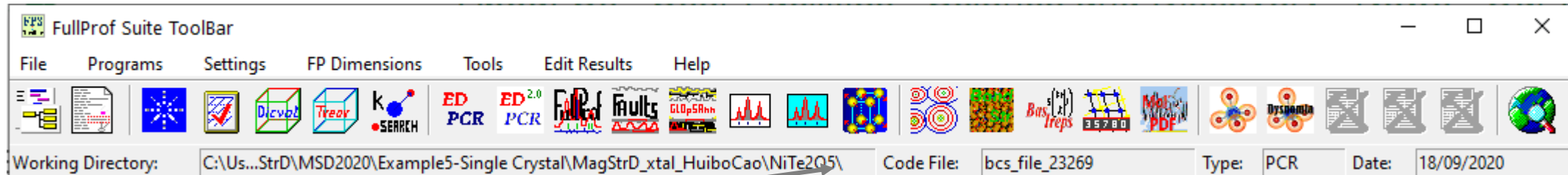
Choose File No file chosen

Convert

bcs_file_23269.pcr

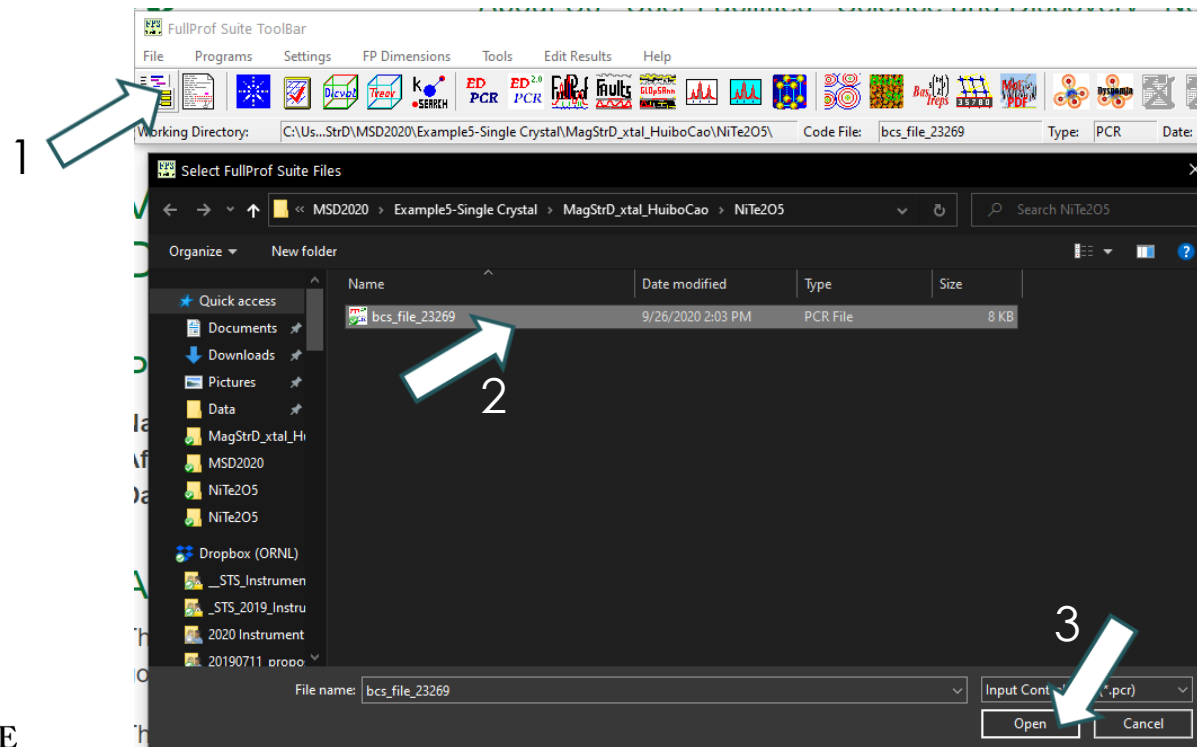
bcs_file_23269.mcif

Single crystal diffraction example NiTe_2O_5

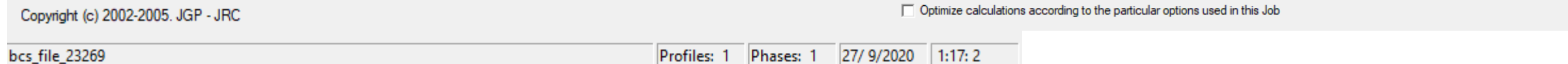
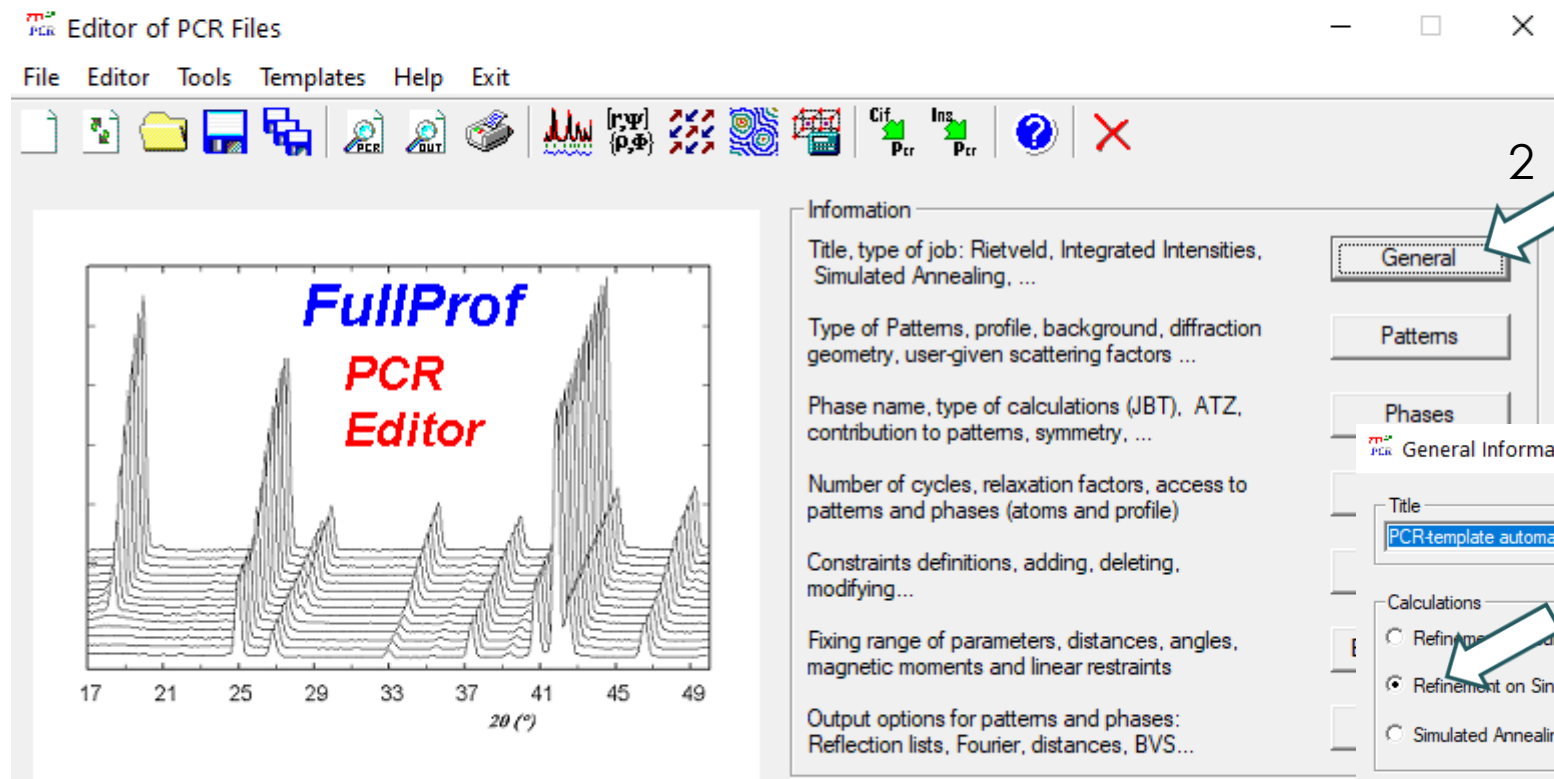
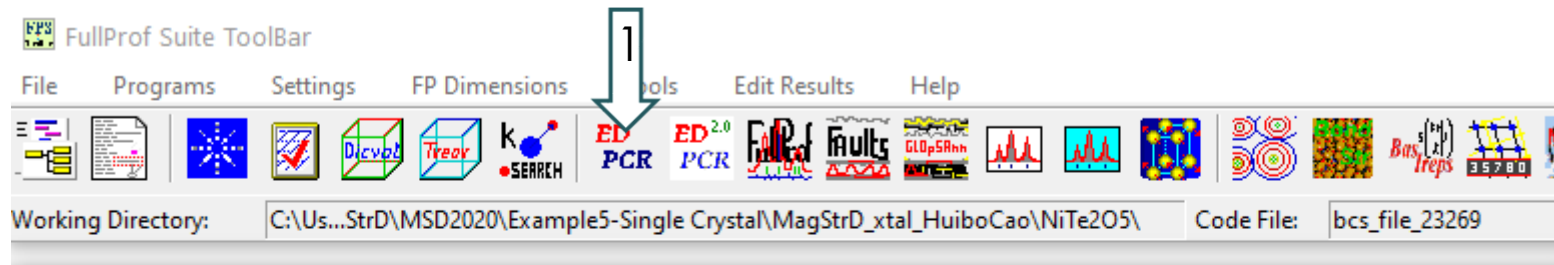


bcs_file_23269.pcr

Copy the created pcr to the folder with the data file (.int), then select the bcs_file_23269.pcr



Single crystal diffraction example NiTe_2O_5



19



Single crystal diffraction example NiTe_2O_5

The screenshot shows the FullProf PCR Editor interface. The main window displays a diffraction pattern with the text "FullProf PCR Editor". A green arrow labeled "1" points to the "Refinement" button in the Information panel. Below the main window, the "Refinement Information" dialog is open, showing various settings for refinement, including "Cycles of Refinement" (1), "Stop Criterion of Convergence" (Forced Termination when shifts < 0.02 x E.S.D.), and "Reflections ordering" (Only at the first cycle). A green arrow labeled "2" points to the "Intensities" button in the "Refinement Information" dialog. Below the "Refinement Information" dialog, the "Integrate Intensities: Phase 1 Pattern 1" dialog is open, showing "Cell Parameters" (a, b, c, alpha, beta, gamma) and "Scale Factors" (1, 2, 3, 4, 5, 6). A green arrow labeled "3" points to the "Scale Factors" table.

	a	b	c	alpha	beta	gamma
Coefficients	8.6	12.12040	8.44180	90.00	90.00	90.00

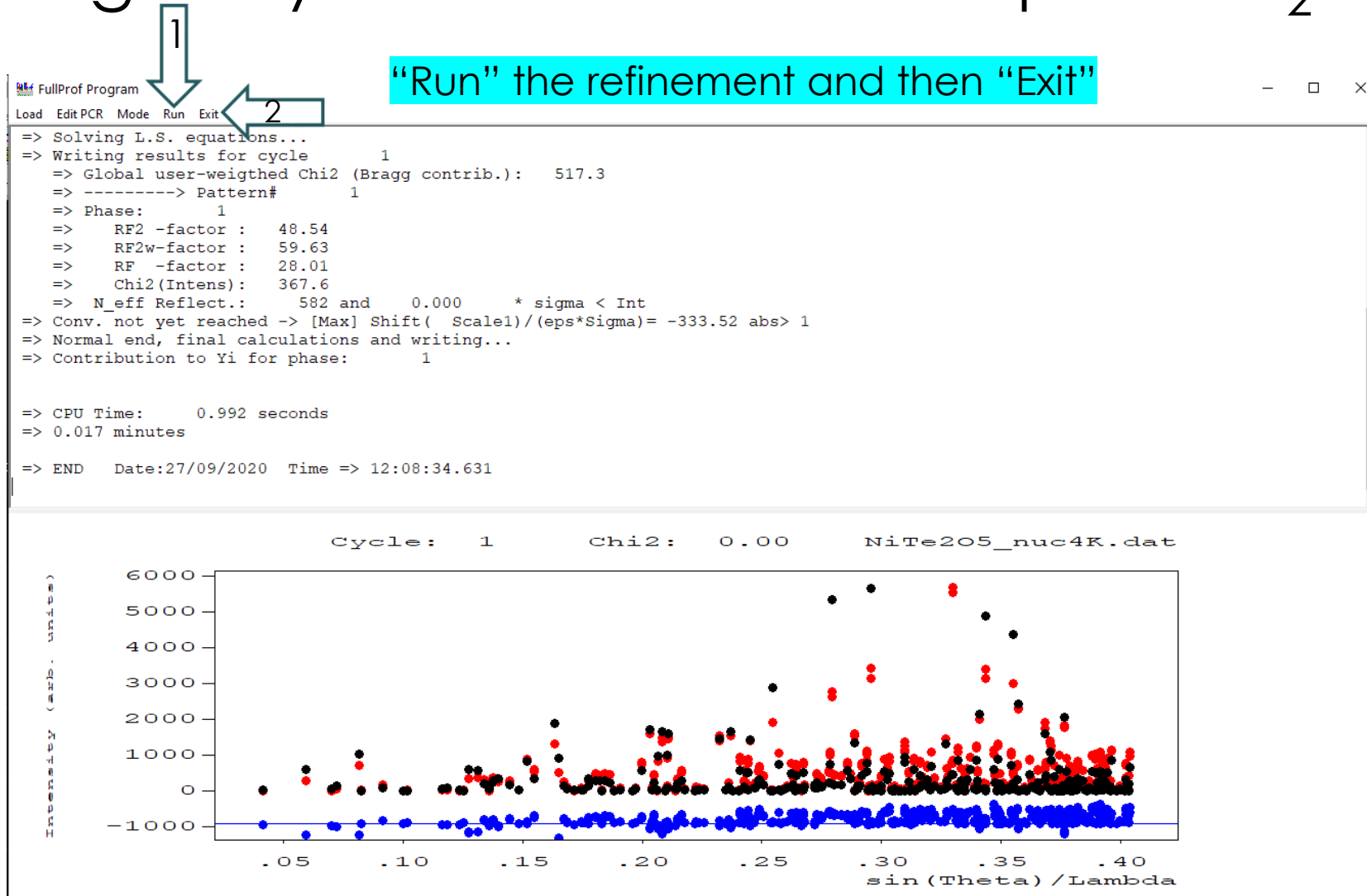
	1	2	3	4	5	6
Coefficients	1.0000	0.0000	0.0000	0.0000	0.0000	0.0000

	1	2	3	4	5	6
Coefficients	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

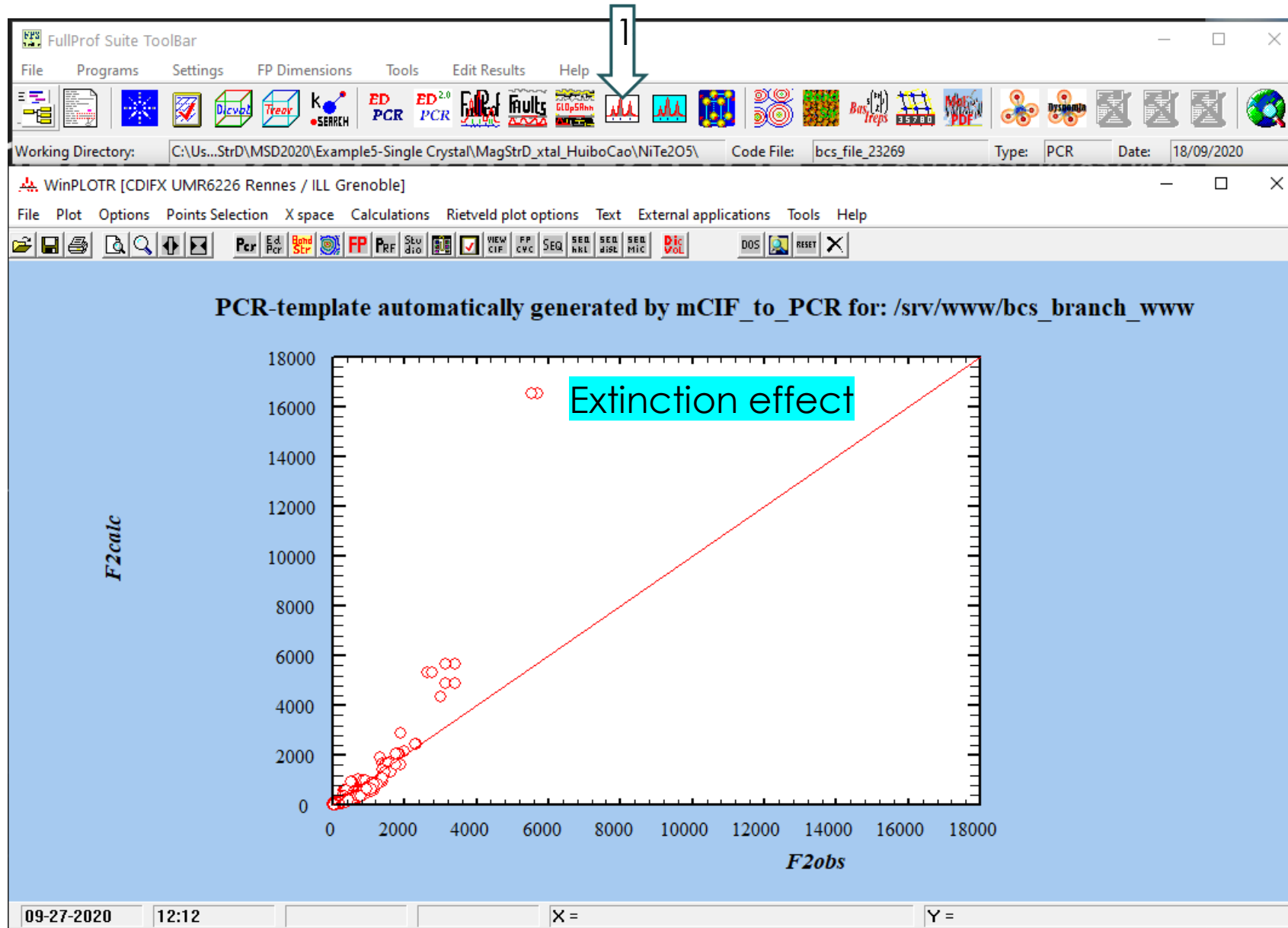
The screenshot shows the "Fullprof Files (DAT, INT, etc)" dialog. The "File name" field is set to "NiTe2O5_nuc4K". The "Fullprof Data File" dropdown is set to "Fullprof Data File". The "Open" button is highlighted. A green arrow labeled "1" points to the "FullProf PCR Editor" icon in the top toolbar. A green arrow labeled "2" points to the "NiTe2O5_nuc4K" file in the file list.

Name	Date modified	Type	Size
NiTe2O5_nuc4K	6/17/2018 1:53 PM	INT File	47 KB
NiTe2O5_nuc300K	6/17/2018 2:45 PM	INT File	18 KB

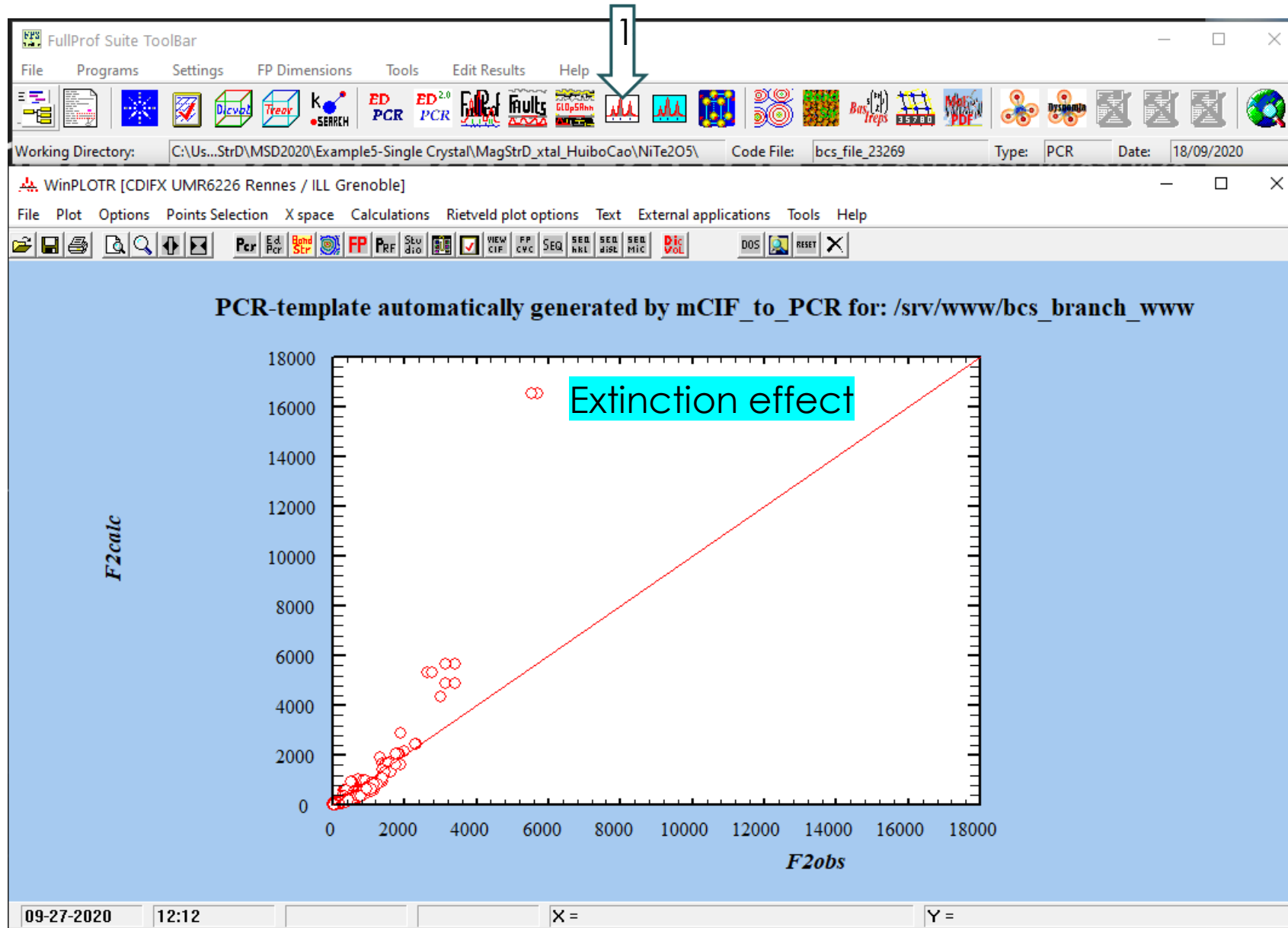
Single crystal diffraction example NiTe_2O_5



Single crystal diffraction example NiTe_2O_5



Single crystal diffraction example NiTe_2O_5



Single crystal diffraction example NiTe_2O_5

FullProf Suite ToolBar

File Programs Settings FP Dimensions Tools Edit Results Help

Working Directory: C:\Usr\MSD2020\Example5-Single Crystal\MagStrD_xtal_HuiboCao\NiTe2O5\ Code File: bcs_file_23269 Type: PCR Date: 18/09/2020

Editor of PCR Files

Information

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

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

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

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

Constraints definitions, adding, deleting, modifying...

Fixing range of parameters, distances, angles, magnetic moments and linear restraints

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

General Patterns Phases Refinement Constraints

Refinement Information

Cycles of Refinement: 10

Stop Criterion of Convergence

Forced Termination when shifts < 0.02 x E.S.D.

Others: None

Reflections ordering

Only at the first cycle Each cycle

Pattern 1 Pattern 2 Pattern 3 Pattern 4 Pattern 5 Pattern 6

Phase 1 Phase 2 Phase 3 Phase 4 Phase 5 Phase 6 Phase 7

Refinement weighting model

Least Squares Maximum Likelihood Unit Weights

Background Instrumental Micro-Absorption

Integrate Intensities: Phase 1 Pattern 1

Cell Parameters

	a	b	c	alpha	beta	gamma
Coefficients	8.86640	12.12040	8.44180	90.00	90.00	90.00

Scale Factors

	1	2	3	4	5	6
Coefficients	12.370	0.0000	0.0000	0.0000	0.0000	0.0000

Extinction Parameters

	1	2	3	4	5	6
Coefficients	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

After "OK" all the changes, "Save" and "Run" the refinement again

FullProf Program

Load Edit PCR Mode Run Exit

```
=> N_eff Reflect.: 582 and 0.000 * sigma < Int
=> Convergence reached at this CYCLE !!!!: CYCLE No. 1
=> Global user-weighted Chi2 (Bragg contrib.): 12.00
=> -----> Pattern# 1
=> Phase: 1
=> RF2 -factor : 6.708
=> RF2w-factor : 10.77
=> RF -factor : 5.989
=> Chi2(Intens): 12.00
=> N_eff Reflect.: 582 and 0.000 * sigma < Int
=> Normal end, final calculations and writing...
=> Contribution to Yi for phase: 1

=> CPU Time: 1.094 seconds
=> 0.018 minutes
=> END Date:27/09/2020 Time => 12:19:47.071
```

Cycle: 10 Chi2: 0.00 NiTe2O5_nuc4K.dat

Intensity (arb. units)

sin(Theta)/Lambda

PCR-template automatically generated by mCIF_to_PCR for: /srv/www/bcs_branch_www

Check the fitting quality

Single crystal diffraction example NiTe_2O_5

FullProf Suite ToolBar

File Programs Settings FP Dimensions Tools Edit Results Help

Working Directory: C:\Users\StrD\MSD2020\Example5-Single Crystal\MagStrD_xtal_HuiboCao\NiTe2O5\ Code File: bcs_file_23269 Type: PCR Date: 18/09/2020

FullProf Editor of PCR Files

File Editor Tools Templates Help Exit

Information

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

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

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

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

Refinement Information

Cycles of Refinement: 10

Stop Criterion of Convergence

Forced Termination when shifts < 0.02 x E.S.D.

Others: None

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 6

Phase 1 Phase 2 Phase 3 Phase 4 Phase 5 Phase 6

Refinement weighting model

Least Squares Background

OK Cancel

Atoms

Prop. Vectors

Atoms Information: Phase 1

List of Atoms

Number of Atoms: 10

Atom #	Label	Ntyp	Mag. Rot.	Prog. V...	X	Y	Z	B	Occ
Atom # 1	Te1	Te	1	0	0.85193	0.48685	0.16031	0.28596	1.00000
Atom # 2	Te2	Te	1	0	0.10888	0.25000	0.19749	0.34603	0.50000
Atom # 3	Te3	Te	1	0	0.33586	0.25000	0.67935	0.21725	0.50000
Atom # 4	Ni1	MN12	1	0	0.51769	0.12314	0.98469	0.29089	1.00000

Atom #	Re[x]	Re[y]	Re[z]	Im[x]	Im[y]	Im[z]	MPhase
Atom #4	0.31067	2.12667	0.26563	0.00000	0.00000	0.00000	0.00000

#	B11/F1	B22/F2	B33/F3	B12/F4	B13/F5	B23/F6	F7
#							
#							
#							

Refine Position

Refine B_{iso}

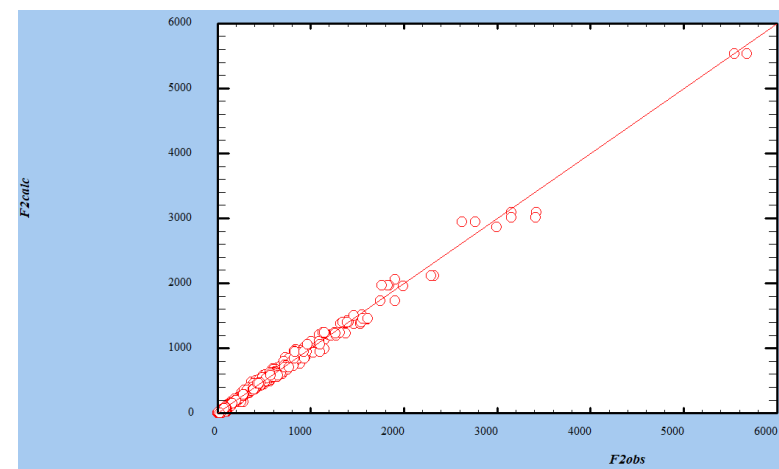
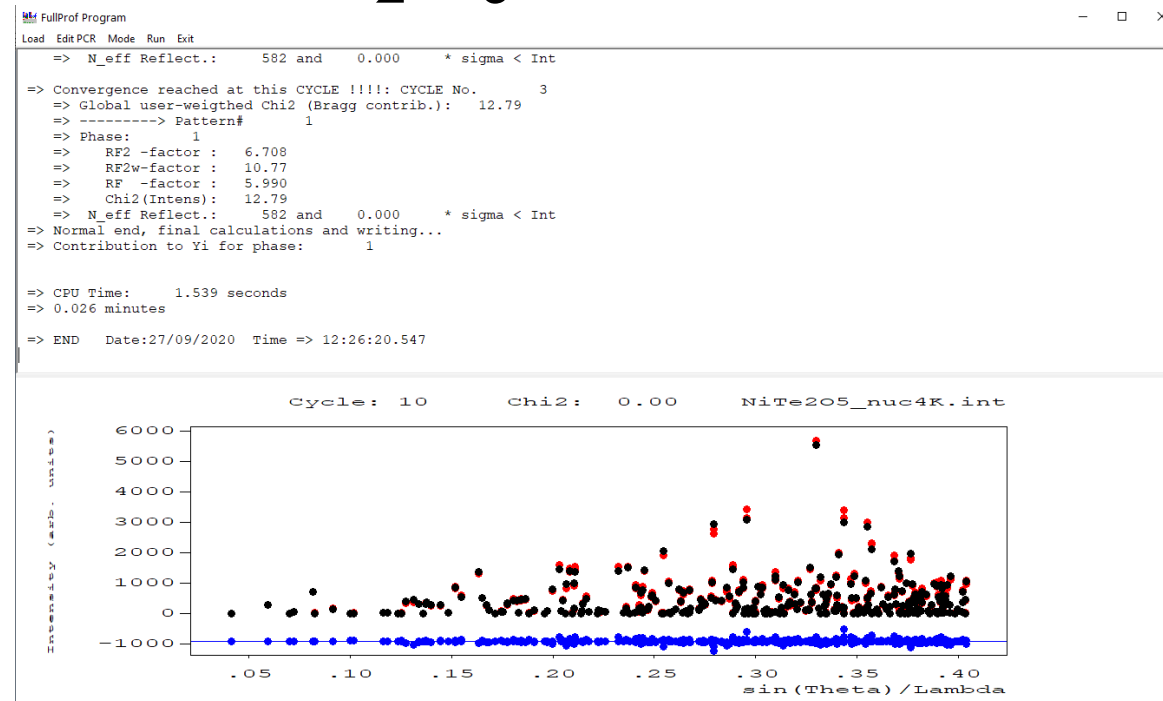
Refine B_{aniso}

Fix All

Cancel

OK

Refine all the atomic parameters



Single crystal diffraction example NiTe_2O_5

FullProf Suite ToolBar

File Programs Settings FP Dimensions Tools Edit Results Help

Working Directory: C:\Us...StrD\MSD2020\Example5-Single Crystal\MagStrD_xtal_HuiboCao\NiTe2O5\ Code File: bcs_file_23269 Type: PCR Date: 18/09/2020

```

Te3   Te      1  0   0.33585  0.25000  0.67935  0.21603  0.50000  0  0  #
      131.00      0.00      151.00      201.00      0.00
Ni1   MNI2     1  0   0.51769  0.12313  0.98469  0.28998  1.00000  1  0  #
      161.00      171.00      181.00      231.00      0.00
      0.31025  2.12711  0.26565  0.00000  0.00000  0.00000  0.00000  <-MagPar
      31.00      21.00      11.00      0.00      0.00      0.00
O1    O        1  0   0.12318  0.25000  0.65558  0.49032  0.50000  0  0  #
      191.00      0.00      211.00      371.00      0.00
O2    O        1  0   0.46745  0.25000  0.13392  0.36884  0.50000  0  0  #
      221.00      0.00      241.00      381.00      0.00
O3    O        1  0   0.49227  0.12742  0.40601  0.54906  1.00000  0  0  #
      251.00      261.00      271.00      391.00      0.00
O4    O        1  0   0.69194  0.11397  0.14618  0.45197  1.00000  0  0  #
      281.00      291.00      301.00      401.00      0.00
O5    O        1  0   0.38275  0.48647  0.12018  0.47116  1.00000  0  0  #
      311.00      321.00      331.00      411.00      0.00
O6    O        1  0   0.34572  0.12987  0.81991  0.64499  1.00000  0  0  #
      341.00      351.00      361.00      421.00      0.00

!-----> Scale, Extinction and Cell Parameters for Pattern # 1
! Scale Factors
! Sc1      Sc2      Sc3      Sc4      Sc5      Sc6
! 55.74     0.000    0.000    0.000    0.000    0.000
! 41.00     0.00     0.00     0.00     0.00     0.00
! Extinction Parameters
! Ext1      Ext2      Ext3      Ext4      Ext5      Ext6      Ext7      Ext-Model
! 32.50     0.000    0.000    0.000    0.000    0.000    0.000    1
! 61.00     0.00     0.00     0.00     0.00     0.00     0.00     0.00
! a          b          c          alpha      beta      gamma      #Cell Info
! 8.866400  12.120400  8.441799  90.000000  90.000000  90.000000  #
! 0.000000  0.000000  0.000000  0.000000  0.000000  0.000000
! x-Lambda/2 + Not yet used parameters
! 0.00     0.00000  0.00000  0.00000  0.00000
! 1         2         0.00     0.00     0.00
! 2Th1/TOF1 2Th2/TOF2 Pattern to plot
! 2.000    130.000    1
  
```

! x-Lambda/2 + Not yet used parameters
0.01158 0.00000 0.00000 0.00000 0.00000
51.00 0.00 0.00 0.00 0.00

Open the .pcr file in a text editor, flag Lambda/2 (half lambda contamination) on by change the "code" "0" to "1". Then do the refinement again

Single crystal diffraction example NiTe_2O_5

Open the .sum file generated from the refinement to see the refined atomic and ordered moment parameters. The parameters can be found in the .cif and the .mcif files too.

=> No. of reflections for pattern#: 1: 582

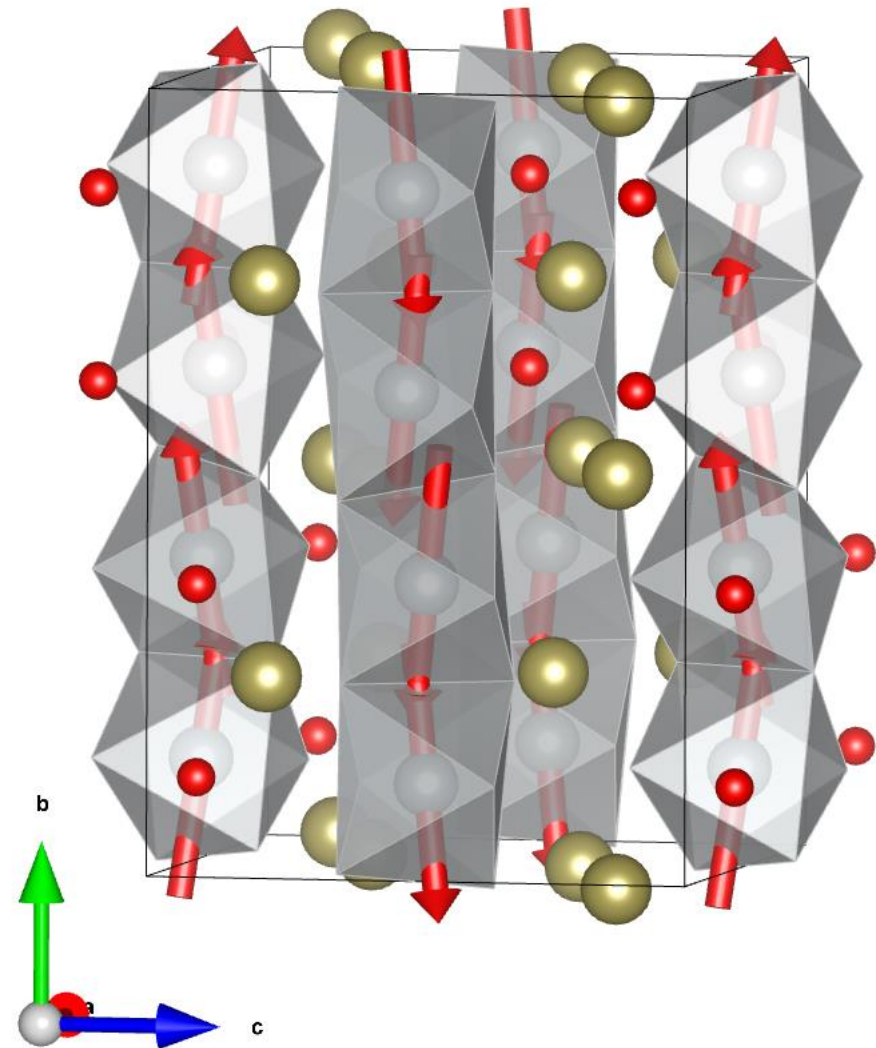
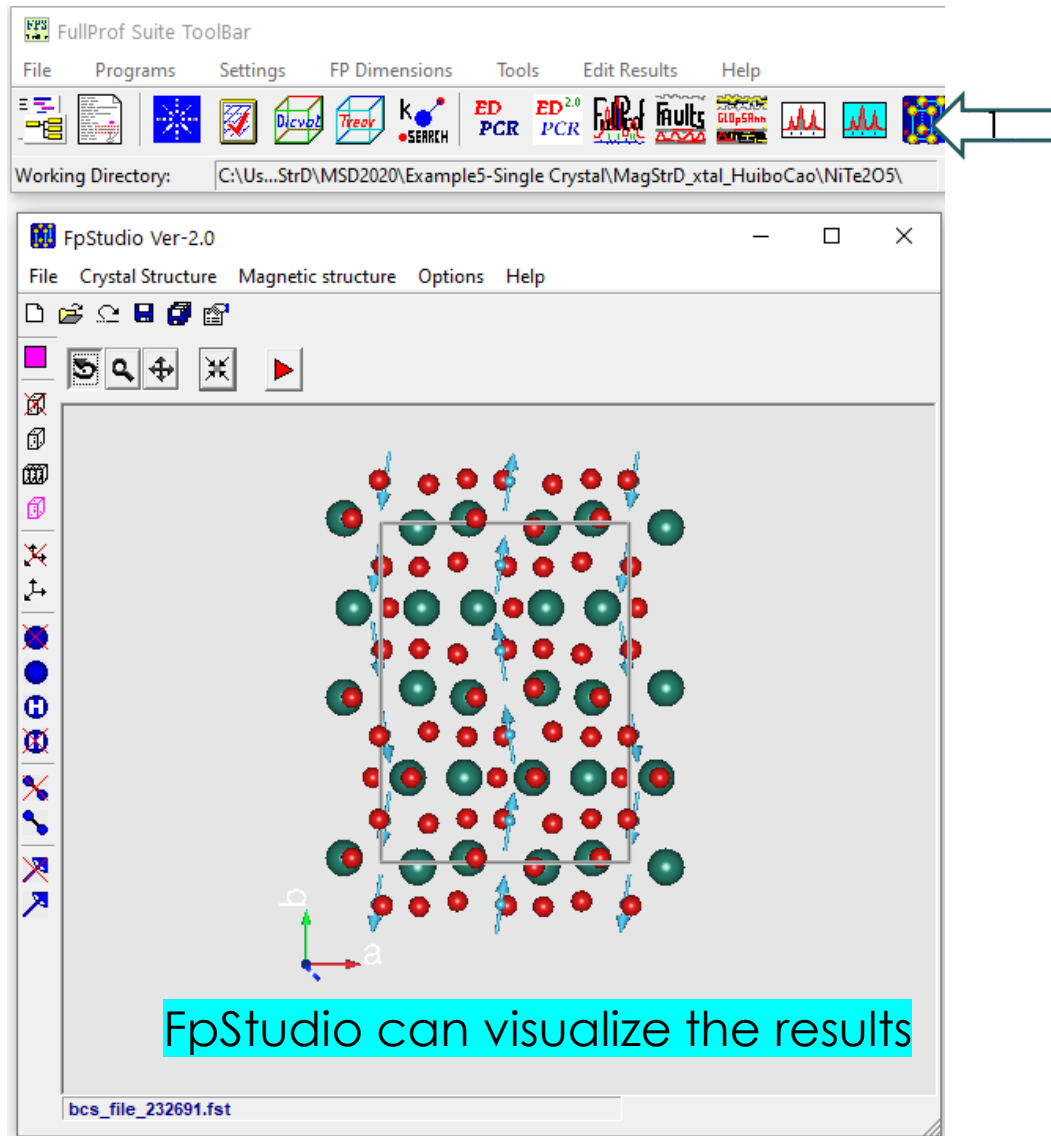
==> ATOM PARAMETERS:

Name	x	sx	y	sy	z	sz	B	sB	occ.	socc.	Mult
Te1	0.85193(37)		0.48684(22)		0.16031(35)		0.285(85)		1.000(0)		8
Te2	0.10888(53)		0.25000(0)		0.19749(47)		0.345(112)		0.500(0)		4
Te3	0.33585(51)		0.25000(0)		0.67935(47)		0.216(109)		0.500(0)		4
Ni1	0.51769(21)		0.12313(14)		0.98469(19)		0.290(69)		1.000(0)		8
O1	0.12318(53)		0.25000(0)		0.65558(51)		0.490(109)		0.500(0)		4
O2	0.46745(54)		0.25000(0)		0.13392(51)		0.369(107)		0.500(0)		4
O3	0.49227(40)		0.12742(24)		0.40601(37)		0.549(92)		1.000(0)		8
O4	0.69194(37)		0.11397(22)		0.14618(39)		0.452(89)		1.000(0)		8
O5	0.38275(37)		0.48647(22)		0.12018(37)		0.471(81)		1.000(0)		8
O6	0.34572(39)		0.12987(23)		0.81991(37)		0.645(85)		1.000(0)		8

==> MAGNETIC MOMENT PARAMETERS:

Name	Mx	sMx	My	sMy	Mz	sMz	M	sM	MPhas	sMPhas
Ni1	0.310(57)		2.127(35)		0.266(42)		2.1660(367)		0.0000(0)	

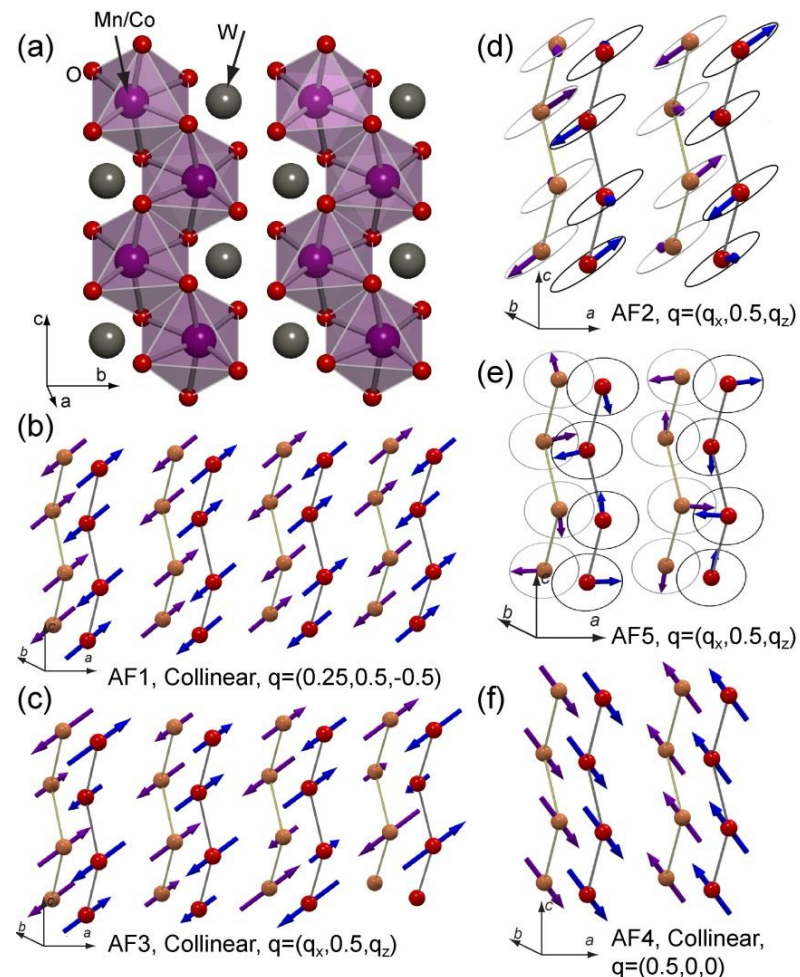
Single crystal diffraction example NiTe_2O_5



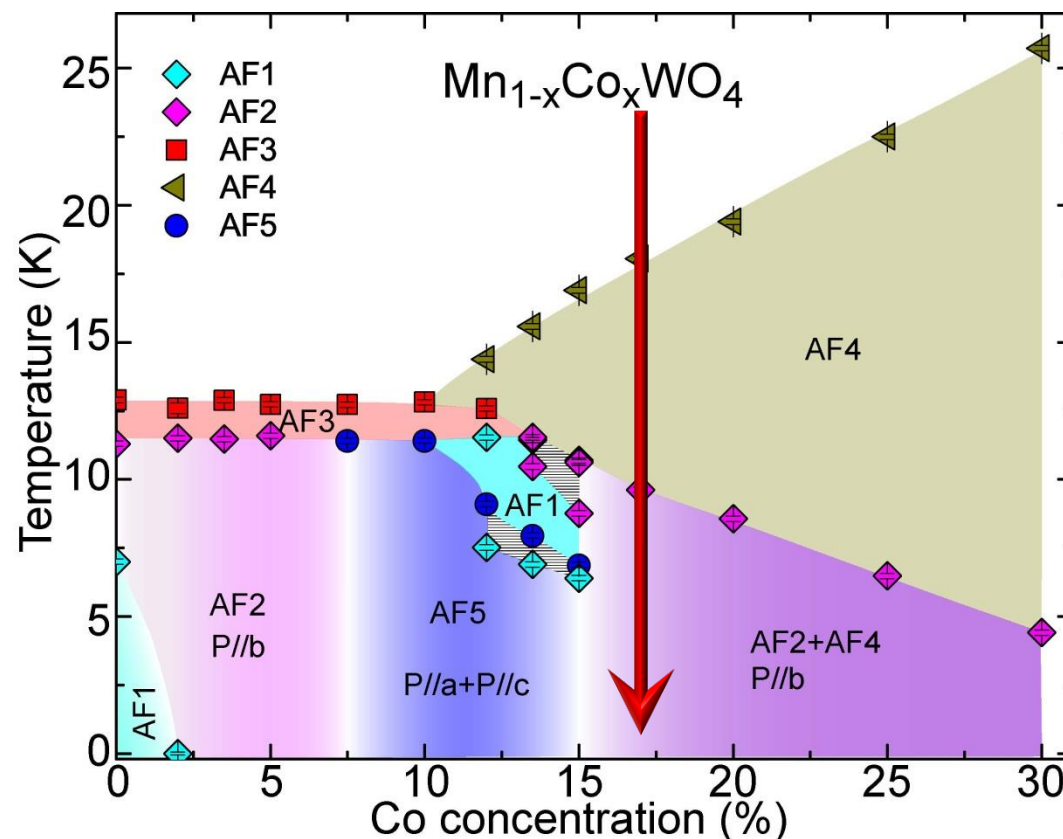
Vesta is another software can read the .mcif or .vesta file generated from FP refinement

Single crystal diffraction **Magnetic Structures in $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$**

Phase with both Commensurate AF and Incommensurate AF is selected: 17% Co-doping MnWO_4



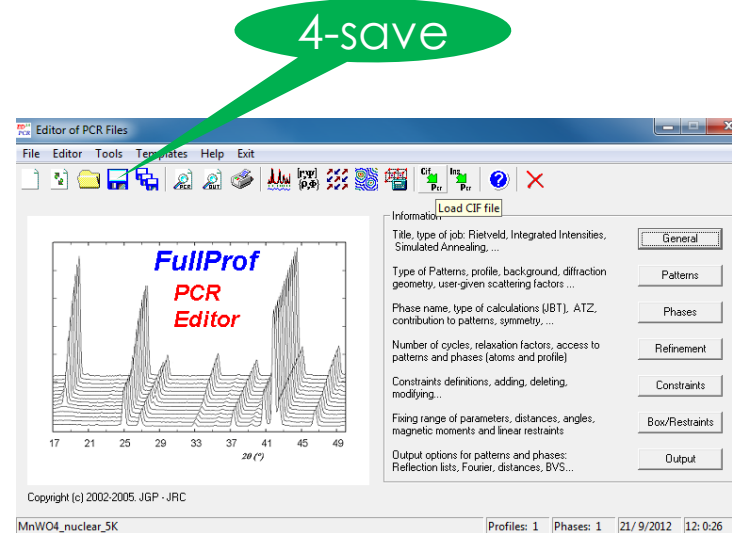
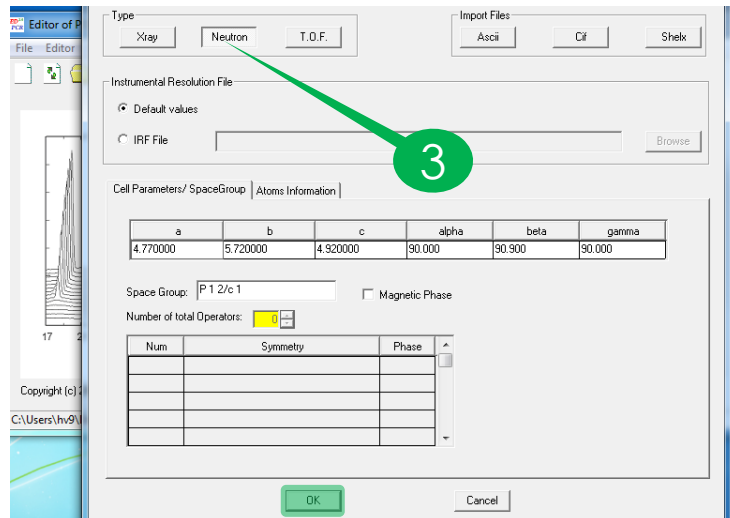
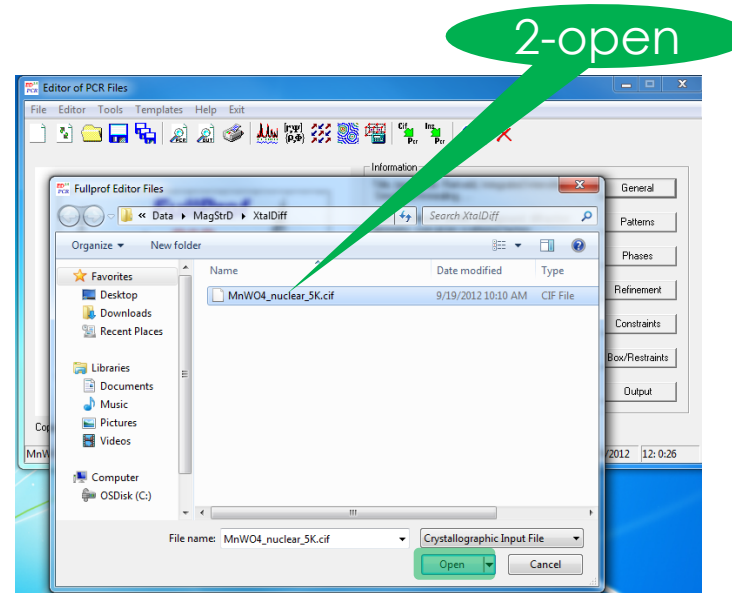
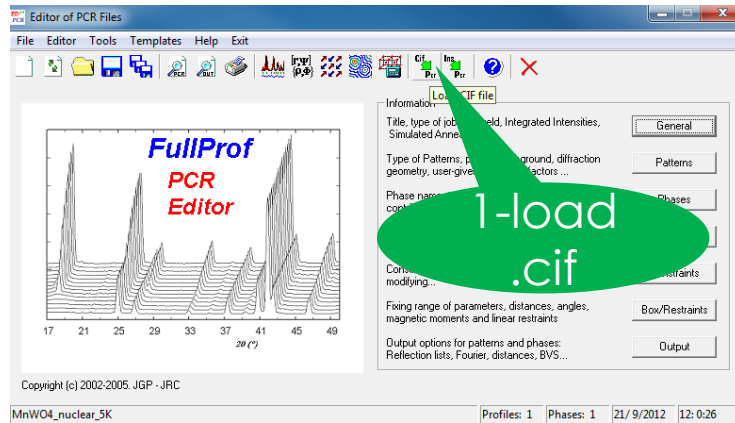
Feng Ye et al., Phys. Rev. B (2012)



$P 1 2/c 1$ $a=4.77 \text{ \AA}$ $b=5.72 \text{ \AA}$ $c=4.92 \text{ \AA}$ $\beta=90.9^\circ$

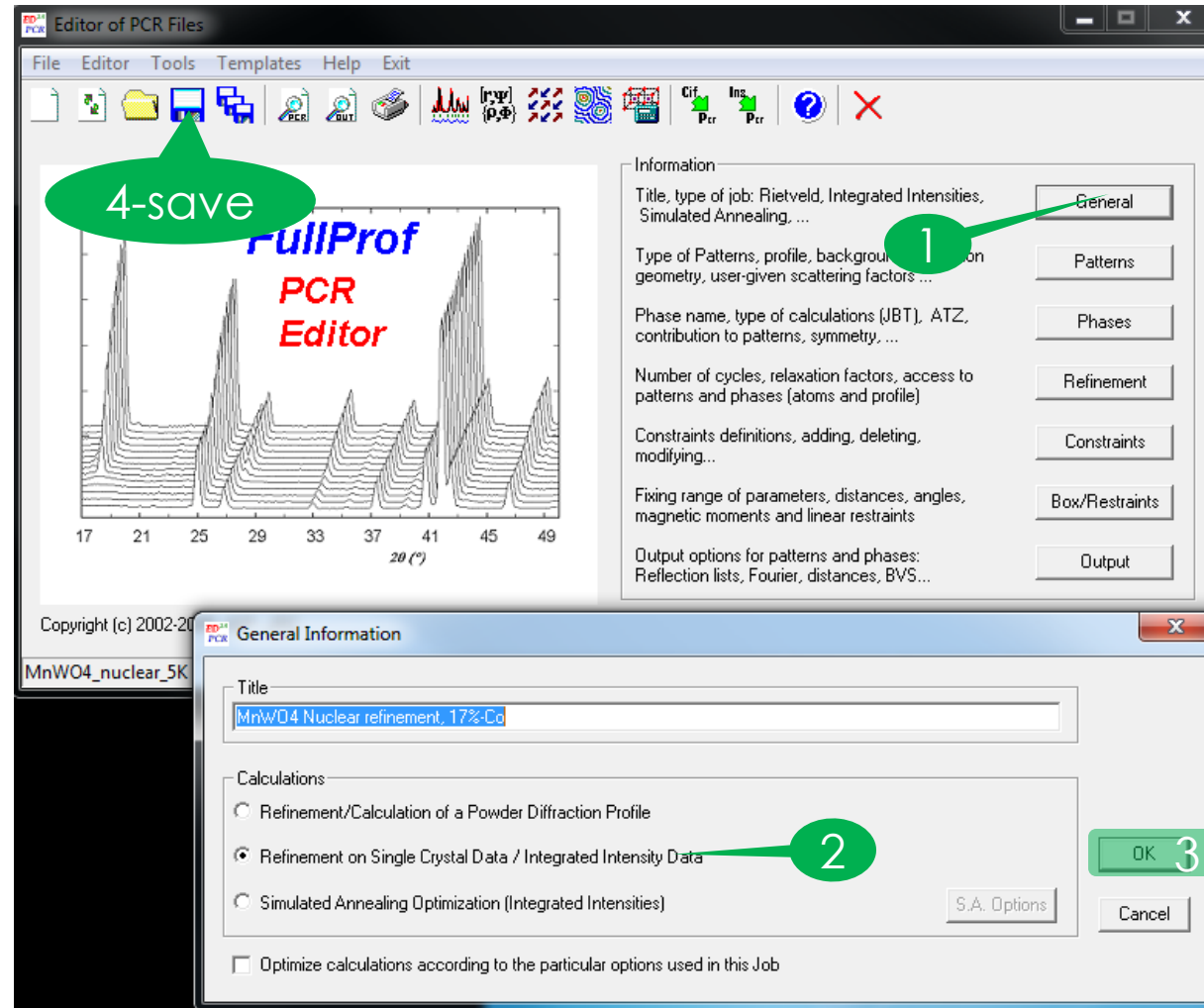
Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Create .pcr file from .cif file



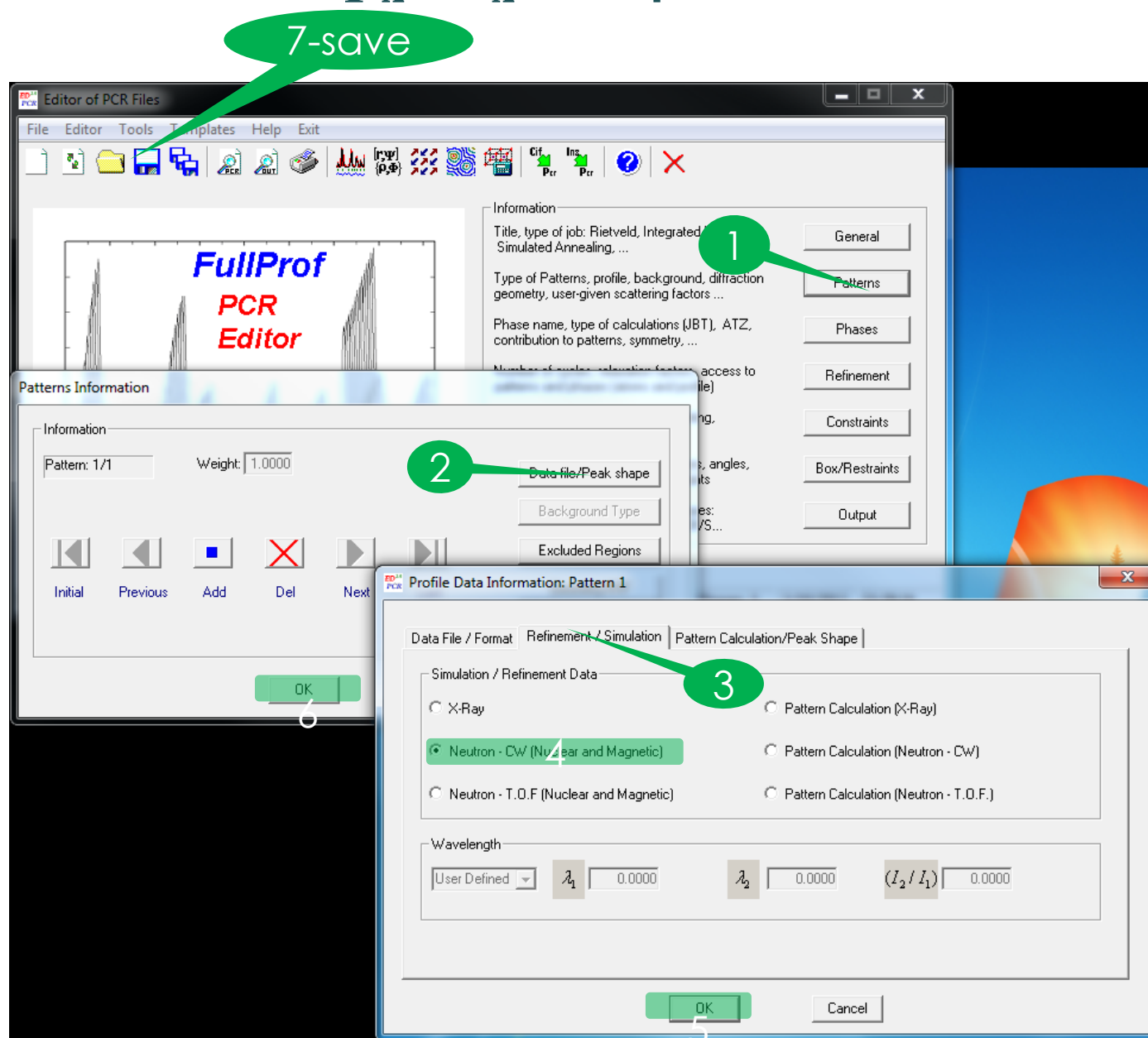
Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

General-TAB



Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Pattern



Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Phase

9-save

FullProf
PCR
Editor

Information

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

Type of Patterns, profile, background geometry, user-given scattering factor, ...

Phase name, type of calculations (JBT, FINE, contribution to patterns, symmetry, ...)

General

Patterns

Phases

Phase Information: Phase 1

General Information on Phases

Name of Phase: MnWO4 Nuclear refinement, 17%-Co

Calculation: Structural Model (Rietveld Method)

Coefficient to calculate the weight percentage of the Phase: 606.91 ☐ Calculated automatically ☒ Provided by user

Contribution to patterns, preferred orientation direction, reflection list, ...

Space Group symbol/number, symmetry operators, basis functions, etc

Initial Previous Add Del Next Last

OK Cancel

Pattern Contribution Information for Phase 1

Pattern 1 | Pattern 2 | Pattern 3 | Pattern 4 | Pattern 5 | Pattern 6 | Pattern 7

☒ Current Phase contributes to the pattern

Type of Pattern

☐ X-Ray ☐ Pattern Calculation (X-Ray)

☒ Neutron (Constant Wavelength) Nuclear and Magnetic ☐ Pattern Calculation (Neutron - Constant Wavelength)

☐ Neutron (T.O.F.) Nuclear and Magnetic ☐ Pattern Calculation (Neutron - T.O.F.)

Peak Shape

Gaussian

Codefil.shp Global.shp

Intensities

Reflection list: Integrated Intensities

☒ Use special control of parameters for peak overlap, rejected reflections for current phase

Brindley coefficient: 1.000

Global weight of the integrated intensity data vs profile data: 1.000

Factor for excluding reflections [< Factor * Sigma(I)]: 2.000

Weights are divide by reduced Chi² of precedent cycle: 0.000

OK Cancel

Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine

8-save

1

2

3

4-all zero

5-all zero

6

9- Go back to the scale factor tab (see flag-3) and be sure it is clicked on for refining. And **save again.**

FullProf
PCR
Editor

Information

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

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

Phase name, type of calculation, contribution to patterns, symmetry

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

General

Patterns

Phases

Refinement

Refinement Information

Cycles of Refinement: 10

Stop Criterion of Coverage

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 6

Refinement weighting model

☒ Least Squares ☐ Maximum Likelihood ☐ Unit Weights

Background

Instrumental

Micro-Absorption

Reduction factor of number of data points: 0

OK Cancel

Integrate Intensities: Phase 1 Pattern 1

Cell Parameters

	a	b	c	alpha	beta	gamma
Coefficients	4.77000	5.72000	4.92000	90.00	90.90	90.00

Refine All

Fix All

Scale Factors

	1	2	3	4	5	6
Coefficients	1.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Extinction Parameters

	1	2	3	4	5	6
Coefficients	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

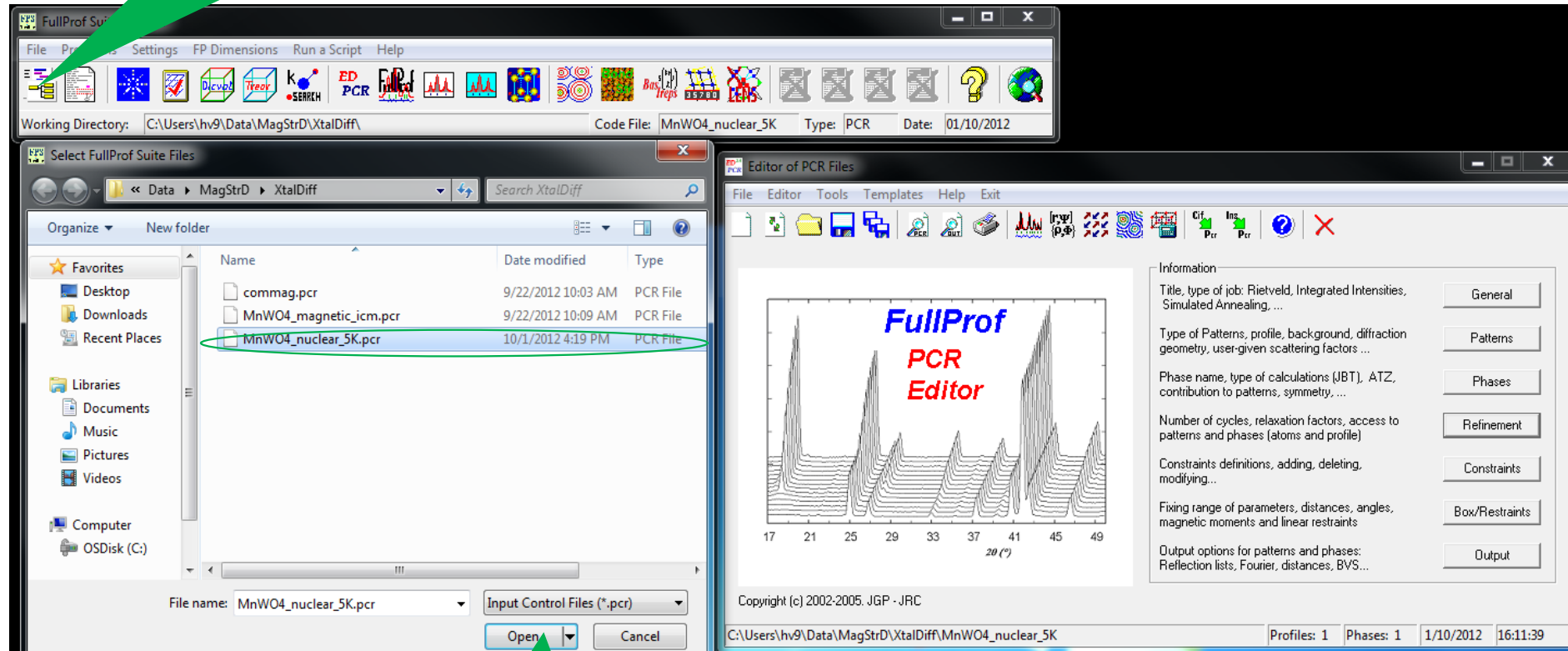
Cancel

OK

Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine

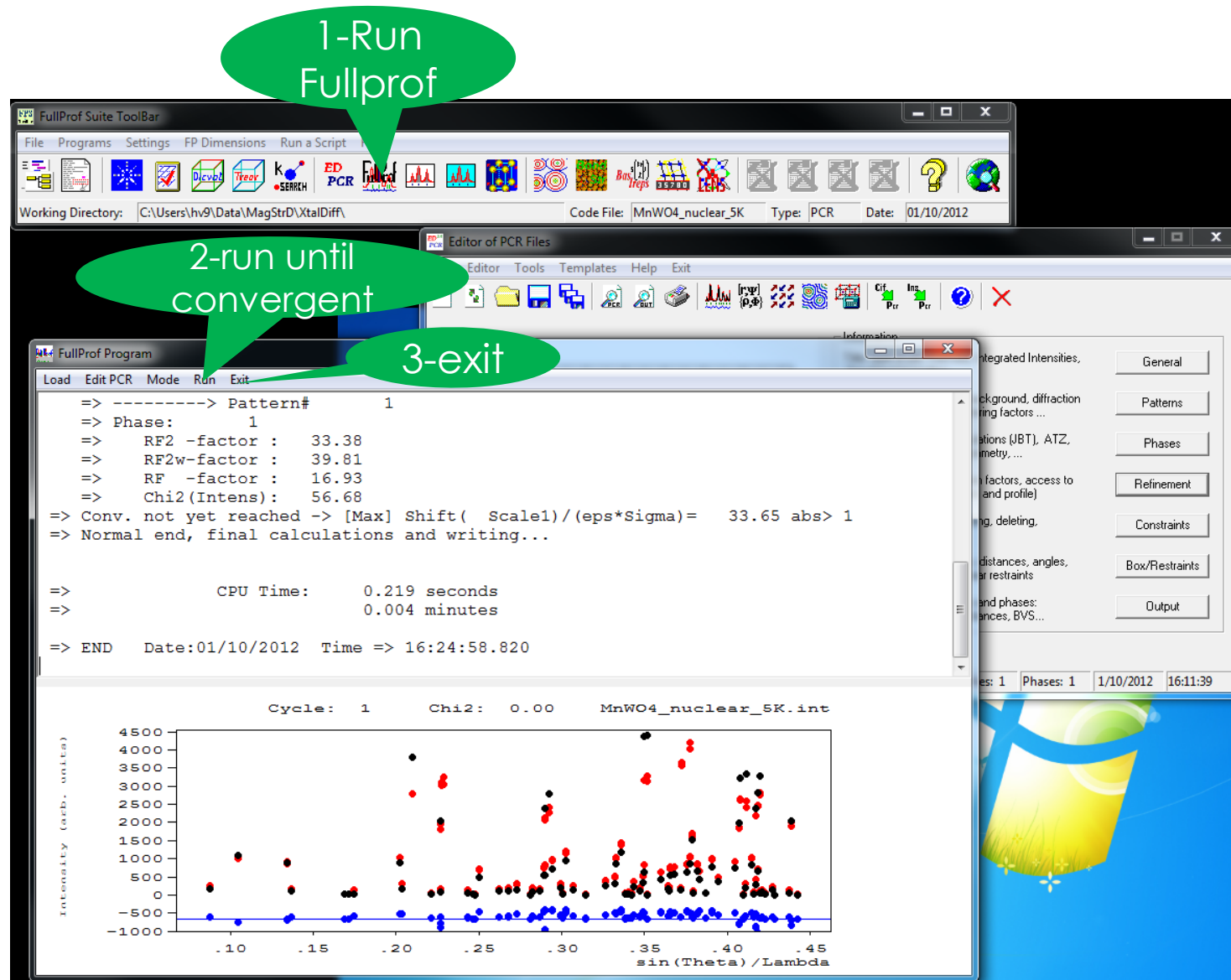
1-select the
created .pcr file



2-open

Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine



Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine

1-Open
EDPCR

8

9-exit

2

3

4

5

6

7

FullProf Suite ToolBar

File Programs Settings FP Dimensions Run a Simulation Help

Working Directory: C:\Users\hv9\Documents\MagStrD\XtalDiff\ Code File: MnWO4_nuclear_SK Type: PCR Date: 01/10/2012

Editor of PCR Files

File Editor Tools Templates Help Exit

Information

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

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

Phase name, type of calculations (Rietveld, profile, contribution to patterns, symmetry, ...)

Number of cycles, relaxation factors, ...

Refinement Information

Cycles of Refinement: 1

Stop Criterion of Convergence

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

Others: None

Relaxation Factors for Shifts

Atomic: 1.00 Anisotropic: 1.00

Reflections ordering

Only at the first cycle Each cycle Bragg R-Factor excluding reflections

Pattern 1 Pattern 2 Pattern 3 Pattern 4 Pattern 5 Pattern 6

Refinement weighting model

Least Squares Maximum Likelihood Unit Weights

Background Instrumental Micro-Absorption

Reduction factor of number of data points: 0

OK Cancel

Atoms Information: Phase 1

List of Atoms

Number of Atoms: 5

Atom #	Label	Ntyp	X	Y	Z	B	Occ	Therm. Fact.
Atom # 1	Mn1	MN	0.50000	0.68510	0.25000	0.63955	0.41600	Isotropic
Atom # 2	Co		0.50000	0.68510	0.25000	0.63955	0.08400	Isotropic
Atom # 3	W1	W	0.00000	0.18020	0.25000	0.79746	0.50000	Isotropic
Atom # 4	O1	O	0.21080	0.10374	0.94040	0.65534	1.00000	Isotropic

Anisotropic Thermal Factors / Form Factors

#	B11/F1	B22/F2	B33/F3	B12/F4	B13/F5	B23/F6	F7
#							
#							
#							

Special Form Factor

#	SASH-Type	Matrix	i=1	i=2	i=3	N. Coeff.	Indices	#1	#2	#3	#4	#5	#6
#	Spherical												
#	Spherical												
#	Spherical												

Refine Positions

Refine B_iso

Refine B_aniso

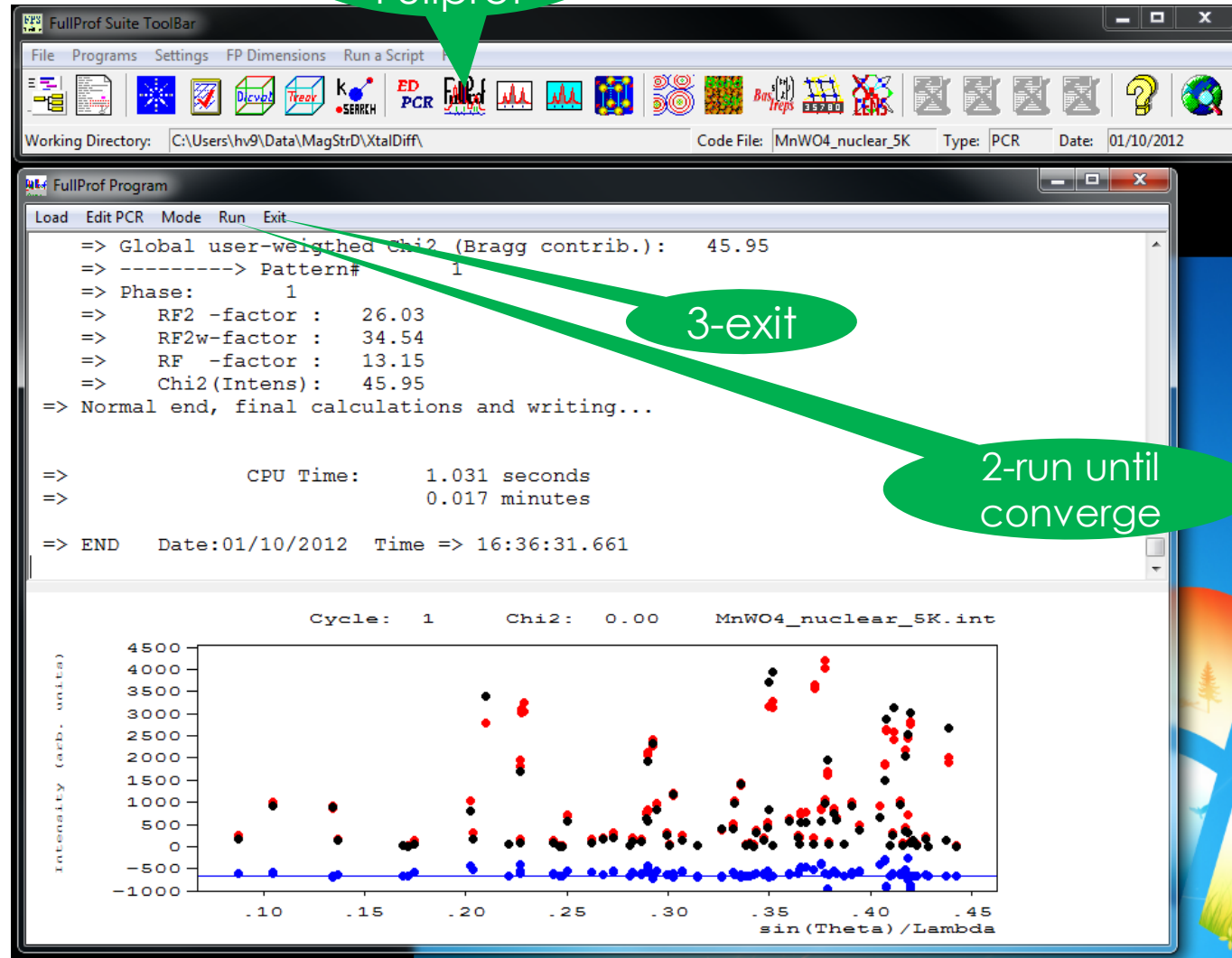
Fix All

Cancel

OK

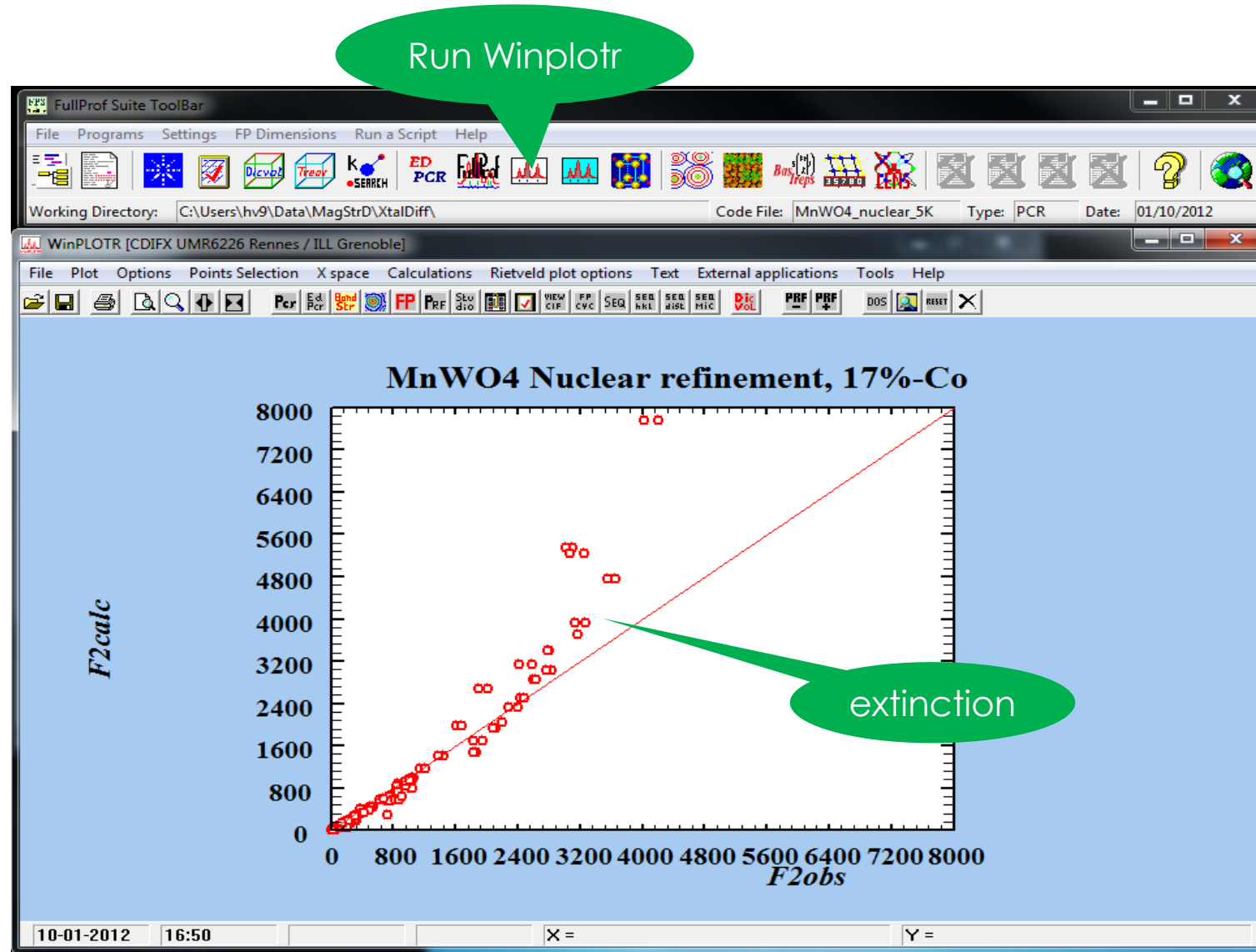
Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine



Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine



Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine

1

2

3

4-extinction

5

6-save

7-exit

PCR Editor

Refinement Information

Cycles of Refinement: 1

Stop Criterion of Convergence
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 6 | Pattern 7

Phase 1 | Phase 2 | Phase 3 | Phase 4 | Phase 5 | Phase 6 | Phase 7

Refinement weighting model
☒ Least Squares ☐ Maximum Likelihood ☐ Unit Weights
Background Instrumental Micro-Structure

Atoms Prop. Vectors
Patterns Intensities Micro-Structure

Integrate Intensities: Phase 1 Pattern 1

Cell Parameters						
	a	b	c	alpha	beta	gamma
Coefficients	4.77000	5.72000	4.92000	90.00	90.90	90.00

Refine All Fix All

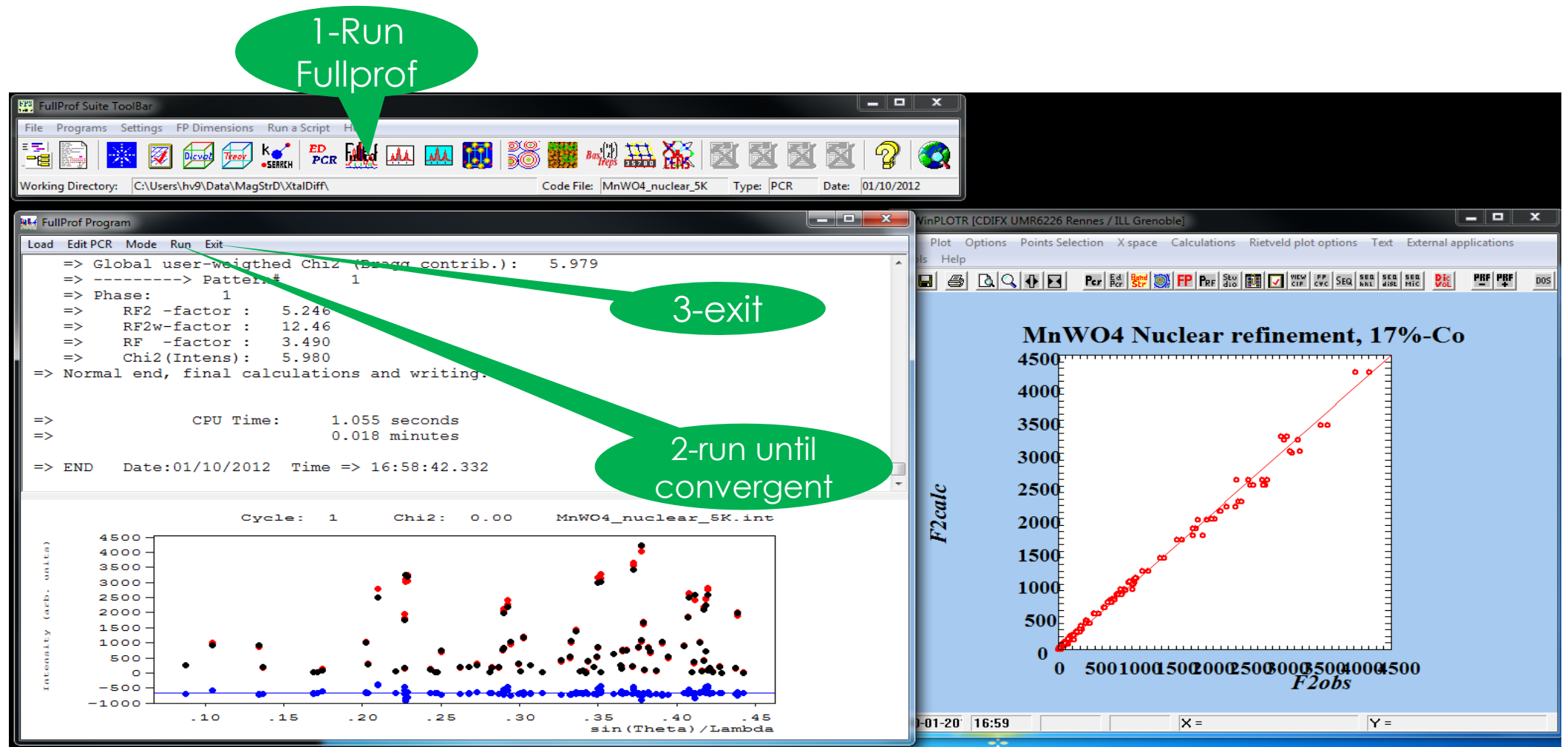
Scale Factors						
	1	2	3	4	5	6
Coefficients	235.90	0.0000	0.0000	0.0000	0.0000	0.0000

Extinction Parameters						
	1	2	3	4	5	6
Coefficients	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Cancel OK

Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine



Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine

1-Open .pcr file in text editor

4-Save & exit

```
FullProf Suite ToolBar
File Programs Settings Run a Script Help
Working Directory: C:\Users\hv9\Data\MagStrD\XtalDiff\ Code File: MnWO4_nuclear_5K Type: PCR Date: 01/10/2012

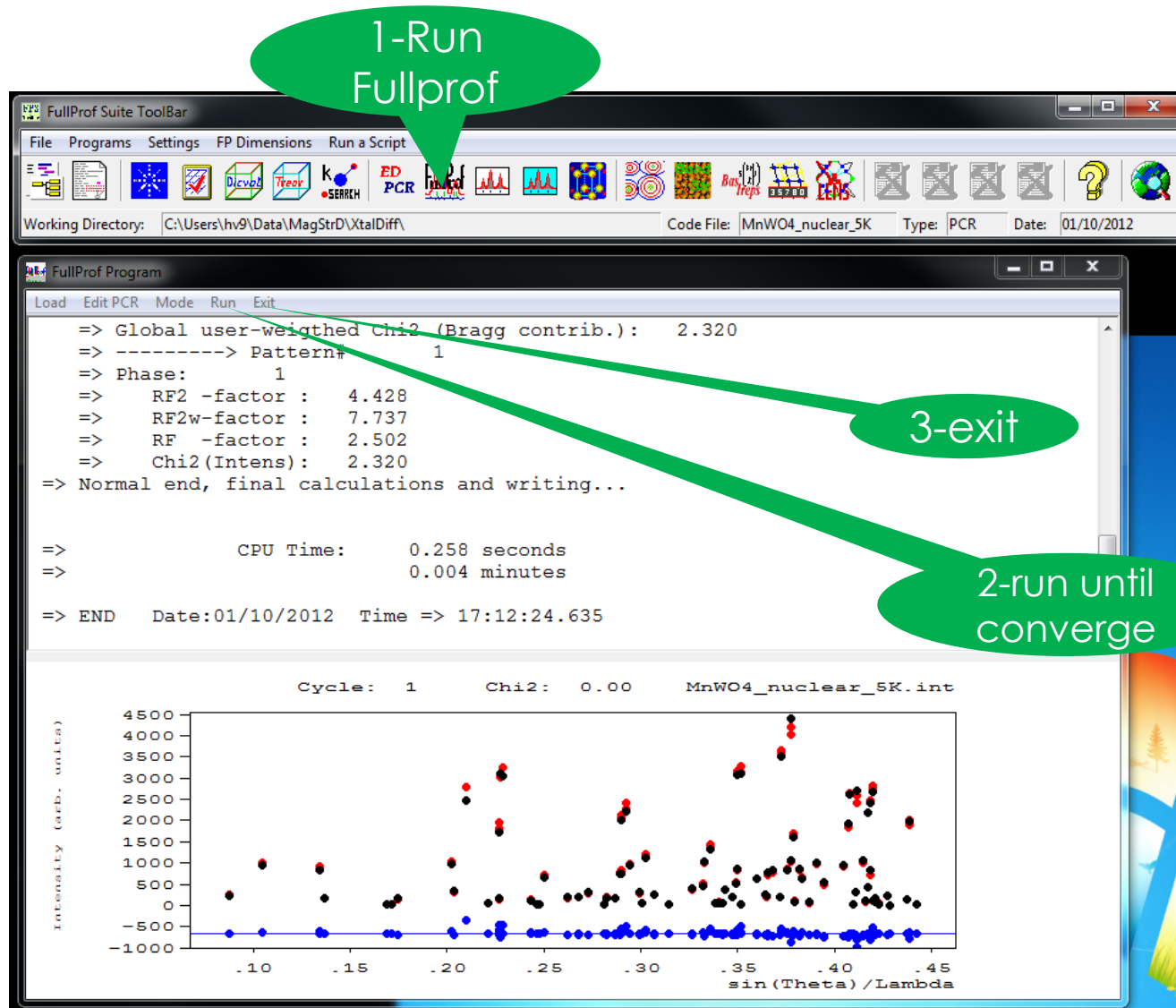
!Jvi Jdi Hel Sol Mom Ter Brind RMua RMub RMuc Jtyp Nsp_Ref Ph_Shift N_Domains
! 0 0 0 0 0 0 1.0000 1.0000 2.0000 0.0000 1 0 0 0
!
!P 1 2/c 1 <--Space group symbol
!Atom Typ X Y Z Biso Occ In Fin N_t Spc /Codes
Mn1 MN 0.50000 0.68446 0.25000 0.74033 0.41600 0 0 0 0
0.00 31.00 0.00 21.00 301.00
Co Co 0.50000 0.68446 0.25000 0.74033 0.08400 0 0 0 0
0.00 31.00 0.00 21.00 -301.00
W1 W 0.00000 0.18018 0.25000 1.12549 0.50000 0 0 0
0.00 61.00 0.00 51.00 0.00
O1 O 0.21237 0.10301 0.94048 1.37386 1.00000 0 0 0
81.00 91.00 101.00 71.00 0.00
O2 O 0.25374 0.37545 0.39459 1.34545 1.00000 0 0 0
111.00 121.00 131.00 41.00 0.00
!-----> Scale, Extinction and Cell Parameters for Pattern # 1
! Scale Factors
! Sc1 Sc2 Sc3 Sc4 Sc5 Sc6
! 543.6 0.000 0.000 0.000 0.000 0.000
! 11.00 0.00 0.00 0.00 0.00 0.00
! Extinction Parameters
! Ext1 Ext2 Ext3 Ext4 Ext5 Ext6 Ext7 Ext-Model
! 97.79 0.000 0.000 0.000 0.000 0.000 0.000 1
! 141.00 0.00 0.00 0.00 0.00 0.00 0.00
! a b c alpha beta gamma #Cell Info
! 4.770000 5.720000 4.920000 90.000000 90.900024 90.000000
! 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
! x-Lambda/2 + Not yet used parameters
! 0.000000 0.000000 0.000000 0.000000 0.000000
! 311.00 0.00 0.00 0.00 0.00
! 2Th1/TOF1 2Th2/TOF2 Pattern # 1
! 1.536 0.000 1
```

3-occup. refine with constrain

3-lambda/2 refine

Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine

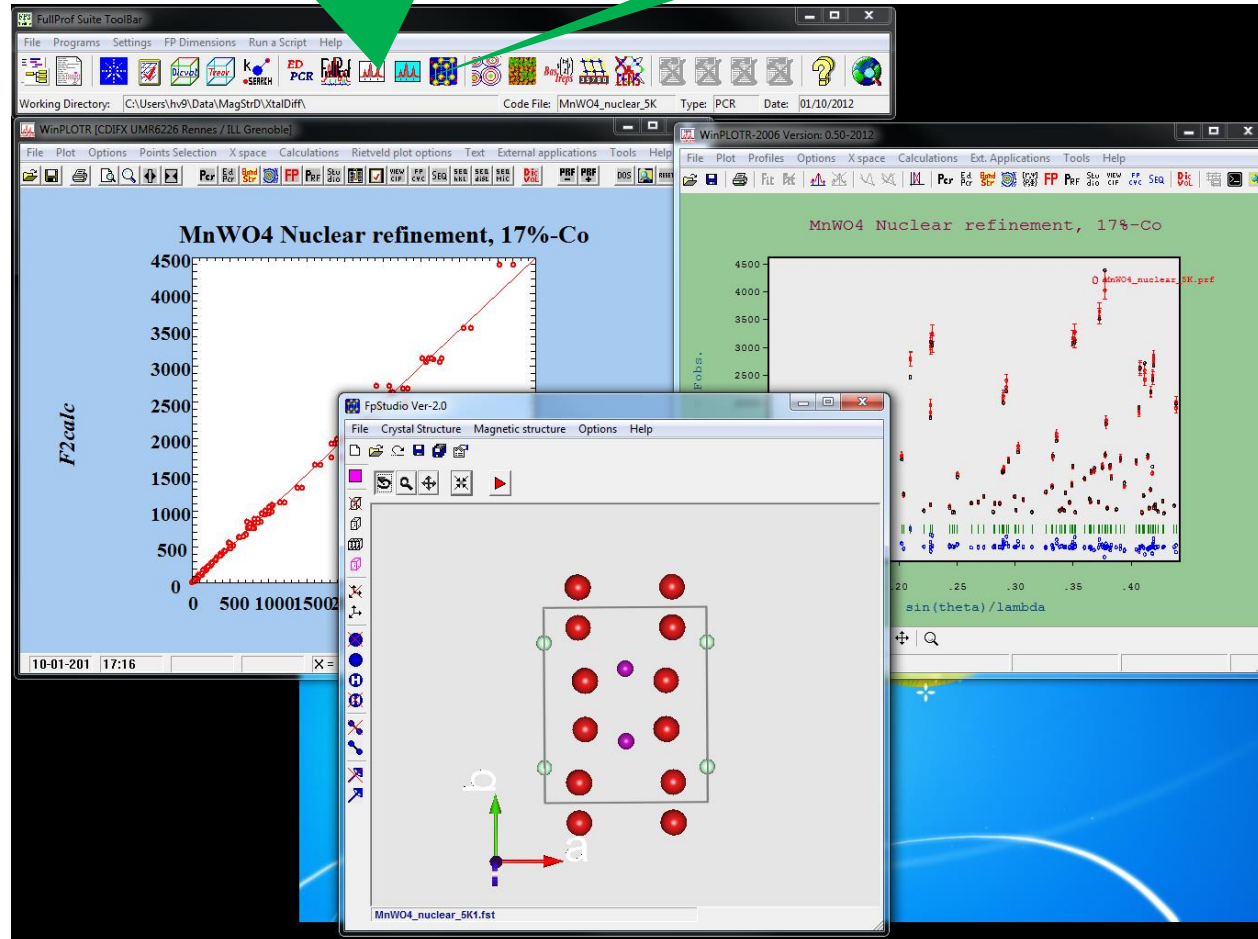


Nuclear structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$ refined by FullProf

Refine

1-WinPlotr/2006

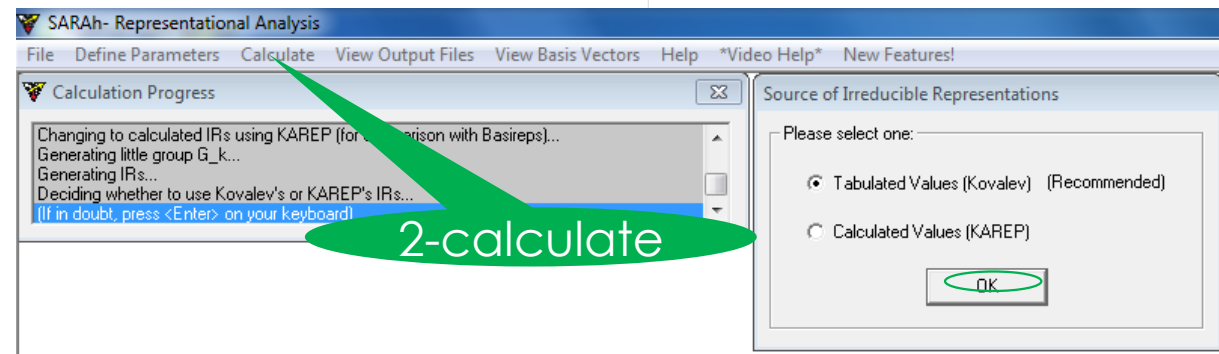
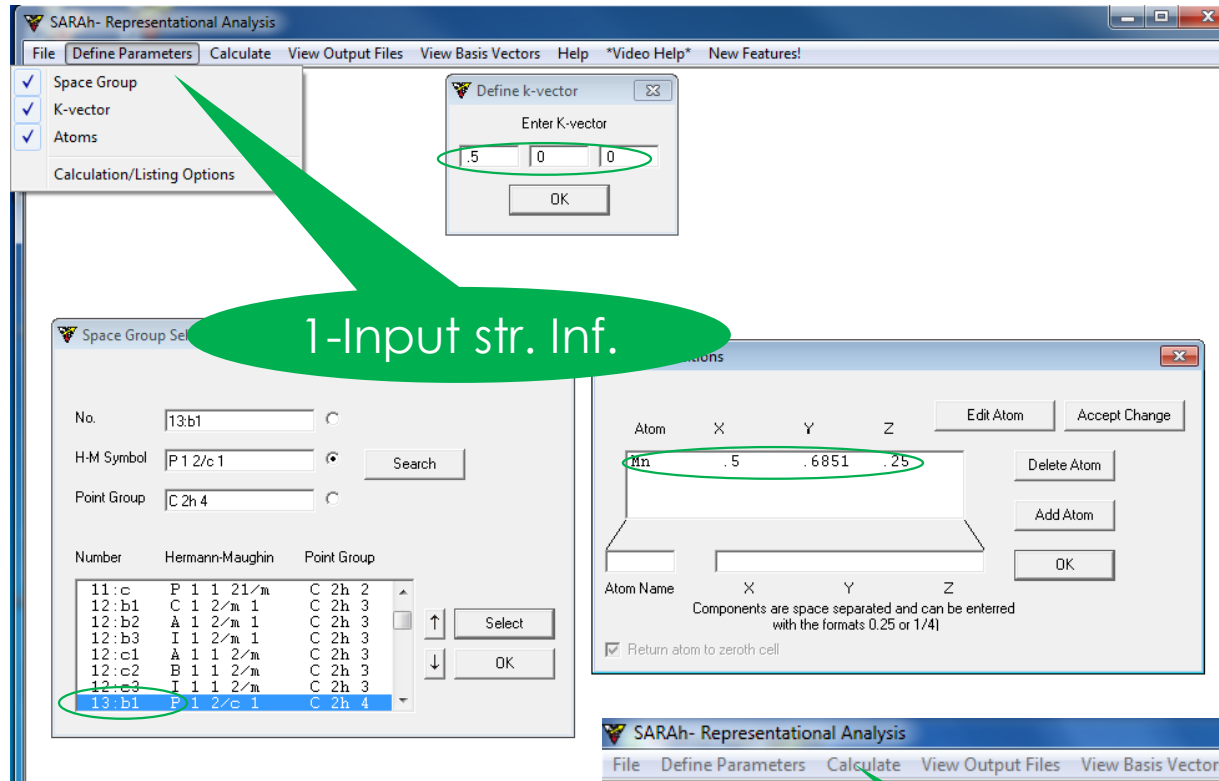
2-FPStudio



can be refined, such as “anisotropic extinction model” and “anisotropic displacement s”, which depends on your physics and the number of reflections. Here it is good enough for the magnetic structure

Commensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Create the magnetic phase using SARAh-Representation Analysis



Commensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Create the magnetic phase using SARAh-Refine

1. SARAh-Refine File menu: Load SARAh MAT file

2. File Explorer: sarah13

3. Open SARAh MAT File dialog: sarah13, MAT (*.MAT), Open

4. SARAh-Refine dialog: Yes

5. SARAh-Refine Generate/Edit FullProf 2K *.PCR File dialog: Single Crystal, Generate model PCR phase, Generate

6. SARAh-Refine dialog: Yes

7. SARAh-Refine dialog: Yes

8. SARAh-Refine dialog: Yes

Generate magnetic phase in sarah13.pcr
We will create the magnetic phase .pcr file from nuclear structure .pcr by inserting the sarah13.pcr

Commensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Create the magnetic .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 Nvk Npr More
1 0 0 0.0 0.0 1.0 1 4 -2 0 0 0.00 1 0 0
P -1
! Nsym Cen Laue Ireps N_Bas
2 1 1 -1 2
! Real(0)-Imaginary(1) indicator for Ci
0 0
SYMM X, Y, Z
BASR 2 0 0 0 0 0 2
BASR 0 0 0 0 0 0 0
SYMM -X+1, -Y+1, -Z+1
BASR 2 0 0 0 0 0 2
BASR 0 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
MN1 MN3 1 0 0.50000 0.68510 0.25000 0.30000 1.00000 0.000 0.000 0.000
0.000 0.000 0.000 0.000 0.000 0.000 0.000
0.000 0.000 0.000 0.000 0.000 0.000 0.000
! Scale Factors
Sc1 Sc2 Sc3 Sc4 Sc5 Sc6
100.0 0.000 0.000 0.000 0.000 0.000
0.00 0.00 0.00 0.00 0.00 0.00
! Extinction Parameters
Ext1 Ext2 Ext3 Ext4 Ext5 Ext6 Ext7
0.000 0.000 0.000 0.000 0.000 0.000 0.000
0.00 0.00 0.00 0.00 0.00 0.00 0.00
a b c alpha beta gamma
273
1 1 1 90 90 90
0.00000 0.00000 0.00000 0.00000 0.00000 0.00000
! Not yet used parameters
0.00000 0.00000 0.00000 0.00000 0.00000
0.00 0.00 0.00 0.00 0.00
! Propagation vectors:
0.00000 0.00000 0.00000 0.00000
0.00000 0.00000 0.00000 0.00000
Propagation Vector 1
```

```
!NCY Eps R_at R_an R_pr R_gl Thmin Step Thmax PSD Sent0
1 0.10 1.00 1.00 1.00 1.00 1.5360 0.000000 0.0000 0.000 0.000

16 !Number of refined parameters

Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 4.43
MnWO4 Nuclear refinement, 17%-Co
!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 4 0 0 0 606.914 0 0 1
!Jvi Jdi Hel Sol Mom Ter Brind RMua RMub RMuc Jtyp Nsp_Ref Ph_Shift N_Domains
0 0 0 0 0 0 1.0000 1.0000 2.0000 0.0000 1 0 0 0
P 1 2/c 1
!Atom Typ X Y Z Biso Occ In Fin N_t Spc /Codes
Mn1 MN 0.50000 0.68491 0.25000 0.63149 0.41661 0 0 0 0
0.00 0.00 0.00 0.00 0.00 0.00
Co Co 0.50000 0.68491 0.25000 0.63149 0.08339 0 0 0 0
0.00 0.00 0.00 0.00 0.00 0.00
W1 W 0.00000 0.18007 0.25000 0.76064 0.50000 0 0 0 0
0.00 0.00 0.00 0.00 0.00 0.00
O1 O 0.21097 0.10361 0.94038 0.66934 1.00000 0 0 0 0
81.00 91.00 101.00 71.00 0.00
O2 O 0.25098 0.37544 0.39396 0.72572 1.00000 0 0 0 0
111.00 121.00 131.00 41.00 0.00
!-----> Scale, Extinction and Cell Parameters for Pattern # 1
! Scale Factors
Sc1 Sc2 Sc3 Sc4 Sc5 Sc6
455.1 0.000 0.000 0.000 0.000 0.000
11.00 0.00 0.00 0.00 0.00 0.00
! Extinction Parameters
Ext1 Ext2 Ext3 Ext4 Ext5 Ext6 Ext7 Ext-Model
78.96 0.000 0.000 0.000 0.000 0.000 0.000 1
141.00 0.00 0.00 0.00 0.00 0.00 0.00
a b c alpha beta gamma #Cell Info
4.770000 5.720000 4.920000 90.000000 90.900024 90.000000
0.00000 0.00000 0.00000 0.00000 0.00000 0.00000
! x-Lambda/2 + Not yet used parameters
0.02137 0.00000 0.00000 0.00000 0.00000
151.00 0.00 0.00 0.00 0.00
! 2Th1/TOF1 2Th2/TOF2 Pattern # 1
1.536 0.000 1
```

sarah13.pcr

insert/replace

MnWO4_nuclear_5K.pcr

Save as

commag.pcr

Commag.pcr

```

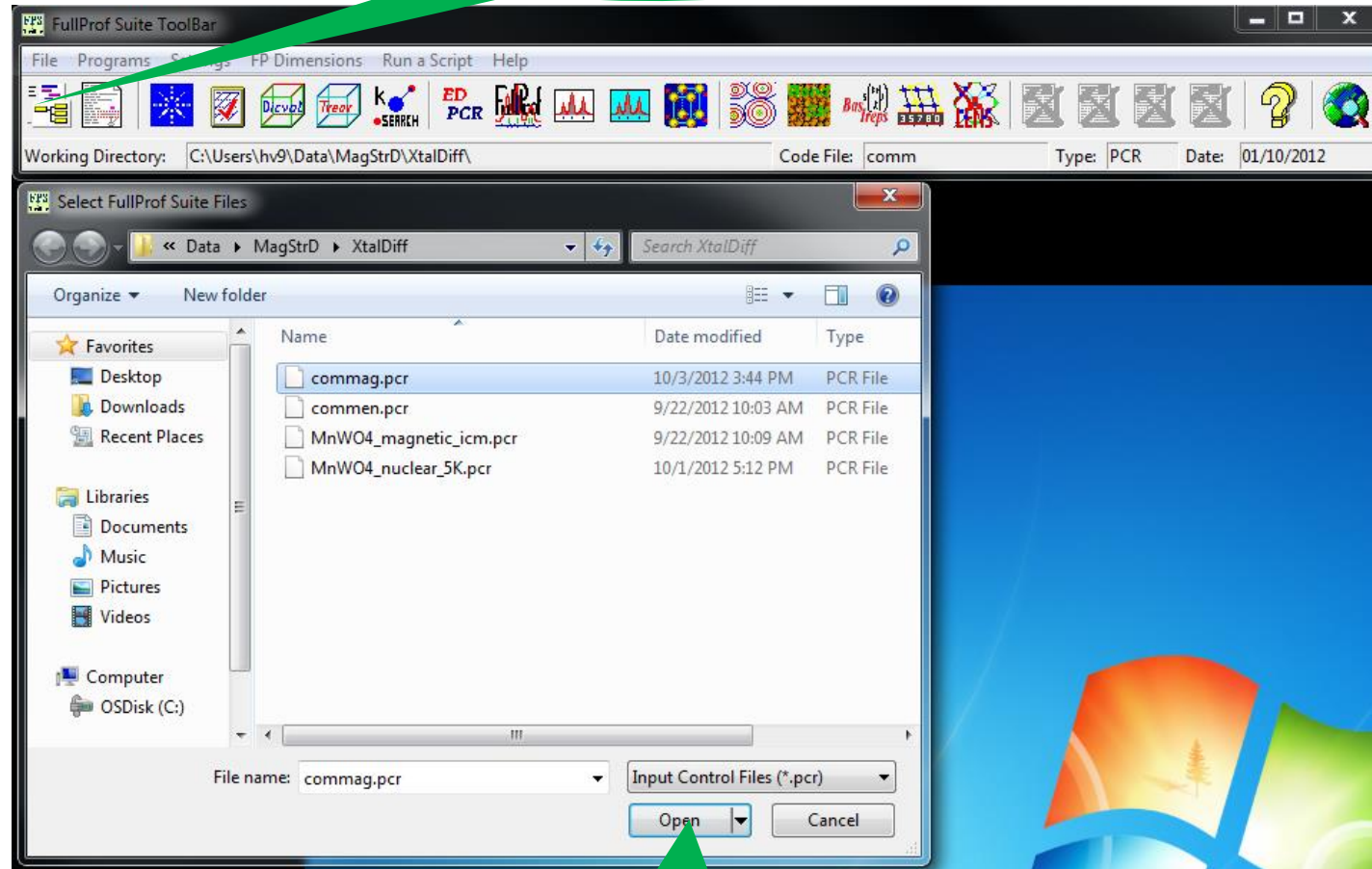
Saved...
COMM MnWO4 commensurate magnetic structure refinement, 17%-Co
! Current global Chi2 (Bragg contrib.) = 2.320
! Files => DAT-file: MnWO4_nuclear_5K.int, PCR-file: MnWO4_nuclear_5K
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
1 0 1 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 1
!
!Pr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
0 0 1 0 1 0 4 0 0 3 0 0 0 0 0 0 0 0 0
!
!NCY Eps R_at R_an R_pr R_gl Thmin Step Thmax PSD Sent0
1 0.10 1.00 1.00 1.00 1.00 1.5360 0.000000 0.0000 0.000 0.000
!
16 !Number of refined parameters
-----
! Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 4.43
-----
MnWO4 Nuclear refinement, 17%-Co
!Nat Dis Mom Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth ATZ Nvk Npr More
1 0 0 0.0 0.0 1.0 1 4 -2 0 0 0.00 1 0 0
!
P -1 <--Space group symbol
! Nsym Cen Laue Ireps N_Bas
2 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, -Y+1, -Z+1
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
MN1 CN3 C5 C6 C7 C8 C9 MagPh
0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000
0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000
0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000 0.0000
!-----> Scale, Extinction and Cell Parameters for Pattern # 1
! Scale Factors
! Sc1 Sc2 Sc3 Sc4 Sc5 Sc6
455.1 0.000 0.000 0.000 0.000 0.000
0 0.00 0.00 0.00 0.00 0.00
! Extinction Parameters
! Ext1 Ext2 Ext3 Ext4 Ext5 Ext6 Ext7 Ext-Mode
78.96 0.000 0.000 0.000 0.000 0.000 0.000 1
0 0.00 0.00 0.00 0.00 0.00 0.00
! a b c alpha beta gamma #Cell Info
4.770000 5.720000 4.920000 90.000000 90.900024 90.000000
0.00000 0.00000 0.00000 0.00000 0.00000 0.00000
! x-Lambda/2 + Not yet used parameters
0.02137 0.00000 0.00000 0.00000 0.00000 0.00000
0 0.00 0.00 0.00 0.00 0.00
! Propagation vectors:
5000000 0.000000 0.000000 Propagation Vector 1
0.000000 0.000000 0.000000
! 2Th1/TOF1 2Th2/TOF2 Pattern # 1
1.536 0.000 1

```

Commensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Commag.pcr

1-select the
created .pcr file



2-open

Commensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Commag.pcr

The image shows a screenshot of the FullProf Suite software interface, specifically the 'Editor of PCR Files' window. The interface includes a menu bar (File, Programs, Settings, FP Dimensions, Run a Script, Help), a toolbar with various icons, and a main workspace. A green callout '1-open .pcr file' points to the 'Open' icon in the toolbar. Another green callout '2' points to the 'Phases' button in the right-hand panel. A third green callout '3' points to the 'Contribution to Patterns' button in the 'Phase Information: Phase 1' dialog box. A fourth green callout '4-click on' points to the 'Integrated Intensities' reflection list in the 'Pattern Contribution Information for Phase 1' dialog box. A fifth green callout '5-2sigma' points to the 'Factor for excluding reflections' field in the same dialog box. A sixth green callout '6' points to the 'OK' button at the bottom of the 'Pattern Contribution Information for Phase 1' dialog box. The 'Phase Information: Phase 1' dialog box also contains fields for 'Name of Phase' (MnWO4 Nuclear refinement, 17%Co), 'Calculation' (Magnetic Phase [Rietveld Method]), and 'Coefficient to calculate the weight percentage of the Phase' (0.00). The 'Pattern Contribution Information for Phase 1' dialog box includes options for 'Current Phase contributes to the pattern', 'Type of Pattern' (X-Ray, Neutron), 'Peak Shape' (Gaussian), and 'Intensities' (Reflection list: Integrated Intensities).

1-open .pcr file

8-save

2

3

4-click on

5-2sigma

6

Commensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Commag.pcr

The screenshot shows the FullProf Suite software interface with several windows and annotations:

- FullProf Suite ToolBar**: The top window showing the main menu and toolbar. The working directory is `C:\Users\hv9\Data\MagStrD\XtalDiff\` and the code file is `commag`.
- Editor of PCR Files**: The main editor window. It contains a plot of the diffraction pattern and a list of phases. A green callout labeled "6-save" points to the save icon in the toolbar. A green callout labeled "7-close" points to the close icon in the toolbar. A green callout labeled "1" points to the "Phases" button in the "Information" panel.
- Refinement Information**: A window showing refinement parameters. A green callout labeled "2" points to the "Phase 1" tab. The "Refinement weighting model" is set to "Least Squares". The "Reduction factor of number of data points" is set to 0.1.
- Atoms Information: Phase 1**: A window showing the list of atoms and their refinement parameters. A green callout labeled "3-click on and initial values" points to the "List of Atoms" table. The table has columns for Label, Ntyp, Mag. Rot, Prog. Vec., X, Y, Z, B, and Occ. The first row shows Atom # 1, Label MN1, Ntyp MMN3, Mag. Rot 1, Prog. Vec. 0, X 0.50000, Y 0.68510, Z 0.25000, B 0.30000, and Occ 1.00000. A green callout labeled "4" points to the "OK" button.

Commensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Commag.pcr



Saved...

Single crystal data of MnWO4-17pCo (hb3a)

! Data format
(4i5,2f8.2,i4,3f8.2)

! Wavelength Itypdata Ipow(n)
1.53600 0 0

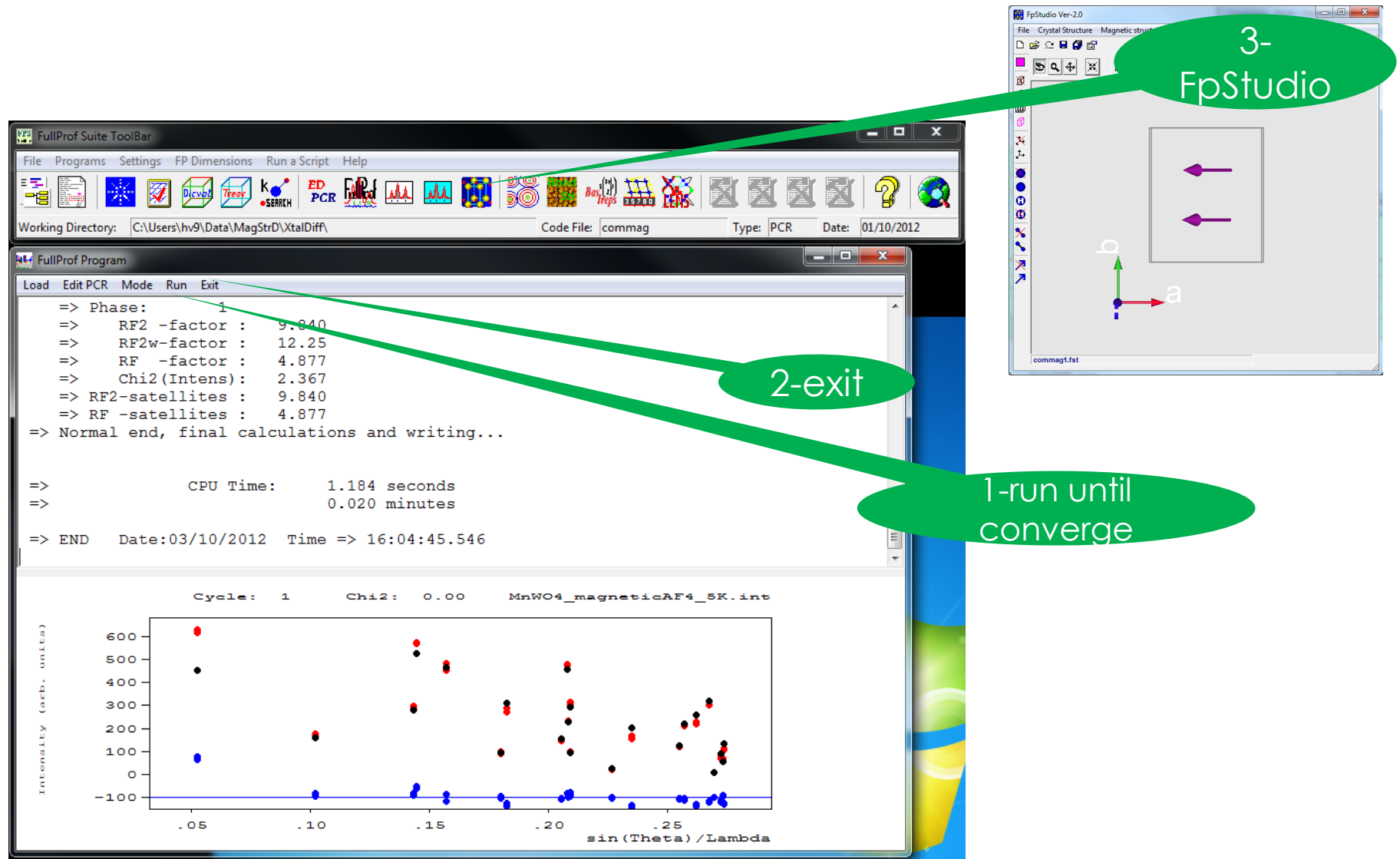
! The number of propagation vectors

1

nv	k-vector	nv	F ²	sigma(F ²)	code
1	0.5 0 0	1	220.77	24.02	1
1	-3 -0 -0	1	6.77	28.95	1
1	-2 -0 -1	1	483.14	26.21	1
1	-2 -0 -0	1	4.23	3.34	1
1	-2 -0 1	1	0.00	0.00	1
1	-2 1 -2	1	67.54	8.40	1
1	-2 1 -1	1	476.88	23.83	1
1	-2 1 -0	1	88.78	8.86	1
1	-2 1 1	1	148.23	10.55	1
1	-2 1 2	1	8.79	1.74	1
1	-2 2 -1	1	211.75	31.62	1
1	-2 2 -0	1	167.75	12.99	1
1	-2 2 1	1	120.06	13.22	1
1	-1 -0 -0	1	630.71	34.79	1
1	-1 -0 1	1	0.00	0.00	1
1	-1 -0 2	1	100.72	9.14	1
1	-1 1 -1	1	575.06	41.47	1
1	-1 1 -0	1	177.01	11.62	1
1	-1 1 1	1	296.13	17.86	1
1	-1 1 2	1	21.93	8.54	1
1	-1 2 -2	1	110.18	17.98	1
1	-1 2 -1	1	315.65	32.47	1
1	-1 2 -0	1	272.73	16.39	1
1	-1 2 1	1	229.09	25.01	1
1	-1 2 2	1	79.71	12.33	1
1	-1 3 -0	1	302.73	21.20	1
1	-0 0 -2	1	99.62	10.96	1
1	-0 0 -1	1	0.00	0.00	1
1	-0 0 -0	1	615.47	37.11	1
1	-0 1 -2	1	26.86	7.11	1
1	-0 1 -1	1	288.24	19.69	1
1	-0 1 -0	1	163.86	12.48	1
1	-0 1 1	1	568.64	40.37	1
1	-0 2 -2	1	70.87	16.70	1
1	-0 2 -1	1	234.70	27.29	1
1	-0 2 -0	1	289.51	15.90	1
1	-0 2 1	1	306.09	32.96	1
1	-0 2 2	1	105.75	18.82	1
1	-0 3 -0	1	302.12	18.40	1
1	1 0 -2	1	7.58	19.89	1
1	1 0 -1	1	3.09	3.09	1
1	1 0 -0	1	452.89	28.44	1
1	1 0 1	1	0.00	0.00	1
1	1 1 -2	1	2.77	7.06	1
1	1 1 -1	1	151.09	12.34	1
1	1 1 -0	1	97.32	10.11	1
1	1 1 1	1	474.56	23.26	1
1	1 1 2	1	61.17	8.99	1
1	1 2 -1	1	123.40	14.72	1
1	1 2 -0	1	156.49	13.42	1
1	1 2 1	1	218.98	29.27	1
2	0 -0	1	229.92	26.76	1

Commensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Commag.pcr



Incommensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

Commag.pcr

```

COMM MnWO4 commensurate magnetic structure refinement, 17%-Co
| Current global Chi2 (Bragg contrib.) = 2.364
| Files => DAT-file: MnWO4_magneticAF4_5K.int, PCR-file: commagg
| Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
| 1 1 0 0 0 0 0 0 0 0 0 0 0 0 0 1 0 0 0 1
|
| Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
| 0 0 1 0 1 0 1 0 4 0 0 0 3 0 0 0 0 0 0 0 0 0 0 0
|
| NCY Eps R_at R_an R_pr R_gl Thmin Step Thmax PSD Sent0
| 1 0.10 1.00 1.00 1.00 1.00 1.5360 0.000000 0.0000 0.000 0.000
|
| 2 |Number of refined parameters
|-----|
| Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 9.81
|-----|
| MnWO4 Nuclear refinement, 17%-Co
|
| Nat Dis Mom Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth ATZ Nvk Npr More
| 1 0 0 0.0 0.0 1.0 1 4 -2 0 0 0.000 1 0 1
|
| Jvi Jdi Hel Sol Mom Ter Brind RMua RMub RMuc Jtype Nsp_Ref Ph_Shift N_Domains
| 0 0 0 0 0 0 1.0000 1.0000 2.0000 0.0000 1 0 0 0
|
| P -1 <--Space group symbol for hkl generation
| Nsym Cen Laue Ireds N_Bas
| 2 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, -Y+1, -Z+1
| BASR 2 0 0 0 0 2
| BASI 0 0 0 0 0 0
|
| Atom Type Mag Vek X Y Z Biso Occ C1 C2 C3
| MN1 C4 C5 C6 C7 C8 C9 MagPh
| 1.000000 -0.996 1.360 0.000
| 0.000 0.000 0.000 0.000 0.000 0.000 11.00 21.00 0.00
| 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00
|
| -----> Scale, Extinction and Cell Parameters for Pattern # 1
| Scale Factors
| Sc1 Sc2 Sc3 Sc4 Sc5 Sc6
| 455.1 0.000 0.000 0.000 0.000 0.000 0.000
| 0.00 0.00 0.00 0.00 0.00 0.00
|
| Extinction Parameters
| Ext1 Ext2 Ext3 Ext4 Ext5 Ext6 Ext7 Ext-Model
| 78.96 0.000 0.000 0.000 0.000 0.000 0.000 1
| 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00
|
| a b c alpha beta gamma #Cell Info
| 4.770000 5.720000 4.920000 90.000000 90.900024 90.000000
| 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000
|
| x-Lambda/2 + Not yet used parameters
| 0.02137 0.00000 0.00000 0.00000 0.00000 0.00000
| 0.00 0.00 0.00 0.00 0.00 0.00
|
| Propagation vectors:
| 0.5000000 0.0000000 0.0000000 Propagation Vector 1
| 0.000000 0.000000 0.000000
| 2Th1/TOF1 2Th2/TOF2 Pattern # 1
| 1.536 0.000 1

```

sarah13.pcr

Generated with $k = (0.217 \ 0 \ -0.46)$

```

=====
| 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 Nvk Npr More
| 1 0 0 0.0 0.0 1.0 1 4 -2 0 0 0.00 -1 0 0

P -1
| Nsym Cen Laua Ireps N_Bas <--Space group symbol
| 2 1 1 -1 3
| Real(0)-Imaginary(1) indicator for Ci
| 0 0 0

|
| SYMM X, Y, Z
| BASR 1 0 0 0 0 1 0 0 0 1
| BASI 0 0 0 0 0 0 0 0 0 0
| SYMM X, -Y+1, Z+1/2
| BASR .125 0 0 0 - .125 0 0 0 .125
| BASI .992 0 0 0 - .992 0 0 0 .992

|
| Atom Typ Mag Vek X Y Z Biso Occ C1 C2 C3
| C4 C5 C6 C7 C8 C9 MagPh
| MN1 MNH3 1 0 50000 .68510 25000 30000 1.00000 0.000 0.000 0.000
| 0.000 0.000 0.000 0.000 0.000 0.000 .00000 0.000
| 0.00 0.00 0.00 0.00 0.00 0.00 0.00

| Scale Factors
| Sc1 Sc2 Sc3 Sc4 Sc5 Sc6
| 100.0 0.000 0.000 0.000 0.000 0.000
| .00 0.00 0.00 0.00 0.00 0.00

| Extinction Parameters
| Ext1 Ext2 Ext3 Ext4 Ext5 Ext6 Ext7
| 0.000 0.000 0.000 0.000 0.000 0.000 0.000
| 0.00 0.00 0.00 0.00 0.00 0.00 0.00

| a b c alpha beta gamma

273
1 1 1 90 90 90
0.00000 0.00000 0.00000 0.00000 0.00000 0.00000
| Not yet used parameters
0.00000 0.00000 0.00000 0.00000 0.00000
0.00 0.00 0.00 0.00 0.00

| Propagation vectors:
| .2170000 0.5000000 -.4600000 Propagation Vector 1
| 0.000000 0.000000 0.000000

```

Save as 

incommag.pcr

Incommensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

incommag.pcr

3-Save & exit

```
COMM MnWO4 commensurate magnetic structure refinement, 17%-Co
Current global Chi2 (Bragg contrib.) = 2.364
Files => DAT-file: MnWO4_magneticAF4_5K.int, PCR-file: commag
Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
1 0 1 0 0 0 0 0 0 0 0 0 0 0 0 0 1 0 0 0 0 1

Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
0 0 0 1 0 1 0 4 0 3 0 0 0 0 0 0 0 0 0 0 0 0

NCY Eps R_at R_an R_pr R_gl Thmin Step Thmax PSD Sent0
1 0.10 1.00 1.00 1.00 1.00 1.5360 0.000000 0.0000 0.000 0.000

2 !Number of refined parameters

Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 9.81

MnWO4 Nuclear refinement, 17%-Co
Nat Dis Mom Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth ATZ Nvk Npr More
1 0 0 0.0 0.0 1.0 1 4 -2 0 0 0.000 1 0 1

Jvi Jdi Hel Sol Mom Ter Brind RMua RMub RMuc Jtyp Nsp_Ref Ph_Shift N_Domains
0 0 0 0 0 0 0 1.0000 1.0000 2.0000 0.0000 1 0 0 0

P -1 <--Space group symbol
Nsym Cen Laue Irefs N_Bas
2 1 1 -1 3
Real(0)-Imaginary(1) indicator for Ci
0 0 0

SYMM X, Y, Z
BASR 1 0 0 0 0 0 0 0 0 0 0 1
BASR 0 0 0 0 0 0 0 0 0 0 0 0
SYMM X, -Y+1, Z+1/2
BASR .125 0 0 0 -.992 0 0 0 .125
BASR .992 0 0 0 -.125 0 0 0 .992

Atom Typ Mag Vek X Y Z Biso Occ C1 C2 C3
C4 C5 C6 C7 C8 C9 MagPh
MN1 MMN3 1 0 0.50000 0.68510 0.25000 0.30000 1.00000 -0.996 1.360 0.000
0.000 0.000 0.000 0.000 0.000 0.000 0.000000
0.000 0.000 0.000 0.000 0.000 0.000 0.000

-----> Scale, Extinction and Cell Parameters for Pattern # 1
Scale Factors
Sc1 Sc2 Sc3 Sc4 Sc5 Sc6
455.1 0.000 0.000 0.000 0.000 0.000 0.000

Extinction Parameters
Ext1 Ext2 Ext3 Ext4 Ext5 Ext6 Ext7 Ext-Model
78.96 0.000 0.000 0.000 0.000 0.000 0.000 0.000 1

a b c alpha beta gamma #Cell Info
4.770000 5.720000 4.920000 90.000000 90.900024 90.000000
0.000000 0.000000 0.000000 0.000000 0.000000 0.000000
x-Lambda/2 + Not yet used parameters
0.02137 0.00000 0.00000 0.00000 0.00000
0.00 0.00 0.00 0.00 0.00

Propagation vectors:
.2170000 .5000000 -.4600000 Propagation Vector 1
0.000000 0.000000 0.000000
2Th1/TOF1 2Th2/TOF2 Pattern # 1
1.536 0.000 1
```

1-use unique axis b as
imaginary component

2-refine C3

Incommensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

incommag.pcr

1-select the .pcr

2-open

3-open .pcr

4-Save

5-exit

The image displays two screenshots of the FullProf Suite software interface, illustrating the steps to open and edit a .pcr file.

Left Screenshot: Select FullProf Suite Files

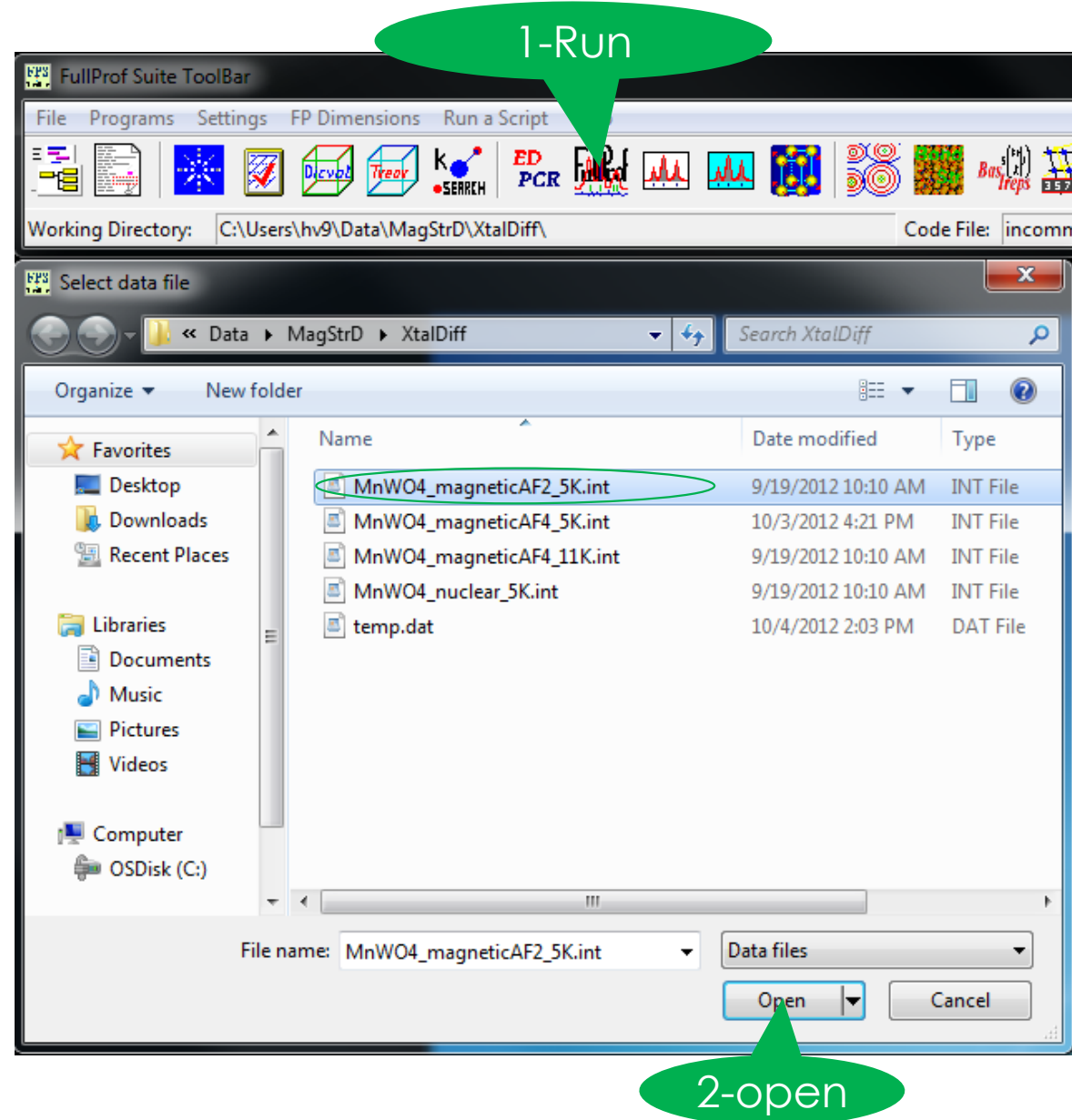
The 'Select FullProf Suite Files' dialog box is shown. The 'File name' field contains 'incommag.pcr'. The 'File type' is set to 'Input Control Files (*.pcr)'. The 'Open' button is highlighted.

Right Screenshot: FullProf PCR Editor

The 'FullProf PCR Editor' window is shown. The main plot area displays a diffraction pattern with peaks labeled 'FullProf PCR Editor'. The x-axis is labeled '2θ (°)' and ranges from 17 to 49. The y-axis represents intensity. The 'Information' panel on the right shows various settings for the job, including 'Title, type of job: Rietveld, Integrated Intensities, Simulated Annealing, ...', 'Type of Patterns, profile, background, diffraction geometry, user-given scattering factors ...', 'Phase name, type of calculations (JBT), ATZ, contribution to patterns, symmetry, ...', 'Number of cycles, relaxation factors, access to patterns and phases (atoms and profile)', 'Constraints definitions, adding, deleting, modifying...', 'Fixing range of parameters, distances, angles, magnetic moments and linear restraints', and 'Output options for patterns and phases: Reflection lists, Fourier, distances, BVS...'. The status bar at the bottom indicates 'incommag', 'Profiles: 1', 'Phases: 1', '4/10/2012', and '14:50:40'.

Incommensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

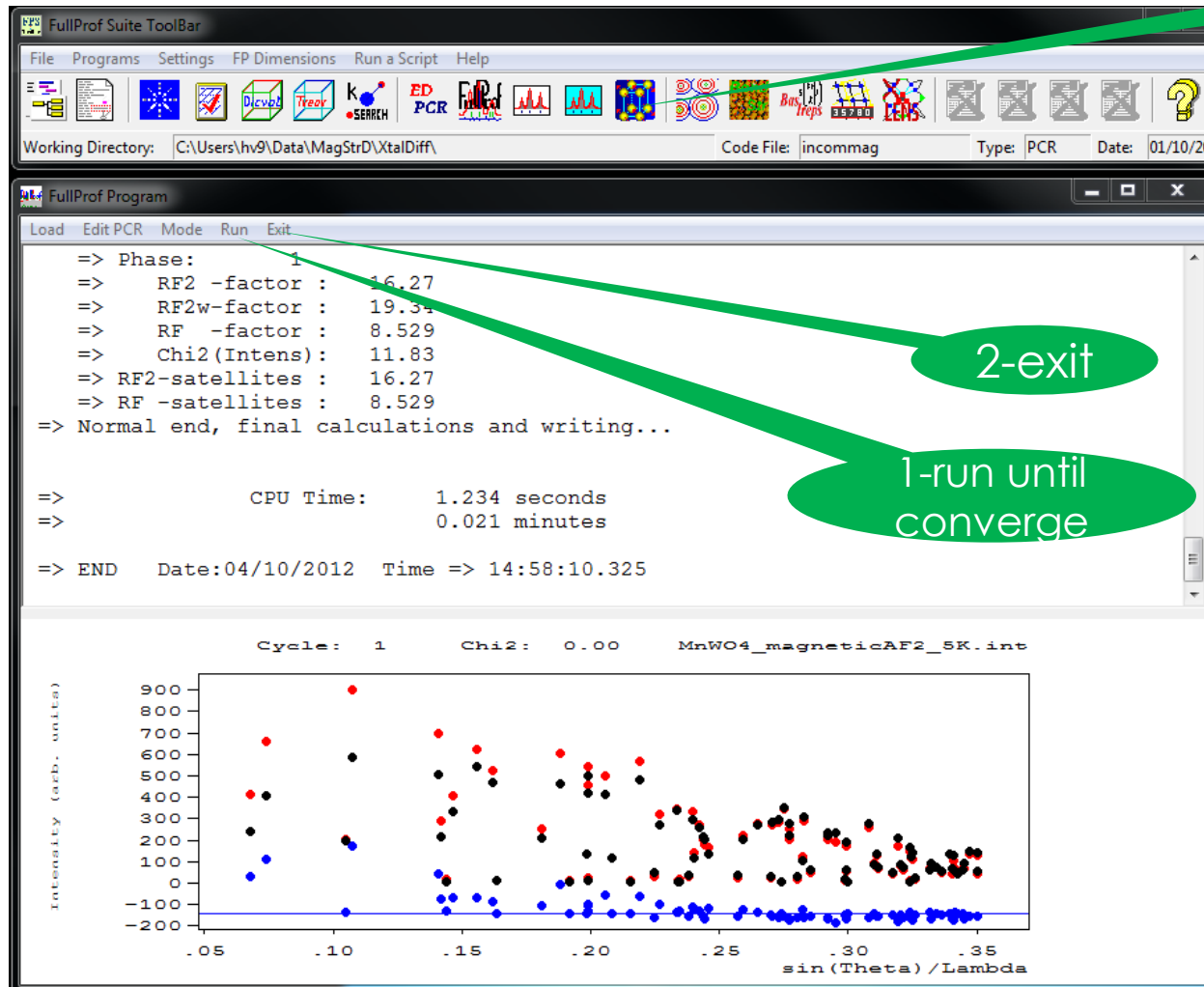
incommag.pcr



Incommensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

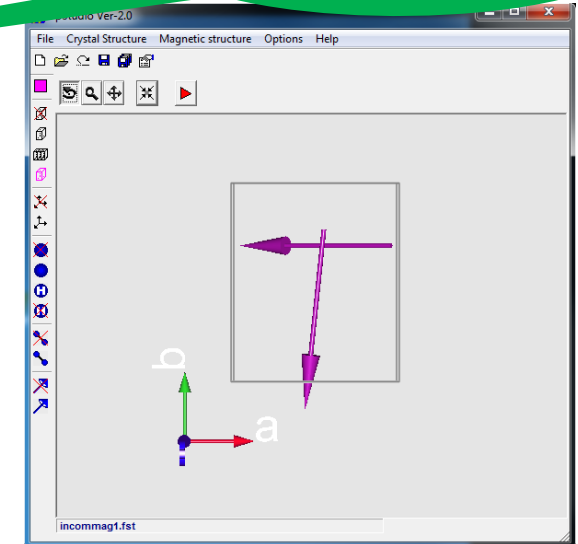
incommag.pcr

3-FpStudio



2-exit

1-run until converge



Incommensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

incommag.fst

4-reload 1-open 2-save 3-exit

The screenshot shows the FullProf Studio interface. The left window, titled 'FpStudio 2.0', displays a 3D model of the magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$. The model shows two overlapping purple ellipsoids representing the magnetic structure, with a coordinate system (a, b, c) at the bottom. The right window, titled 'Edition of incommag1.fst - [C:\Users\hv9\Data\MagStrD\XtalDiff\incommag1.fst]', displays the file content. The file content includes the title 'MnWO4 Nuclear refinement, 17%-Co', the space group 'P -1', the cell parameters, and the magnetic structure definition. The magnetic structure is defined by two Mn atoms, each with a magnetic moment (MN) and a scale factor (SCALE 1.0). The magnetic moments are labeled 'ENVELOPE' and are circled in green. A blue arrow points from the '4-reload' callout to the '1-open' callout.

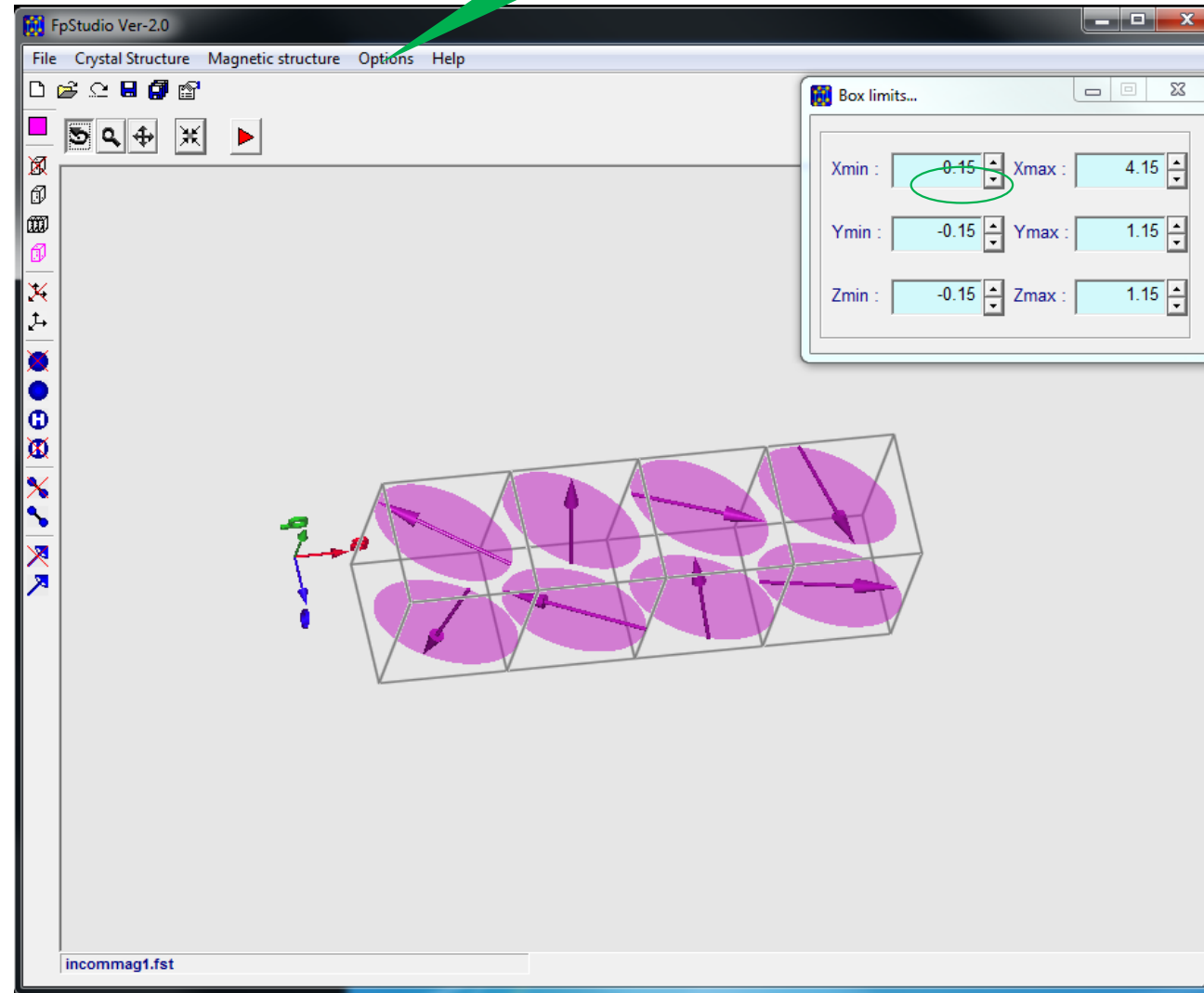
```
! FILE for FullProf Studio: generated automatically by FullProf
!Title: MnWO4 Nuclear refinement, 17%-Co
SPACEG P -1
CELL 4.770000 5.720000 4.920000 90.0000 90.9000 90.0000 DISPLAY MULTIPLE
BOX -0.15 1.15 -0.15 1.15 -0.15 1.15

{
LATTICE P
K 0.21700 0.50000 -0.46000
SYMM x,y,z
MSYM u,v,w,0.0
MATOM MN1_1 MN 0.50000 0.68510 0.25000 SCALE 1.0 GROUP ENVELOPE
SKP 1 1 -4.38685 0.00000 -3.09192 0.00000 5.21172 0.00000 0.00000
MATOM MN1_2 MN 0.50000 0.31490 0.75000 SCALE 1.0 GROUP ENVELOPE
SKP 1 1 -0.54836 -5.17003 -0.38649 -4.35175 -0.65147 -3.06718 0.00000
}
```

Incommensurate magnetic structure of $\text{Mn}_{1-x}\text{Co}_x\text{WO}_4$

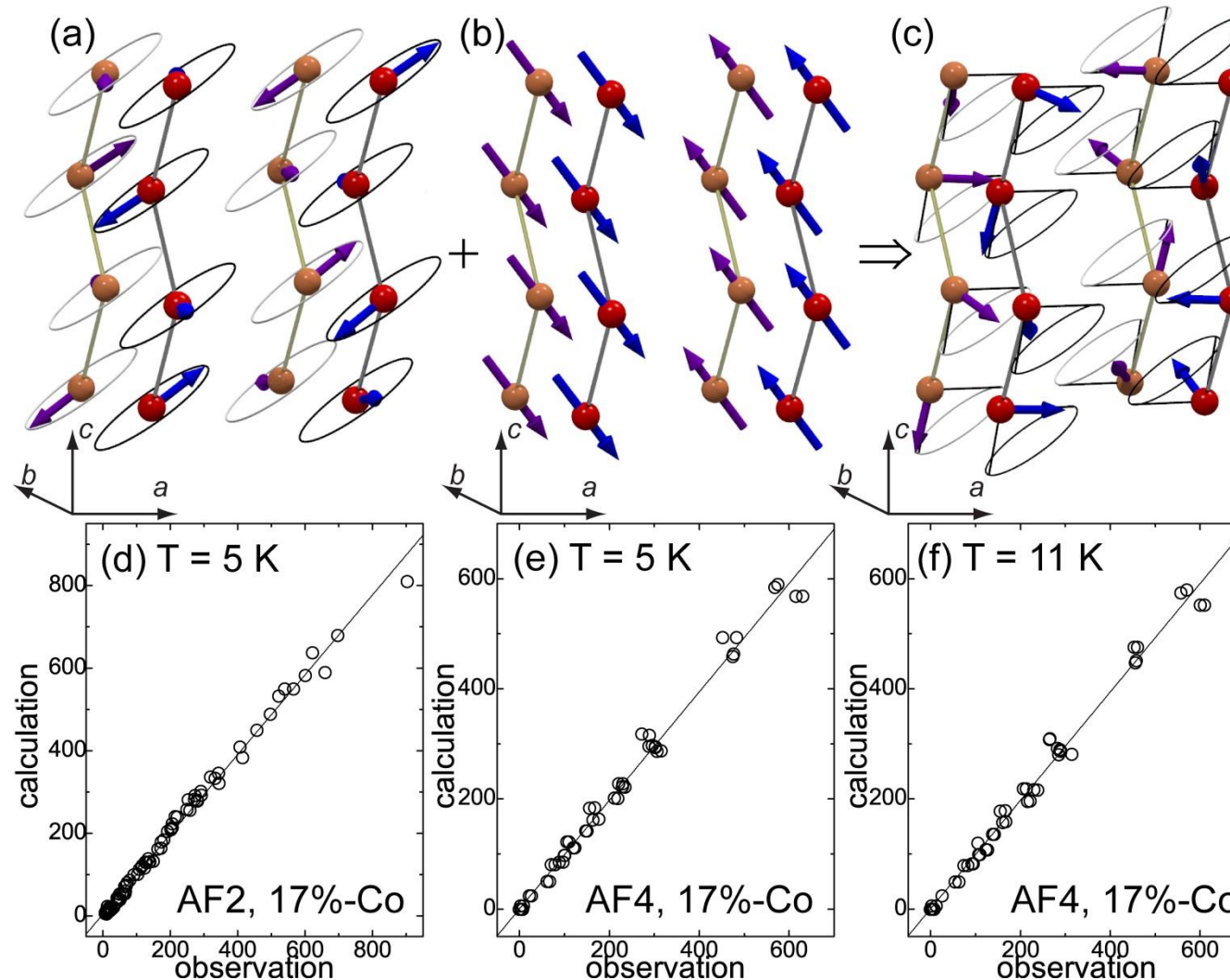
incommag.fst

Box limits



What is the magnetic structure at 5 K?

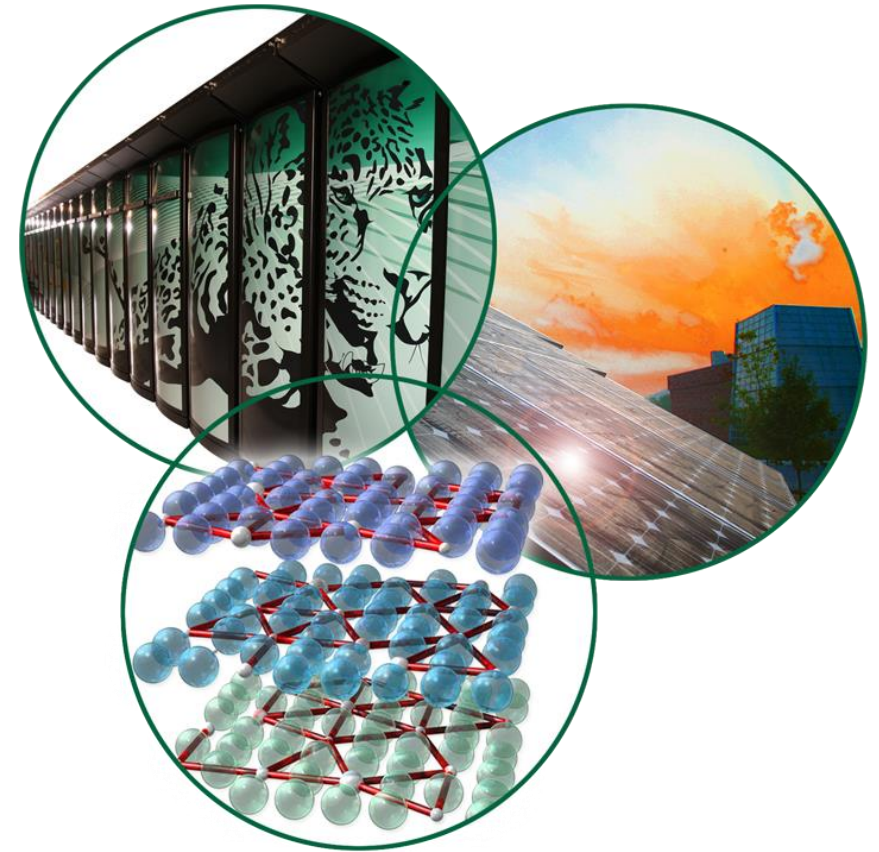
Combine two magnetic structures together



Atomic displacements and thermal motion in triphylite - lithiophilite, Li(Fe,Mn)PO_4 solid solution

Huibo Cao
Bryan Chakoumakos

Neutron Scattering Science Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee



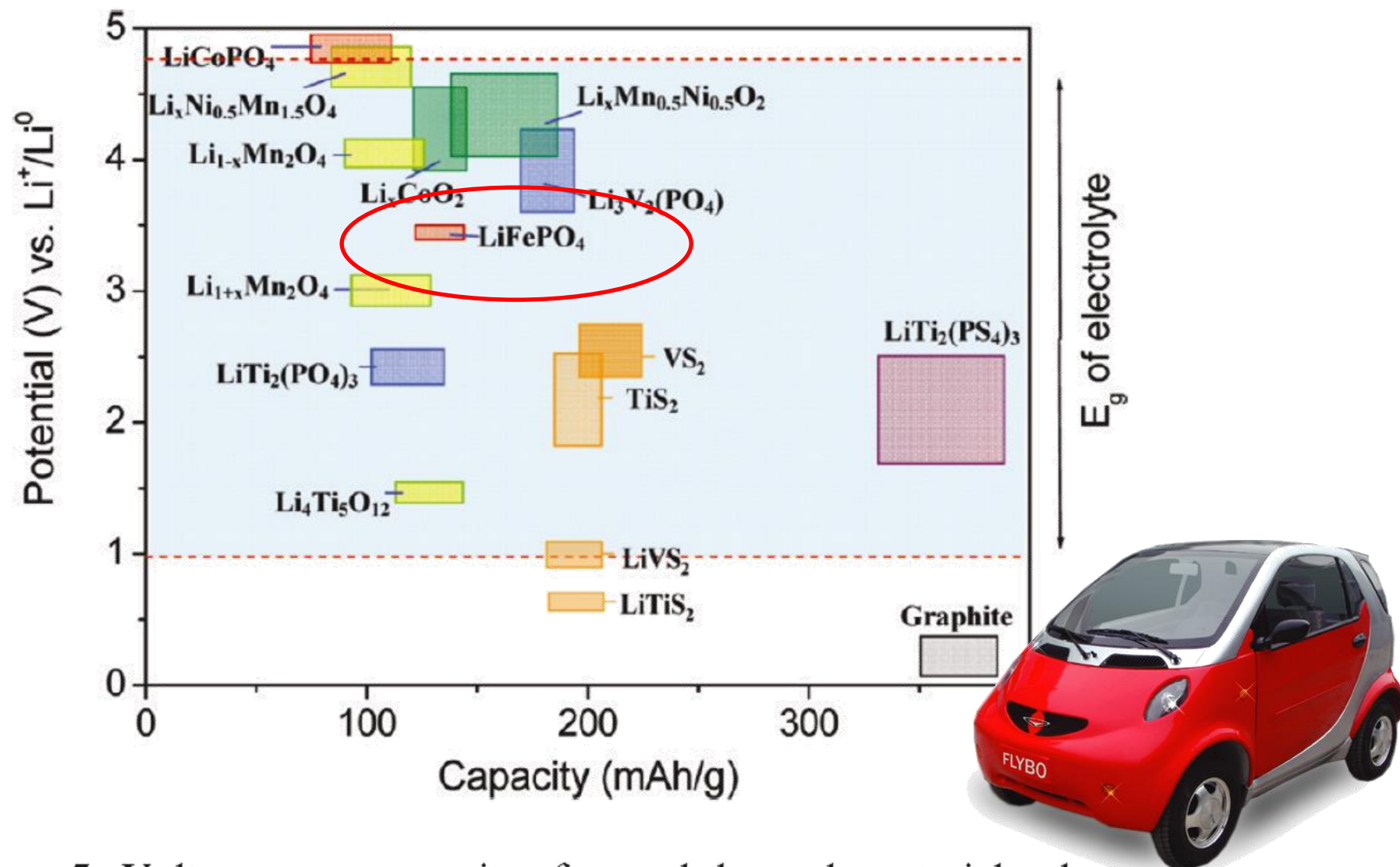
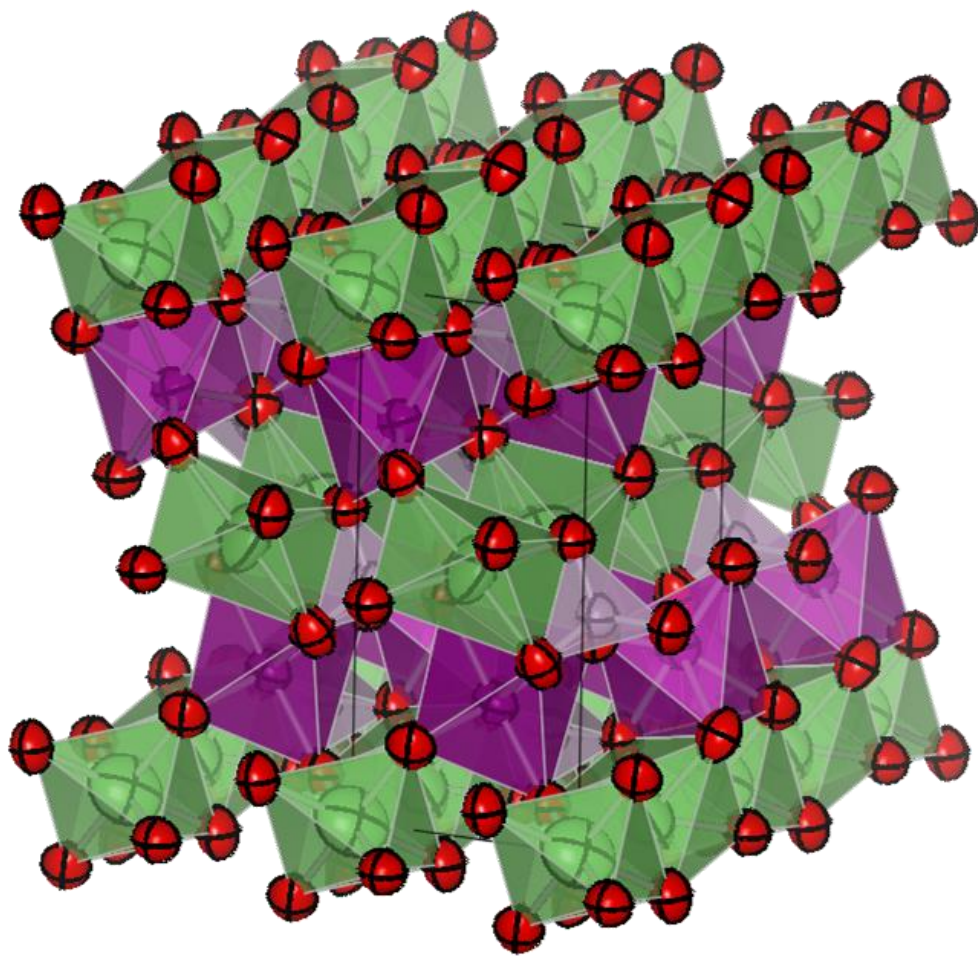
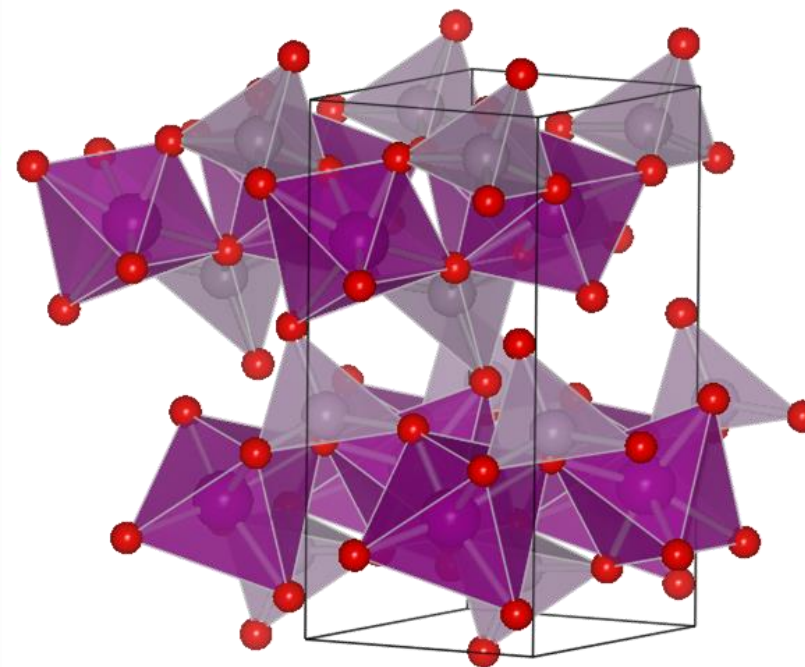


Figure 5. Voltage versus capacity of several electrode materials relative to the window of the electrolyte 1 M LiPF_6 in EC/DEC (1:1).
(Goodenough and Kim 2010)



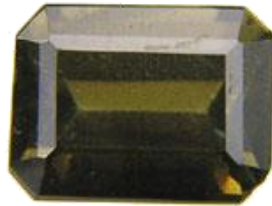
$\text{LiFe}^{2+}\text{PO}_4$

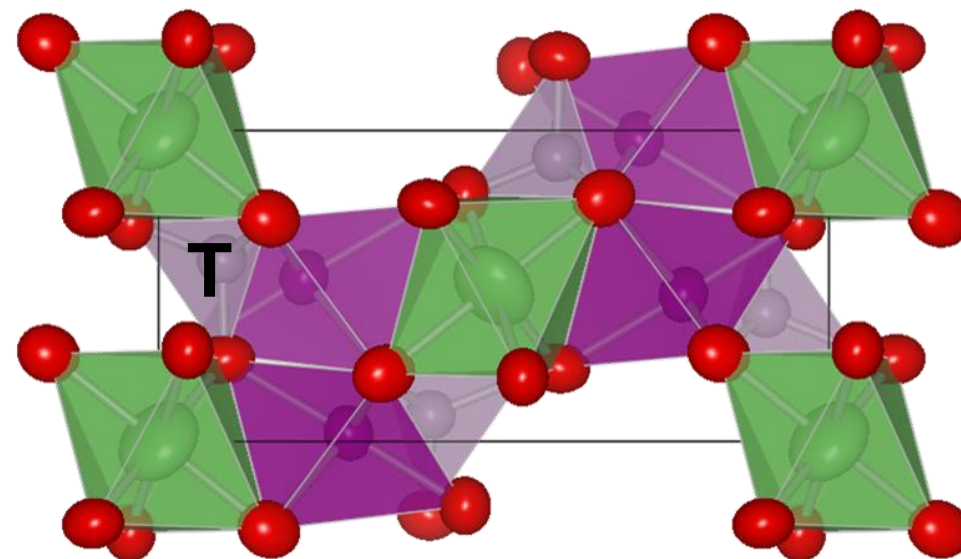
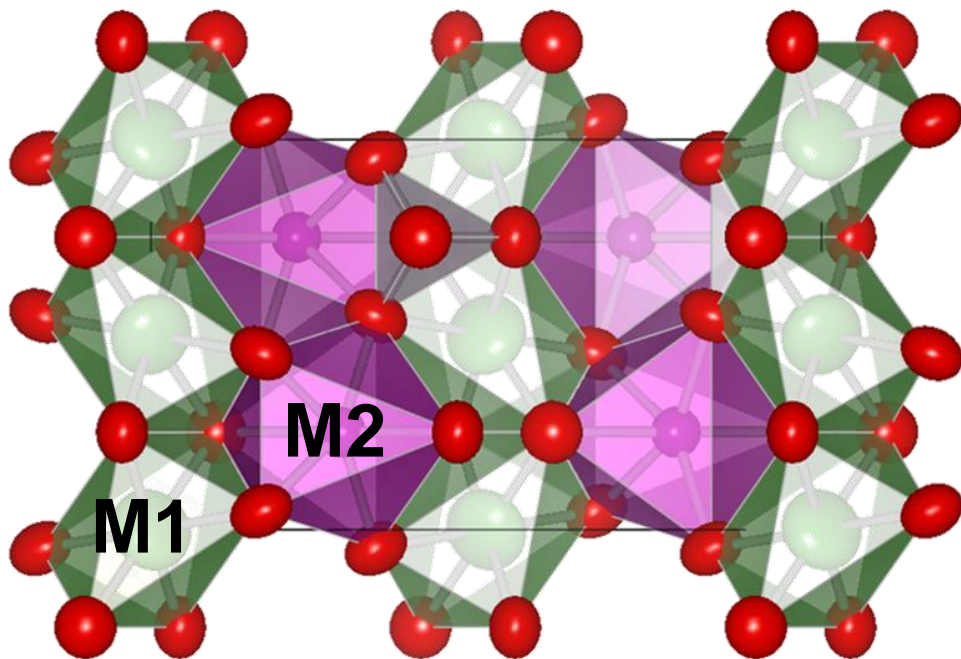


$\text{Fe}^{3+}\text{PO}_4$
Delacourt et al. (2005)



**Triphylite – lithiophilite
(LiFePO_4 – LiMnPO_4) occurs
naturally as large crystals in
granitic pegmatites, which are
noted for their giant crystal
textures.**





Olivine

Structure-Type

$M1M2TX_4$

Pnma

atom	site	position	<i>ADP</i>
M1	4a	-1 0 0 0	<i>U</i>
M2	4c	. <i>m.</i> <i>x</i> ¼ <i>z</i>	<i>U</i>
T	4c	. <i>m.</i> <i>x</i> ¼ <i>z</i>	<i>U</i>
X1	4c	. <i>m.</i> <i>x</i> ¼ <i>z</i>	<i>U</i>
X2	4c	. <i>m.</i> <i>x</i> ¼ <i>z</i>	<i>U</i>
X3	8d	1 <i>x</i> <i>y</i> <i>z</i>	<i>U</i>

Structural parameters:
 20 isotropic
 43 anisotropic

Neutron diffraction refinement details

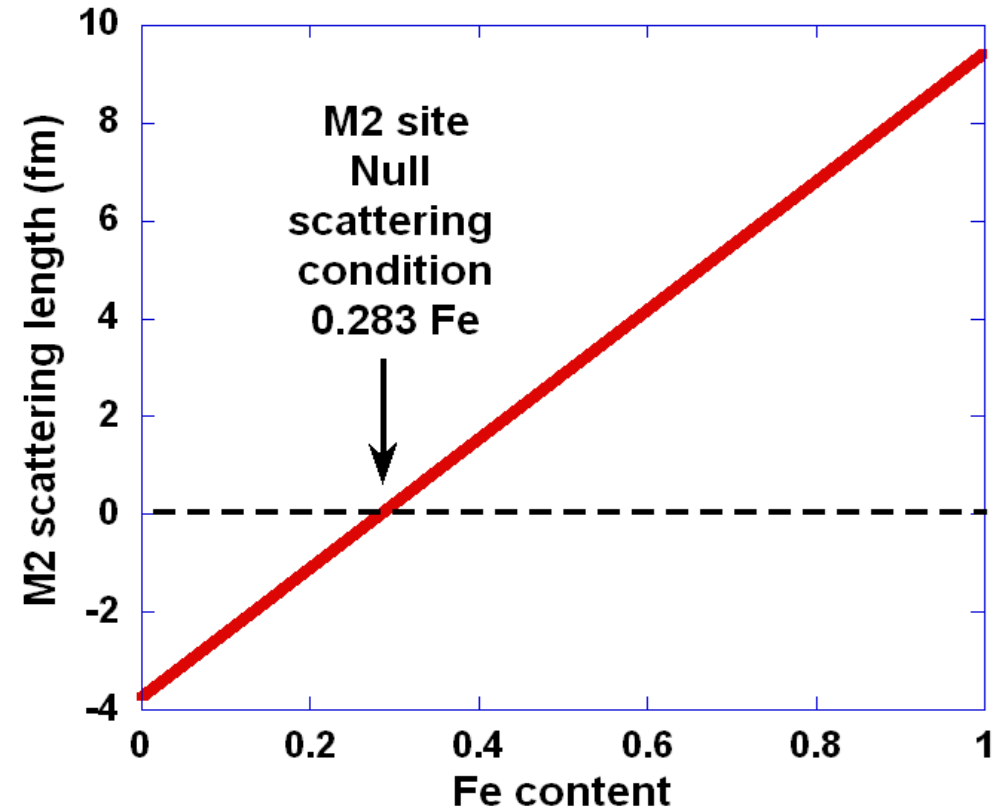
Scattering Lengths

Li **-1.90 fm**

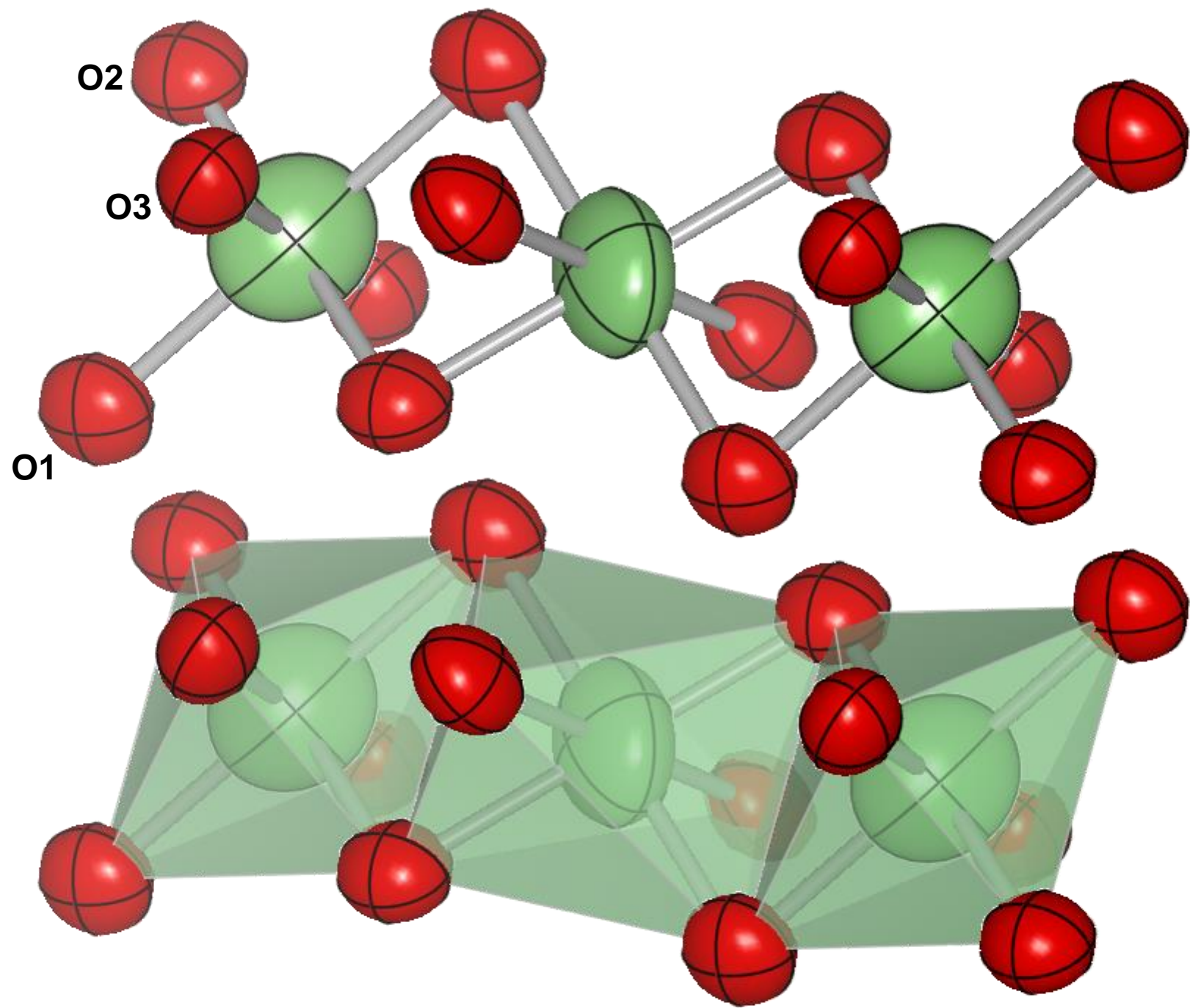
Mn **-3.73 fm**

Fe **9.45 fm**

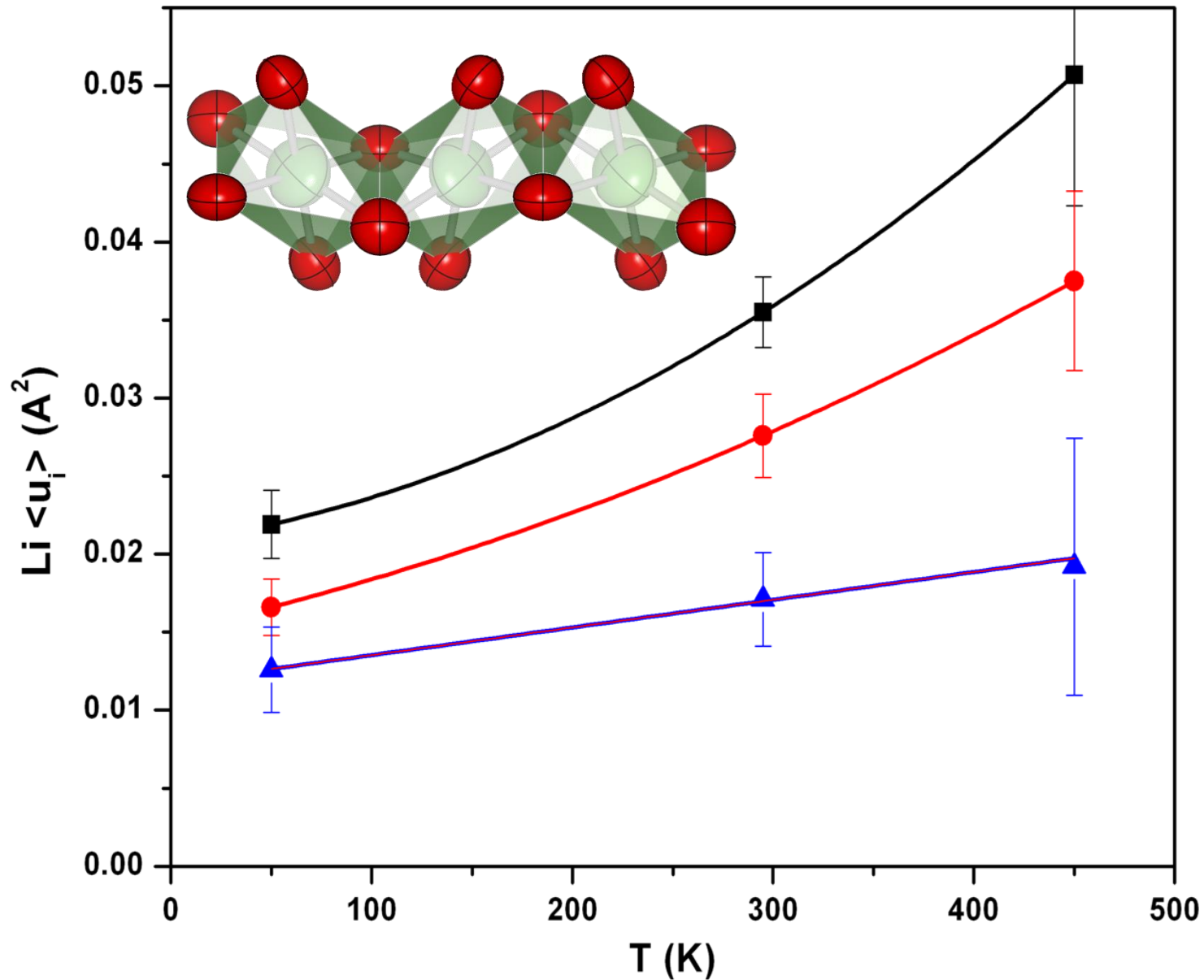
O **5.803 fm**



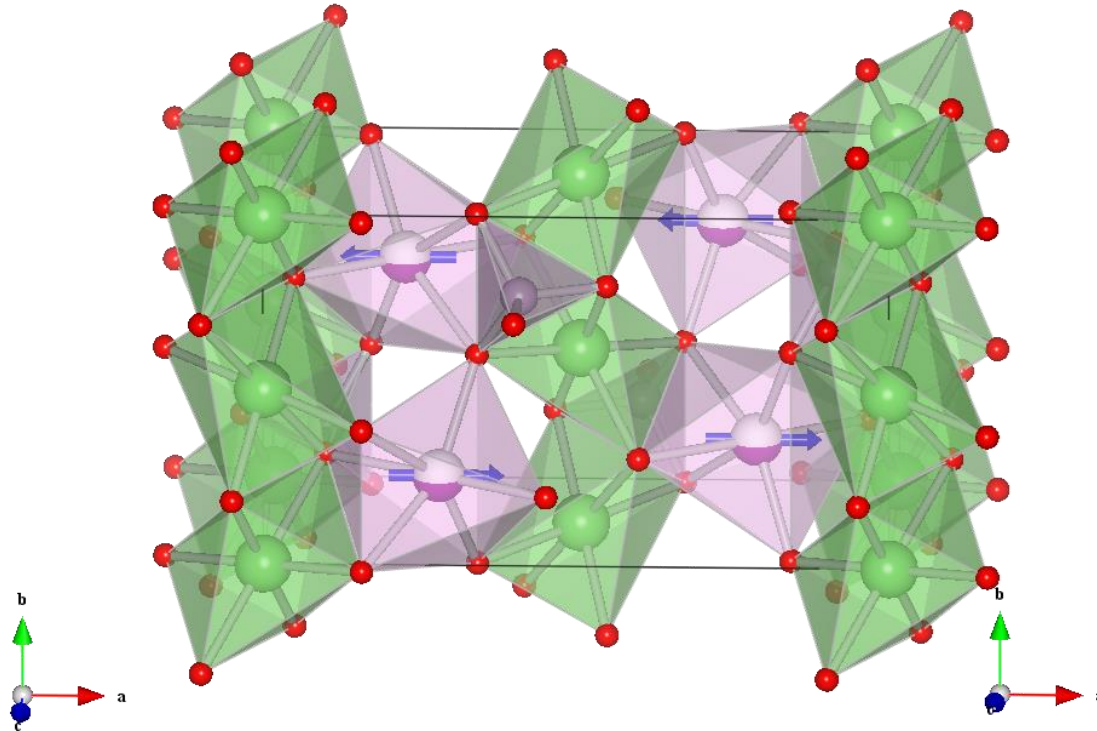
- We need to measure about 450 unique reflections to get a good refinement, i.e., data to parameter ratio ~ 10:1.
- Orthorhombic symmetry allows us to collect one octant of the full sphere of reflections.



Principal Mean Square Atomic Displacements for Li in Triphylite $\text{Li}(\text{Fe}_{0.04}\text{Mn}_{0.96})\text{PO}_4$



Magnetic Structures



Lithiophilite LiMnPO_4

$T_N = 34 \text{ K}$

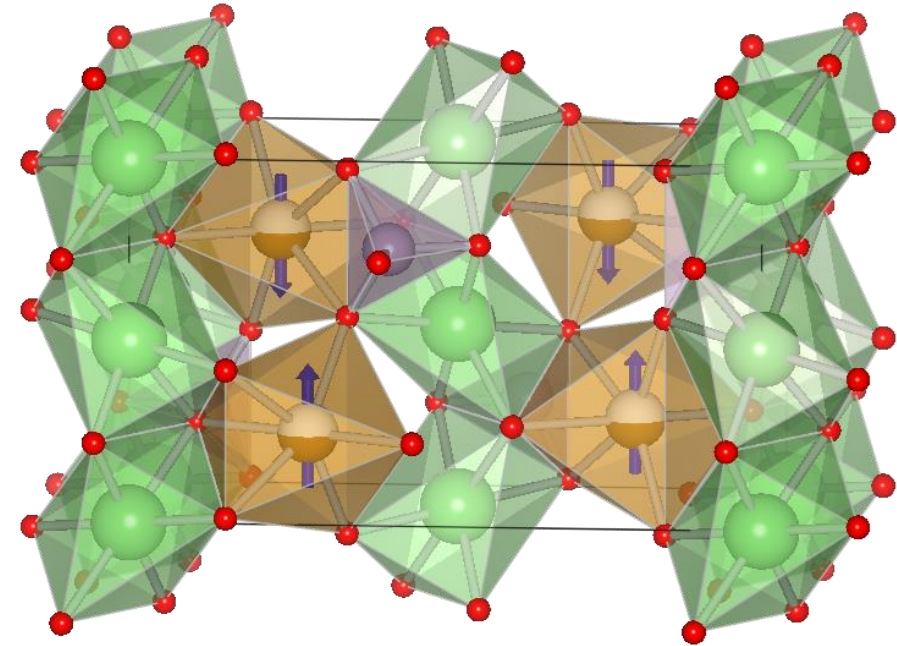
$M = 4.3 \mu_B$

along a axis

Collinear antiferromagnetic

Li et al.

Phys. Rev. B (2009), 79, 144410.



Triphylite LiFePO_4

$T_N = 52 \text{ K}$

$M = 4.6 \mu_B$

along b axis

Collinear antiferromagnet

Santoro and Newnham,

Acta Cryst. (1967) 22, 344.

Single crystal diffraction

Thanks !

Questions to caoh@ornl.gov