

From mCIF to refinement: Using the Bilbao Crystallographic Server and fullprof to refine Cr_2WO_6 data

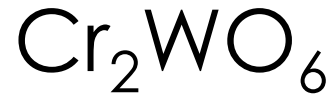
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Magnetic Structure Determination
from Neutron Diffraction Data
October 21-23, 2019

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ENERGY



- There is a Cr_2WO_6 example (data/notes) using representation analysis (SARAh/Basirep) and magnetic matrices (Fullprof).
- We will start from the same .cif file used in the other examples and use the same neutron data set. The k-vector has been previously found using k-search (see other Cr_2WO_6 example in this workshop).
- This example will use the Bilbao server and follow these steps:
 - Step 1: create an mcif file with MAXMAGN on the Bilbao Crystallographic Server.
 - Step 2: convert that mcif file into a .pcr file using mCIF2PCR.
 - Step 3: use the created .pcr file to fit the neutron data using Fullprof to determined the magnetic structure.

Step 1: Creating mCIF file

- Go to Bilbao server: <http://www.cryst.ehu.es/>
- Select “Magnetic Symmetry and Applications” to open the drop-down menu

bilbao crystallographic server UPV EHU

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Magnetic Symmetry and Applications

Space Groups
Plane Groups
Layer Groups
Rod Groups
Frieze Groups
2D Point Groups
3D Point Groups
Magnetic Space Groups

ECM31-Oviedo Satellite
Crystallography online: workshop on the use and applications of the structural tools of the Bilbao Crystallographic Server
20-21 August 2018

News:

- **New Article in Nature**
07/2017: Bradlyn et al. "Topological quantum chemistry"
Nature (2017), 547, 298-305.
- **New program: BANDREP**
04/2017: Band representations and Elementary Band representations of Double Space Groups.
- **New section: Double point and space groups**
 - New program: DGENPOS
04/2017: General positions of Double Space Groups
 - New program: REPRESENTATIONS DPG
04/2017: Irreducible representations of the Double Point Groups
 - New program: REPRESENTATIONS DSG
04/2017: Irreducible representations of the Double Space Groups
 - New program: DSITESYM
04/2017: Site-symmetry induced representations of Double Space Groups
 - New program: DCOMPREL
04/2017: Compatibility relations between the irreducible representations of Double Space Groups

Space-group symmetry
Magnetic Symmetry and Applications
Group-Subgroup Relations of Space Groups
Representations and Applications
Solid State Theory Applications
Structure Utilities
Subperiodic Groups: Layer, Rod and Frieze Groups
Structure Databases
Raman and Hyper-Raman scattering
Point-group symmetry
Plane-group symmetry
Double point and space groups

Step 1: Creating mCIF file

- Then select “MAXMAGN”

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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
MAGMODELER	Magnetic structure models for any given magnetic symmetry
k-SUBGROUPSMAG ⚠	Magnetic subgroups consistent with some given propagation vector(s) or a supercell
MAGNDATA ⚠	A collection of magnetic structures with transportable cif-type files
MVISUALIZE ⚠	3D Visualization of magnetic structures with Jmol
MTENSOR ⚠	Symmetry-adapted form of crystal tensors in magnetic phases

Group-Subgroup Relations of Space Groups

Representations and Applications

Solid State Theory Applications

Step 1: Creating mCIF file

- **[1]** Need to have a propagation vector. This can be determined using k-search in fullprof (see other example for Cr_2WO_6). For Cr_2WO_6 it is (0,0,0).
- **[2]** Input the space group number of the crystal structure [136] (or choose from a list of all space groups)- *Note: if you have a cif file this step is not necessary.*
- **[3]** Check the box “Structure data of the paramagnetic phase will be included”. This allows you to input a .cif file.

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 ISODISTORT, JANA2006, StrConvert, VESTA, etc. These cif-like files can also be submitted to the program MVSUALIZE, which allow 3D visualization of magnetic structures with Jmol. A controlled descent to lower symmetries is also possible.

This program provides an alternative to the traditional representation method for the parameterization of magnetic structures.

MAXMAGN tutorials:

Abbreviated tutorial: [download](#)

Extended tutorial: [download](#)

Last tutorial: [download](#)

Examples and further information can be found in the following paper:

J.M. Perez-Mato, S.V. Gallego, E.S. Tasci, L. Elcoro, G. de la Flor, and M.I. Aroyo
Annu. Rev. Mater. Res. (2015), 45:13.1-13.32

which can be used to cite this program.

3

☒ Structure data of the paramagnetic phase will be included

2

☐ Non-conventional setting

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

choose it

136

Please, enter the propagation vector k:

k_x

0

k_y

0

k_z

0

1

Submit

Step 1: Creating mCIF file

- Choose .cif file (crystal structure only).

The one for this example is
“Cr2WO6.cif”

- Then upload the file.
- If you don't have a cif file then have to fill out the details in “Option 2”

Parent paramagnetic structure cif file

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

Cr2WO6.cif

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

Option 2: Specify structure data by hand:

Space Group: 136 ($P4_2/mnm$)

Lattice parameters (Angstroms and degrees):

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

Number of unique atomic positions:

Step 1: Creating mCIF file

- The paramagnetic phase information is displayed.
 - Select the magnetic atom(s). This case is Cr only.
 - Push Submit. This may take a few minutes to run the calculations.....

Parent phase structure data: Magnetic Atoms

Parent space group: 136 ($P4_2/mnm$)

Lattice parameters (Angstroms and degrees): a=4.57100, b=4.57100, c=8.85300, alpha=90., beta=90., gamma=90.

Atoms: Please select the magnetic ones

N	Atom name	Atom type	Wyckoff Position	Coordinates	Magnetic?
1	W1	W	2a	0.00000 0.00000 0.00000	<input type="checkbox"/>
2	Cr1	Cr	4e	0.00000 0.00000 0.33300	<input checked="" type="checkbox"/>
3	O1	O	4f	0.30800 0.30800 0.00000	<input type="checkbox"/>
4	O2	O	8j	0.30800 0.30800 0.33300	<input type="checkbox"/>

Submit

Step 1: Creating mCIF file

- The possible magnetic space groups are displayed in grey
- To view magnetic structure and export mcif file click on “Show” in last column.
- We will choose the final allowed magnetic structure (#58.395) for this example, but all should be checked to ensure the final model is correct or to determine equivalent solutions.

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	$P4_2/m'n'm'$ (#136.503) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
2	$P4_2/m'n'm'$ (#136.502) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
3	$P4_2/m'n'm'$ (#136.501) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
4	$P4_2/m'n'm'$ (#136.500) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
5	$P4_2/mnm$ (#136.499) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
6	$P4_2/mn'm$ (#136.498) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
7	$P4_2/m'nm$ (#136.497) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
8	$P4_2/mnm$ (#136.495) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
9	$Cmm'm'$ (#65.486) Go to a subgroup	$\begin{pmatrix} 1 & 1 & 0 & 0 \\ -1 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
10	$Cm'mm$ (#65.483) Go to a subgroup	$\begin{pmatrix} 1 & 1 & 0 & 0 \\ -1 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
11	$Pnn'm'$ (#58.398) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show
12	$Pn'm$ (#58.395) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{pmatrix}$ Alternatives (domain-related)	Show	Systematic absences MAGNEXT Tensor properties MTENSOR	Show

Step 1: Creating mCIF file

- [1]** The red values under “Magnetic moment” show allowed spin directions. In this case the spins can only be along x or y.
- Try value along x.
- [2]** Then click “Export to MCIF” file/Visualize

Magnetic Structure

Selected magnetic space group: 12- $Pn'm$ (#58.395)

Setting of the parent group

Parent space group 136 ($P4_2/mnm$)

Lattice parameters: a=4.57100, b=4.57100, c=8.85300, alpha=90., beta=90., gamma=90.

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

[Go to an alternative setting]

2

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	W1 W 0.00000 0.00000 0.00000	(0,0,0 0,0,0) (1/2,1/2,1/2 0,0,0)	2	-	
2	Cr1 Cr 0.00000 0.00000 0.33300	(0,0,z $m_x, m_y, 0$) (1/2,1/2,z+1/2 $-m_x, m_y, 0$) (1/2,1/2,-z+1/2 $m_x, -m_y, 0$) (0,0,-z $-m_x, -m_y, 0$)	4	($M_x, M_y, 0$)	$M_x = 1$ $M_y = 0.00000$
3	O1 O 0.30800 0.30800 0.00000	(x,x,0 0,0, m_z) (-x,-x,0 0,0,- m_z) (-x+1/2,x+1/2,1/2 0,0, m_z) (x+1/2,-x+1/2,1/2 0,0,- m_z)	4	-	-
4	O2 O 0.30800 0.30800 0.33300	(x,x,z m_x, m_y, m_z) (-x,-x,z $m_x, m_y, -m_z$) (-x+1/2,x+1/2,z+1/2 $-m_x, m_y, m_z$) (x+1/2,-x+1/2,z+1/2 $-m_x, m_y, -m_z$) (-x+1/2,x+1/2,-z+1/2 $m_x, -m_y, m_z$) (x+1/2,-x+1/2,-z+1/2 $m_x, -m_y, -m_z$) (x,x,-z $-m_x, -m_y, m_z$) (-x,-x,-z $-m_x, -m_y, -m_z$)	8	-	-

Step 1: Creating mCIF file

- The mcif file is displayed.
- **[1]** This can be downloaded by clicking on “bcs_file.mcif”
- **[2]** Magnetic structure can also be visualized by clicking “Submit to MVSUALIZE”

mCIF file of the structure

Submit this mcif file to MVSUALIZE for 3D visualization of the eststructure using Jmol

Submit to MVSUALIZE

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: 19/06/2018 04:11:35
# Cr2W06.cif

data_5yOhtAoR
_audit_creation_date      2018-06-19
_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
```

Step 1: Creating mCIF file

- MVisualize lets you quickly check magnetic structure.

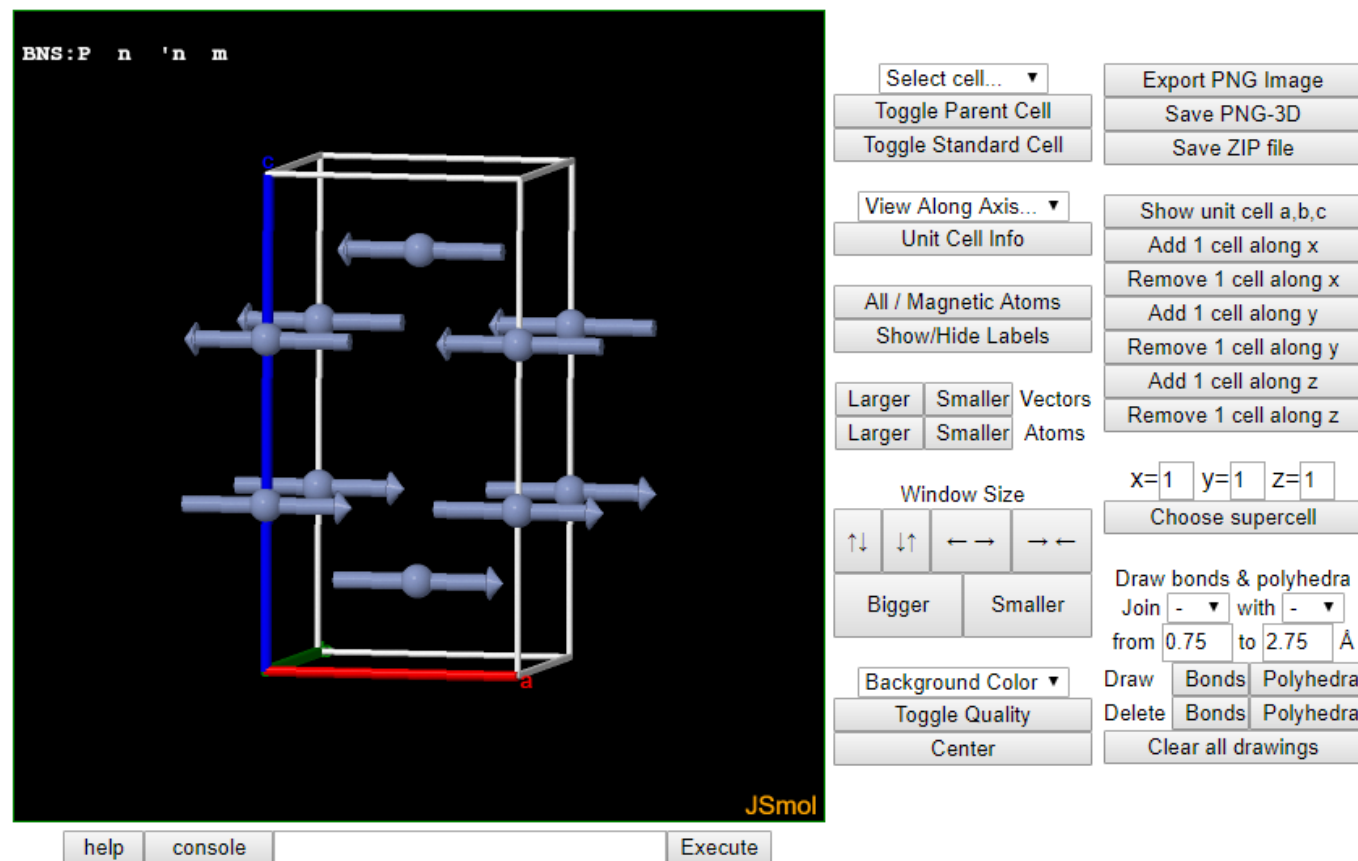
- But we will use the downloaded mcif to fit data using fullprof.

We now need to go to Step 2

[MVisualize Main Page](#)

Show/Hide File

MVisualize: 3D Visualization of magnetic structures with Jmol



Step 2: Creating a pcr from an mcif file

- Go back to the main page on server and open mCIF2PCR

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Space-group symmetry

Magnetic Symmetry and Applications

MGENPOS

MWYCKPOS

MNORMALIZER

IDENTIFY MAGNETIC GROUP

BNS2OG

mCIF2PCR

MPOINT

MAGNEXT

MAXMAGN

MAGMODELIZE

k-SUBGROUPSMAG

MAGNDATA

MVISUALIZE

MTENSOR

General Positions of Magnetic Space Groups

Wyckoff Positions of Magnetic Space Groups

Normalizers of Magnetic Space Groups

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

Transformation of symmetry operations between BNS and OG settings

Transformation from mCIF to PCR format (FullProf).

Magnetic Point Group Tables

Extinction Rules of Magnetic Space Groups

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

Magnetic structure models for any given magnetic symmetry

Magnetic subgroups consistent with some given propagation vector(s) or a supercell

A collection of magnetic structures with transportable cif-type files

3D Visualization of magnetic structures with Jmol

Symmetry-adapted form of crystal tensors in magnetic phases

Group-Subgroup Relations of Space Groups

Representations and Applications

Solid State Theory Applications

Step 2: Creating a pcr from an mcif file

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

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

Choose a structure file (mCIF format):

Choose File bcs_file_21132.mcif

Convert

- **[1]** Choose the mcif we just created through maxmagn
- **[2]** Click convert

The file has been successfully converted.
[Click to download it](#)

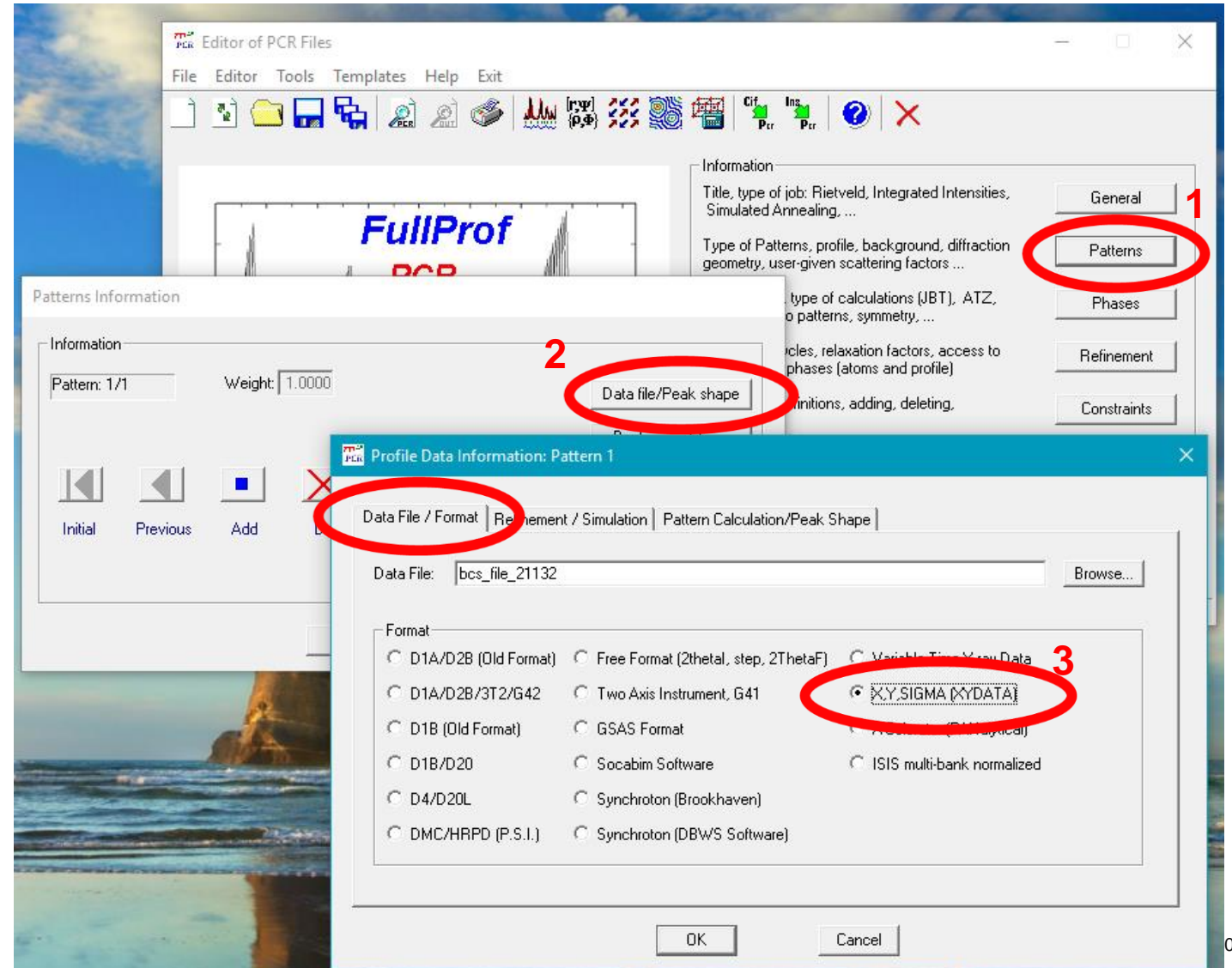
- This will convert the file and “Click to download it” will download the .pcr file
- We will use this .pcr file in Step 3. Put it in the same folder as the data file “Cr2WO6_T4K.dat”

Step 3: Refining the magnetic structure with Fullprof

- Pcr file created contains a single phase with crystal structure and magnetic ions.
- The pcr file calculates the pattern based on default values.
- We need to change the defaults in the .pcr file in the same way done for the examples created from a .cif file.
- To do this we work down the Tabs in the GUI (General/Patterns/Phases/Refinement) and/or edit the text file.
- The values will depend on the instrument the data was collected on.
 - This case is for HB-2A at HFIR measured with the 2.41 Å wavelength.

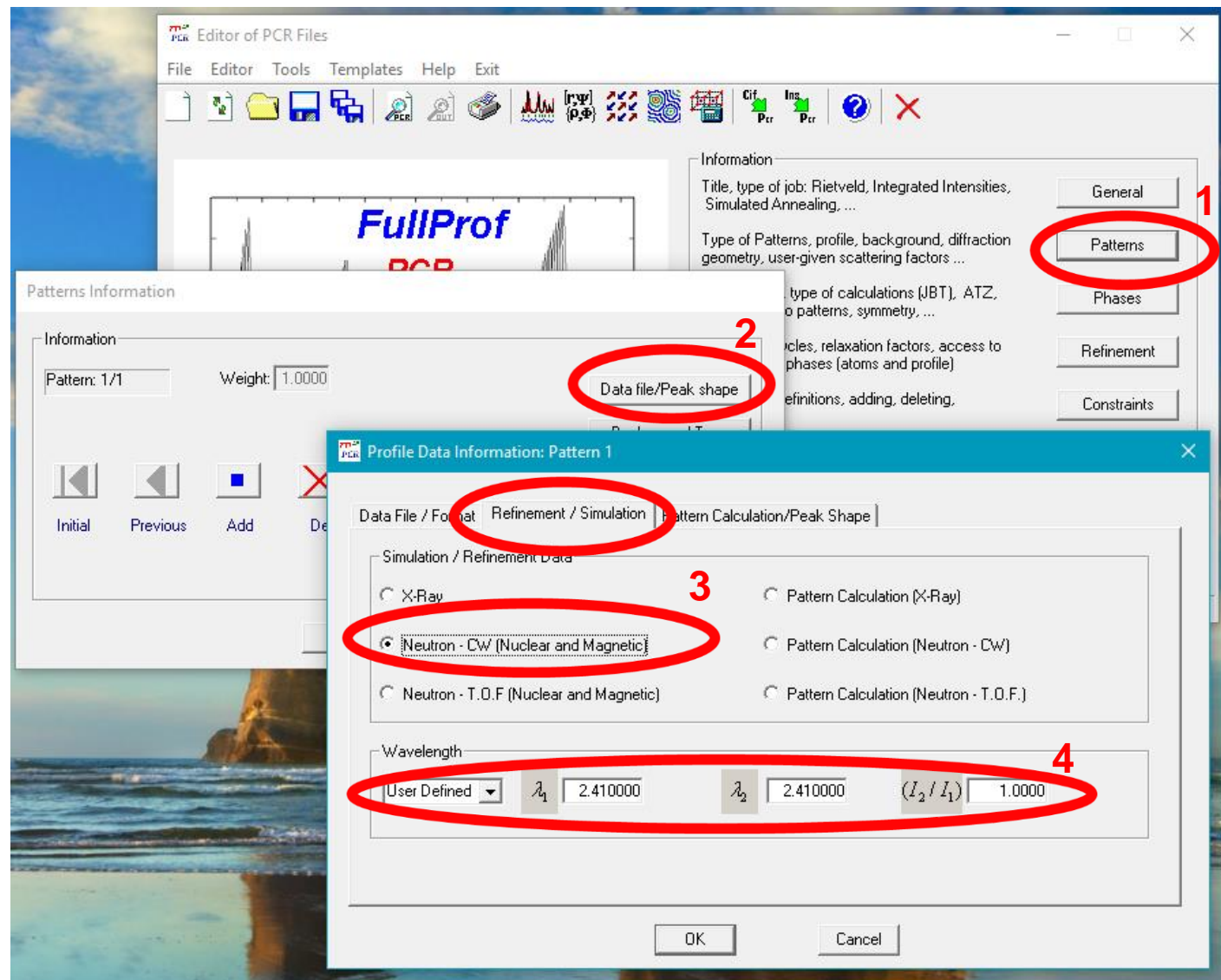
Step 3: Refining the magnetic structure with Fullprof

- Select the format of the data file Fullprof should refine.
- Patterns → Data file/Peak Shape → X,Y,SIGMA (XYDATA)



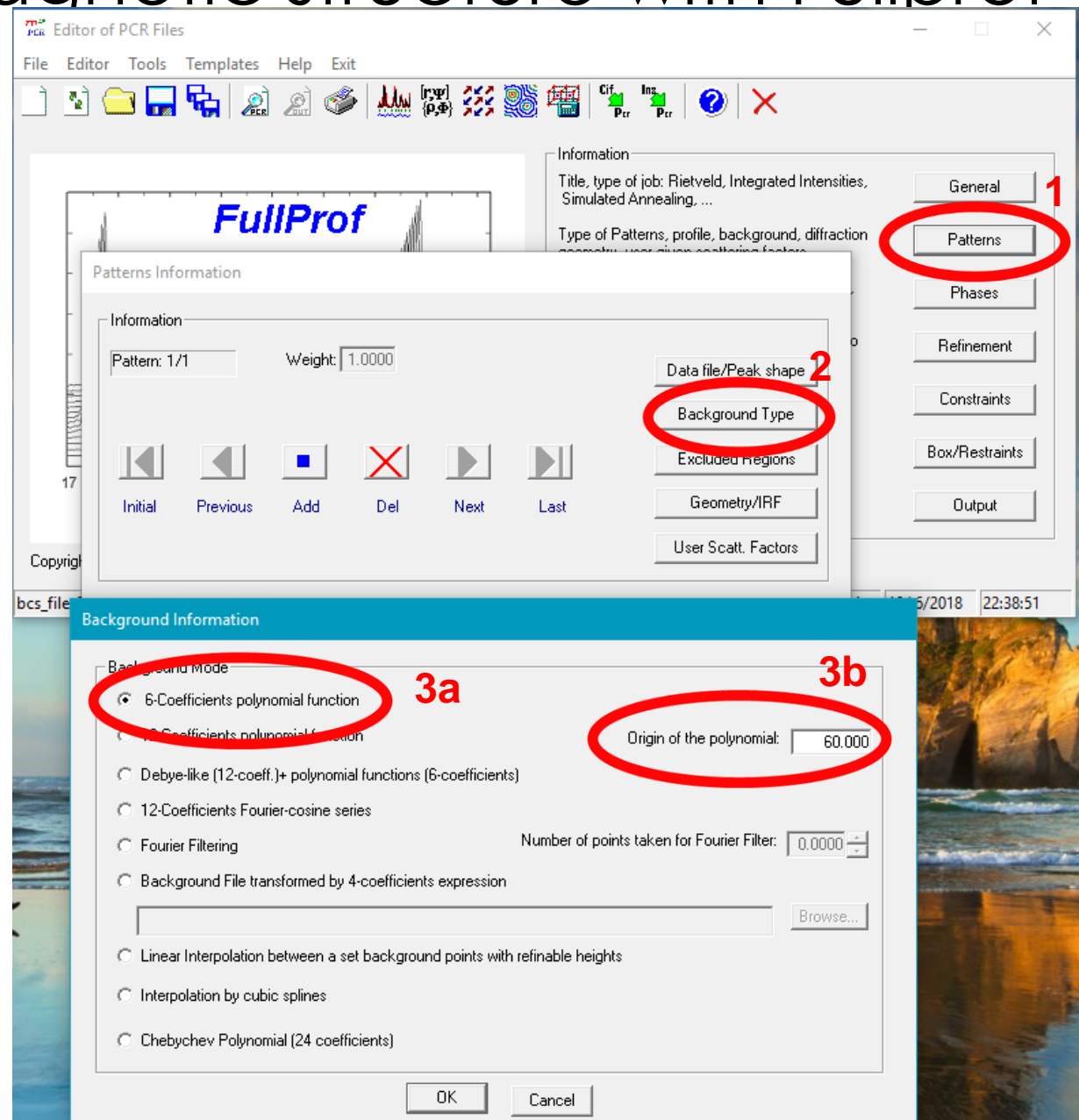
Step 3: Refining the magnetic structure with Fullprof

- Patterns → Data file/Peak Shape
→ Refinement/Simulation
- **[3]** Select Neutron – CW
- **[4]** Change wavelength to user defined and 2.41 Å



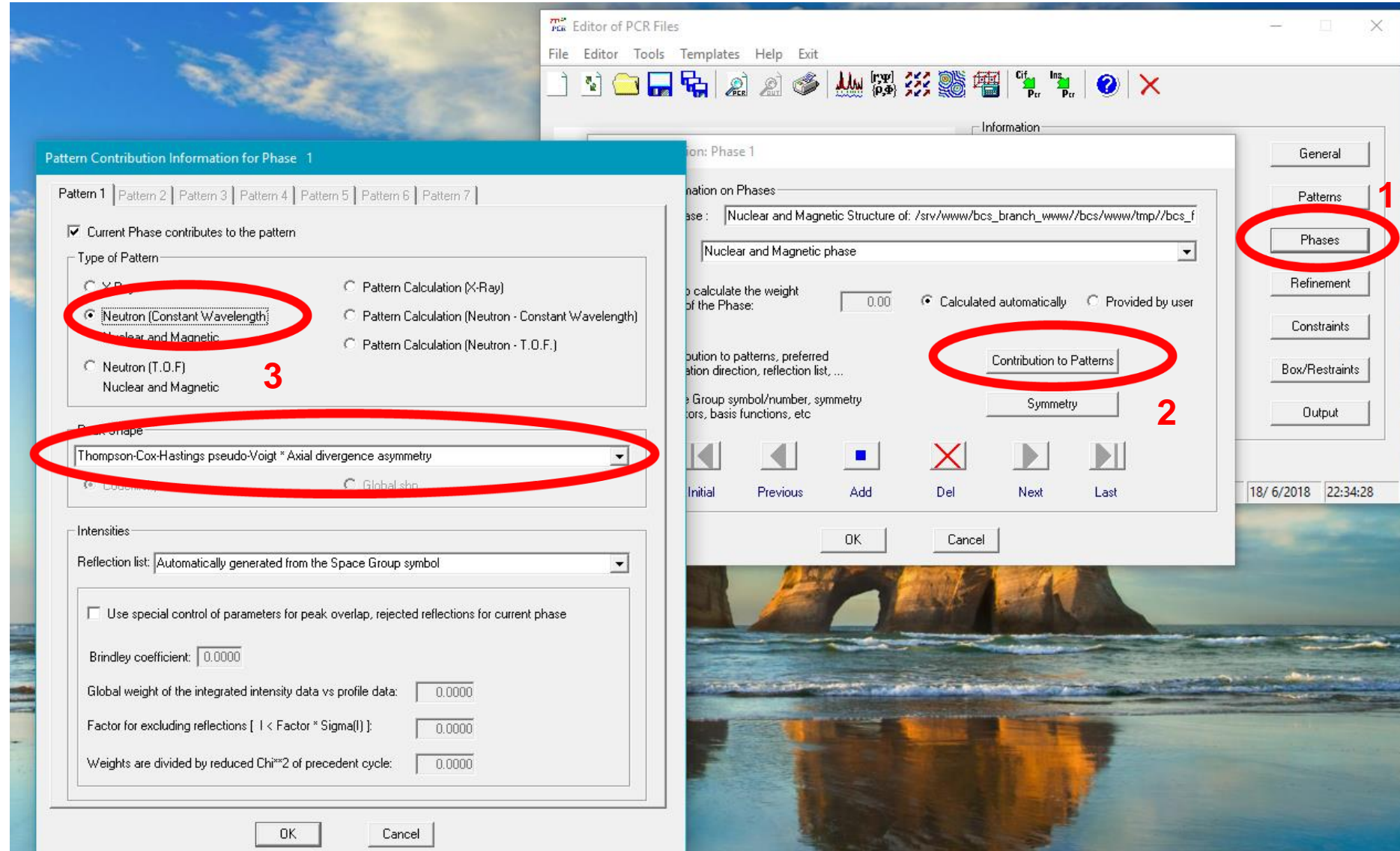
Step 3: Refining the magnetic structure with Fullprof

- Patterns → Background Type → 6-coefficient
- Select background type



Step 3: Refining the magnetic structure with Fullprof

- Make phase contribute to refinement.
- **[1]** Phases → **[2]** Contribution to Patterns → **[3]** Neutron (constant wavelength)
- Peak shape should be “Thompson-Cox....”



Step 3: Refining the magnetic structure with Fullprof

- Setting starting values for refinements
- Starting background values

The screenshot shows the Fullprof software interface. The main window is titled 'Editor of PCR Files'. A 'Refinement Information' dialog box is open, showing various settings for refinement. A red circle labeled '1' highlights the 'Refinement' button in the right-hand pane. Another red circle labeled '2' highlights the 'Background' button in the 'Refinement weighting model' section. A third red circle labeled '3' highlights the input fields for the polynomial coefficients in the '6 Coefficients Polynomial Background: Pattern 1' dialog box.

Refinement Information Dialog Box:

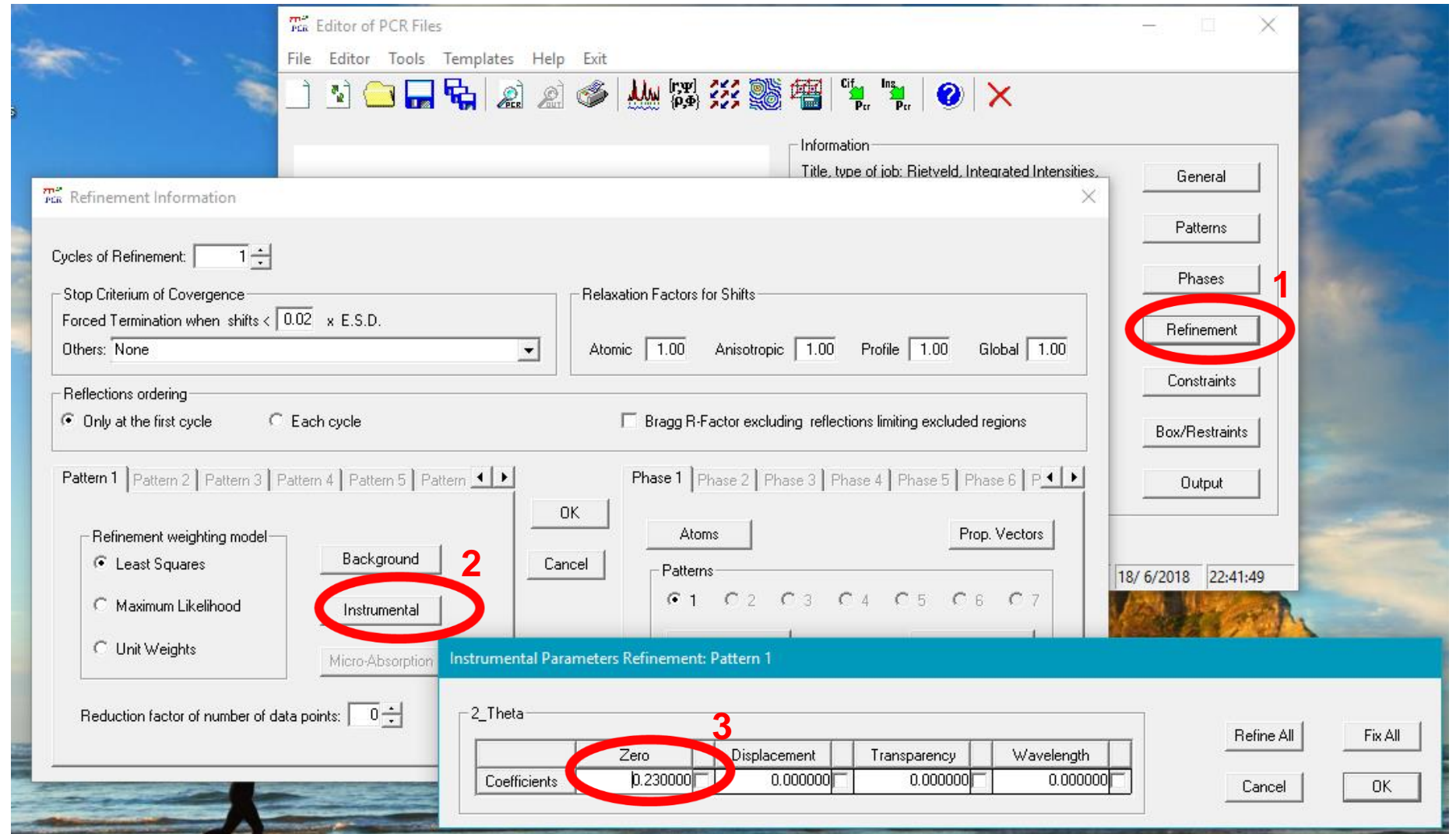
- Cycles of Refinement: 1
- Stop Criterion of Coverage: Forced Termination when shifts < 0.02 x E.S.D.
- Others: None
- Reflections ordering: ☒ Only at the first cycle, ☐ Each cycle
- Relaxation Factors for Shifts: Atomic 1.00, Anisotropic 1.00, Profile 1.00, Global 1.00
- Bragg R-Factor excluding reflections limiting excluded regions: ☐
- Refinement weighting model: ☒ Least Squares, ☐ Maximum Likelihood, ☐ Unit Weights
- Reduction factor of number of data points: 0

6 Coefficients Polynomial Background: Pattern 1 Dialog Box:

	d_0	d_1	d_2	d_3	d_4	d_5
Coefficients	50.000	0.0000	0.0000	0.0000	0.0000	0.0000
	d_6	d_7	d_8	d_9	d_10	d_11
Coefficients						
	d_12	d_13	d_14	d_15	d_16	d_17
Coefficients						
	d_18	d_19	d_20	d_21	d_22	d_23
Coefficients						

Step 3: Refining the magnetic structure with Fullprof

- Instrumental zero value.
- We know this from nuclear refinement done in previous example in this school.



Step 3: Refining the magnetic structure with Fullprof

- Update “Scale” and “Profile” parameters U,V,W,X to those shown

The screenshot displays the FullProf PCR Editor software interface. The main window shows a plot of the diffraction pattern with the text "FullProf PCR Editor" overlaid. On the right, the "Information" panel is visible, with the "Refinement" tab selected and circled in red, marked with a red "1". Below this, the "Refinement Information" panel shows "Cycles of Refinement" set to 1.

The "Profile Parameters: Phase 1 Pattern 1" dialog box is open, showing various refinement parameters. The "Factors" section has "Scale" and "Overall B-factor" circled in red. The "Cell Parameters" section shows a table of coefficients for a, b, c, alpha, beta, and gamma. The "FWHM / Shape Parameters" section has a table of coefficients for U, V, W, IG, X, Y, and SZ, which is circled in red. The "Shape Parameters" section shows a table of coefficients for U2, V2, and W2. On the right side of the dialog, the "Refine All", "Fix All", "Cancel", and "OK" buttons are visible.

Below the main dialog, the "Relaxation Factors for Shifts" panel is visible, showing "Atomic", "Anisotropic", "Profile", and "Global" factors, all set to 1.00. The "Phase 1" tab is selected, and the "Profile" button is circled in red, marked with a red "2".

Step 3: Refining the magnetic structure with Fullprof

- In “Refinement” → “atoms” input thermal parameters (0.3). **[1,2,3]**
- Note that $\text{Re}(x)$ and $\text{Re}(y)$ are set to refine.
- This is a default in files from mcif to pcr that we will turn off in next slide.

The screenshot displays the FullProf PCR Editor software interface. The main window shows a diffraction pattern plot with the text "FullProf PCR Editor" overlaid. To the right, the "Information" panel lists various settings, with the "Refinement" button highlighted by a red circle and labeled with a red "1". Below this, the "Refinement Information" panel shows "Cycles of Refinement" set to 1 and "Stop Criterion of Coverage" set to 0.0001.

In the foreground, the "Atoms Information: Phase 1" window is open. It contains a table of atoms and their refinement parameters. The "B" column is circled in red and labeled with a red "3". The "Re(x)" and "Re(y)" columns for Atom #2 are circled in red and labeled with a red "2".

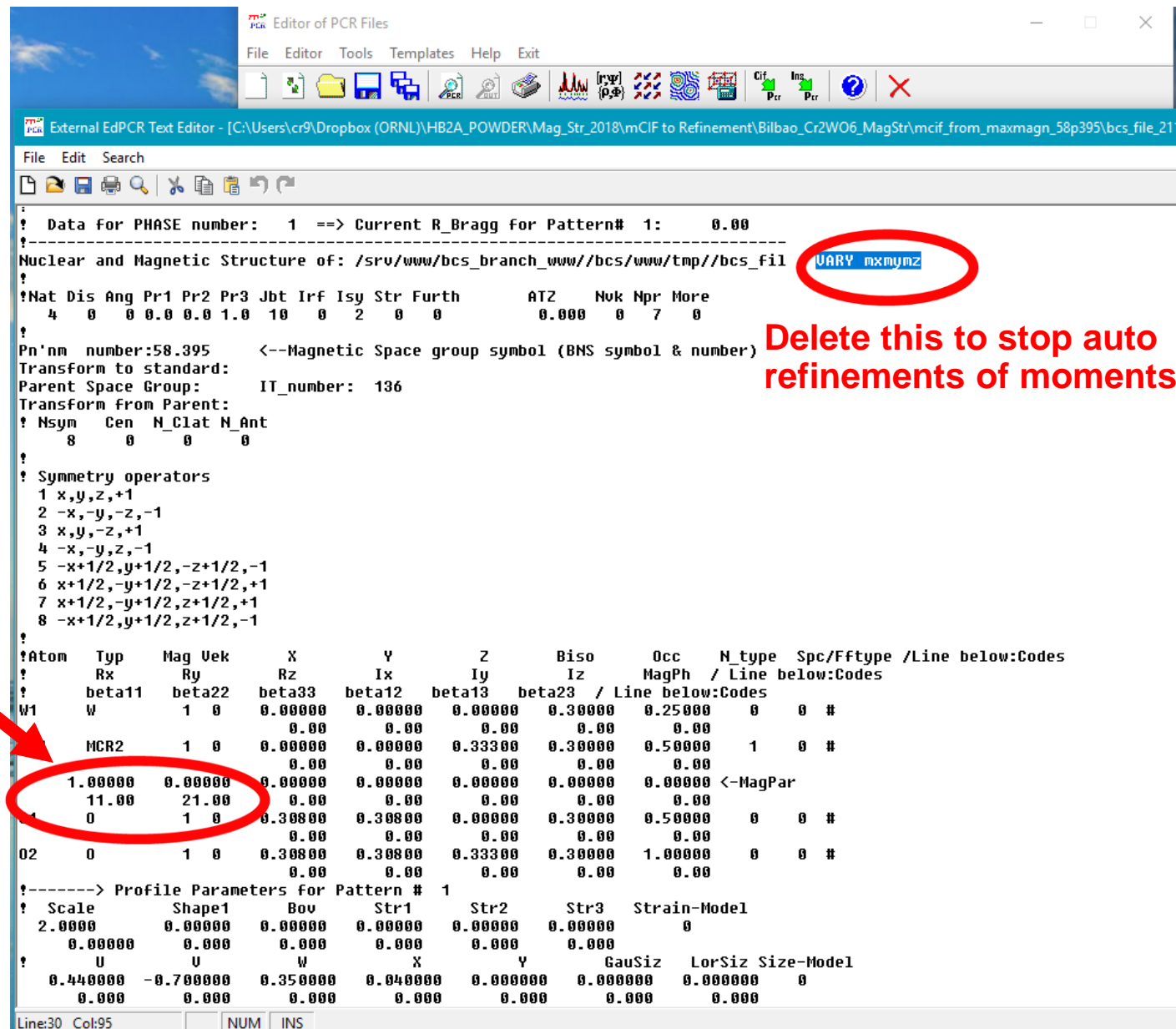
Atom #	Label	Ntyp	Mag. Rot.	Prog. V...	X	Y	Z	B	Occ
Atom # 1	W1	W	1	0	0.00000	0.00000	0.00000	0.30000	0.25000
Atom # 2	Cr1	MCR2	1	0	0.00000	0.00000	0.33300	0.30000	0.50000
Atom # 3	O1	O	1	0	0.30800	0.30800	0.00000	0.30000	0.50000
Atom # 4	O2	O	1	0	0.30800	0.30800	0.33300	0.30000	1.00000

	Re(x)	Re(y)	Re(z)	Im(x)	Im(y)	Im(z)	MPhase
Atom #2	1.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000

Below the atom table, there is a section for refinement parameters (B11/F1, B22/F2, B33/F3, B12/F4, B13/F5, B23/F6, F7) with checkboxes for each.

Step 3: Refining the magnetic structure with Fullprof

- Open pcr text file.
- Delete “VARY mxmymz” to turn off automatically refining moments.
- Turn off the refinement labels 11.0 and 21.0.



Editor of PCR Files

File Editor Tools Templates Help Exit

External EdPCR Text Editor - [C:\Users\cr9\Dropbox (ORNL)\HB2A_POWDER\Mag_Str_2018\mCIF to Refinement\Bilbao_Cr2WO6_MagStr\mcif_from_maxmagn_58p395\bc_file_211

File Edit Search

! Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 0.00

!-----

Nuclear and Magnetic Structure of: /srv/www/bcs_branch/www/bcs/www/tmp/bcs_fil

! VARY mxmymz

! Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth ATZ Nuk Npr More

! 4 0 0 0.0 0.0 1.0 10 0 2 0 0 0.000 0 7 0

! Pn'nm number:58.395 <--Magnetic Space group symbol (BNS symbol & number)

! Transform to standard: IT_number: 136

! Parent Space Group: Transform from Parent:

! Nsym Cen N_Clat N_Ant

! 8 0 0 0

! Symmetry operators

! 1 x,y,z,+1

! 2 -x,-y,-z,-1

! 3 x,y,-z,+1

! 4 -x,-y,z,-1

! 5 -x+1/2,y+1/2,-z+1/2,-1

! 6 x+1/2,-y+1/2,-z+1/2,+1

! 7 x+1/2,-y+1/2,z+1/2,+1

! 8 -x+1/2,y+1/2,z+1/2,-1

! Atom Typ Mag Ueq X Y Z Biso Occ N_type Spc/Fftype /Line below:Codes

! Rx Ry Rz Ix Iy Iz MagPh / Line below:Codes

! beta11 beta22 beta33 beta12 beta13 beta23 / Line below:Codes

! W1 W 1 0 0.00000 0.00000 0.00000 0.30000 0.25000 0 0 #

! HCR2 1 0 0.00000 0.00000 0.33300 0.30000 0.50000 1 0 #

! 1.00000 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 <--MagPar

! 11.00 21.00 0.00 0.00 0.00 0.00 0.00

! 0 1 0 0.30800 0.30800 0.00000 0.30000 0.50000 0 0 #

! 02 0 1 0 0.30800 0.30800 0.33300 0.30000 1.00000 0 0 #

!-----> Profile Parameters for Pattern # 1

! Scale Shape1 Bw Str1 Str2 Str3 Strain-Model

! 2.0000 0.00000 0.0000 0.00000 0.00000 0.00000 0

! 0.00000 0.000 0.000 0.000 0.000 0.000

! U V W X Y GauSiz LorSiz Size-Model

! 0.440000 -0.700000 0.350000 0.040000 0.000000 0.000000 0.000000 0

! 0.000 0.000 0.000 0.000 0.000 0.000 0.000

Line:30 Col:95 NUM INS

Delete this to stop auto refinements of moments

Step 3: Refining the magnetic structure with Fullprof

- Change to values circled to set up pcr file for refinement of crystal structure only first.
- **[1]** NCY → number of refinement cycles
- **[2]** Refine background
- **[3]** Make values under MCR2 zero to add no moment (only crystal structure)
- **[4]** Refine scale, **[5]** a,b,c lattice constants
- Set peak profile values for **[6]** U,V,W,X and **[7]** S_L, D_L. Do not refine these yet since lattice constants may be off (i.e. peak positions not correct).

```

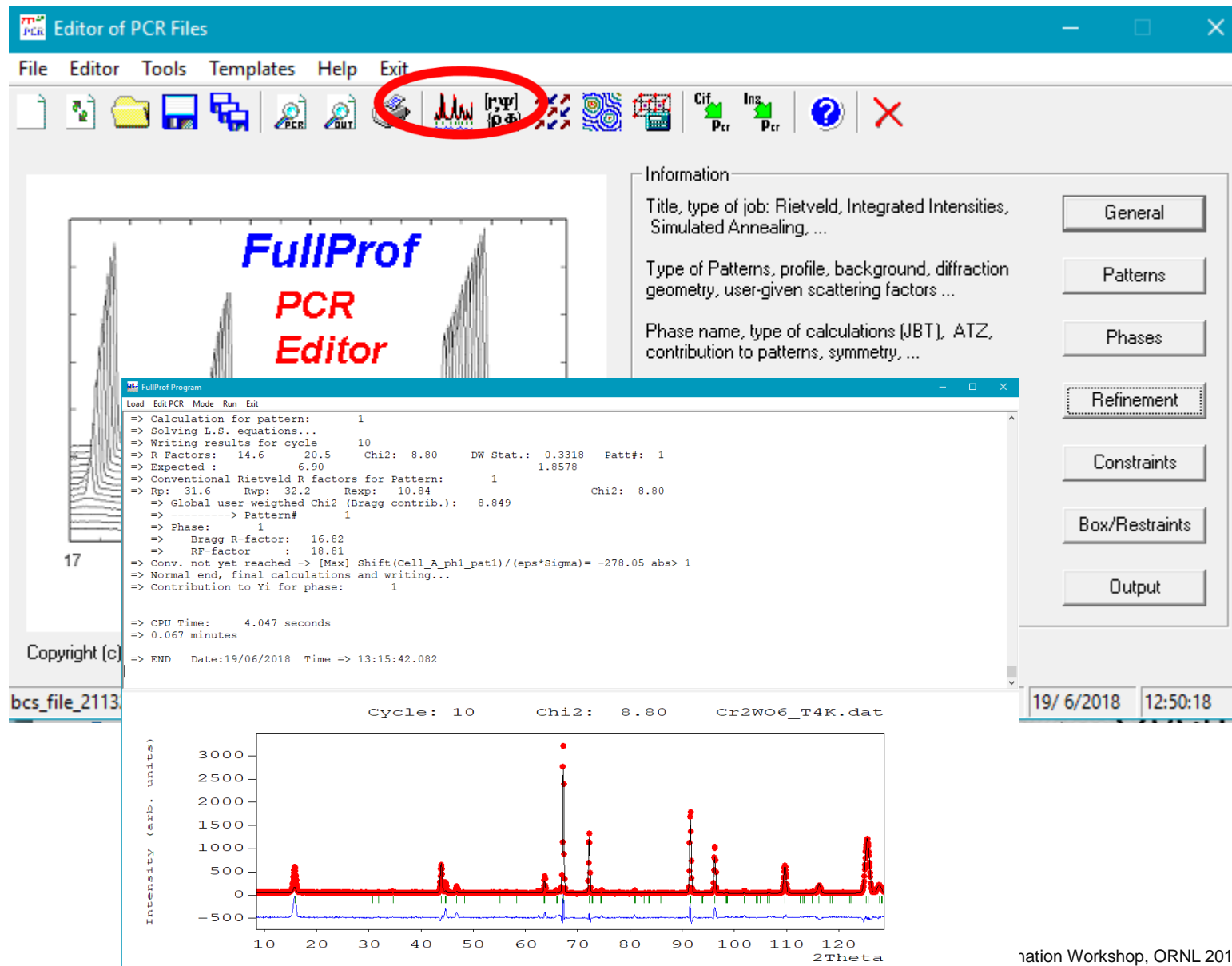
! Lambda1 Lambda2 Ratio Bkpos Wdt Cthm muR AsyLim Rpolaz 2nd-muR -> Patt# 1
2.410000 2.410000 1.00000 60.000 15.0000 0.0000 0.0000 180.00 0.0000 0.0000
! NCY ps R_at R_an R_pr R_gl Thmin Step Thmax PSD Sent0
10 0.02 1.00 1.00 1.00 1.00 8.5000 0.069037 128.6940 0.000 0.000
1 ! Excluded regions (LowT HighT) for Pattern# 1
0.00 2.00
130.00 180.00
!
!
4 !Number of refined parameters
!
! Zero Code SyCos Code SySin Code Lambda Code MORE ->Patt# 1
0.23000 0.0 0.00000 0.0 0.00000 0.0 0.00000 0.00 0
! Background coefficients/codes for Pattern# 1 (Polynomial of 6th degree)
50.000 0.0100 0.010 0.000 0.000 0.000
1 1 1 0.00 0.00
-----
! Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 0.00
!
Nuclear and Magnetic Structure of: /srv/www/bcs_branch/www/bcs/www/tmp/bcs_fil
!
! Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth ATZ Nuk Npr More
4 0 0 0.0 0.0 1.0 10 0 2 0 0 767.677 0 7 0
!
Pn'mn number:58.395 <--Magnetic Space group symbol (BNS symbol & number)
Transform to standard:
Parent Space Group: IT_number: 136
Transform from Parent:
! Nsym Cen N_Clat N_Ant
8 0 0 0
!
! Symmetry operators
1 x,y,z,+1
2 -x,-y,-z,-1
3 x,y,-z,+1
4 -x,-y,z,-1
5 -x+1/2,y+1/2,-z+1/2,-1
6 x+1/2,-y+1/2,-z+1/2,+1
7 x+1/2,-y+1/2,z+1/2,+1
8 -x+1/2,y+1/2,z+1/2,-1
!
! Atom Typ Mag Vek X Y Z Biso Occ N_type Spc/Fftype /Line t
! beta11 beta22 beta33 beta12 beta13 beta23 / Line below:Codes
W1 W 1 0 0.00000 0.00000 0.00000 0.30000 0.25000 0 0 #
Cr1 MCR2 1 0 0.00000 0.00000 0.33300 0.30000 0.50000 1 0 #
1.00000 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 <--MagPar
0.00 0.00 0.00 0.00 0.00 0.00 0.00
01 0 1 0 0.30000 0.30000 0.00000 0.30000 0.50000 0 0 #
0.00 0.00 0.00 0.00 0.00 0.00 0.00
02 0 1 0 0.30000 0.30000 0.33300 0.30000 1.00000 0 0 #
0.00 0.00 0.00 0.00 0.00 0.00 0.00
!-----> Profile Parameters for Pattern # 1
! Scale Shape1 Bou Str1 Str2 Str3 Strain-Model
2.0000 0.00000 0.00000 0.00000 0.00000 0.00000 0
21.00000 0.000 0.000 0.000
! U V W X Y GauSiz LorSiz Size-Model
0.440000 -0.700000 0.350000 0.040000 0.000000 0.000000 0.000000 0
0.000 0.000 0.000 0.000 0.000 0.000 0.000
! a b c alpha beta gamma #Cell Info
4.571000 4.571000 8.853001 90.000000 90.000000 90.000000 #
1 1 1 0.00000 0.00000 0.00000
! Pref1 Pref2 Asy1 Asy2 Asy3 Asy4 S_L D_L
1.00000 0.00000 0.00000 0.00000 0.00000 0.00000 0.03300 0.03300
0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00

```

Step 3: Refining the magnetic structure with Fullprof

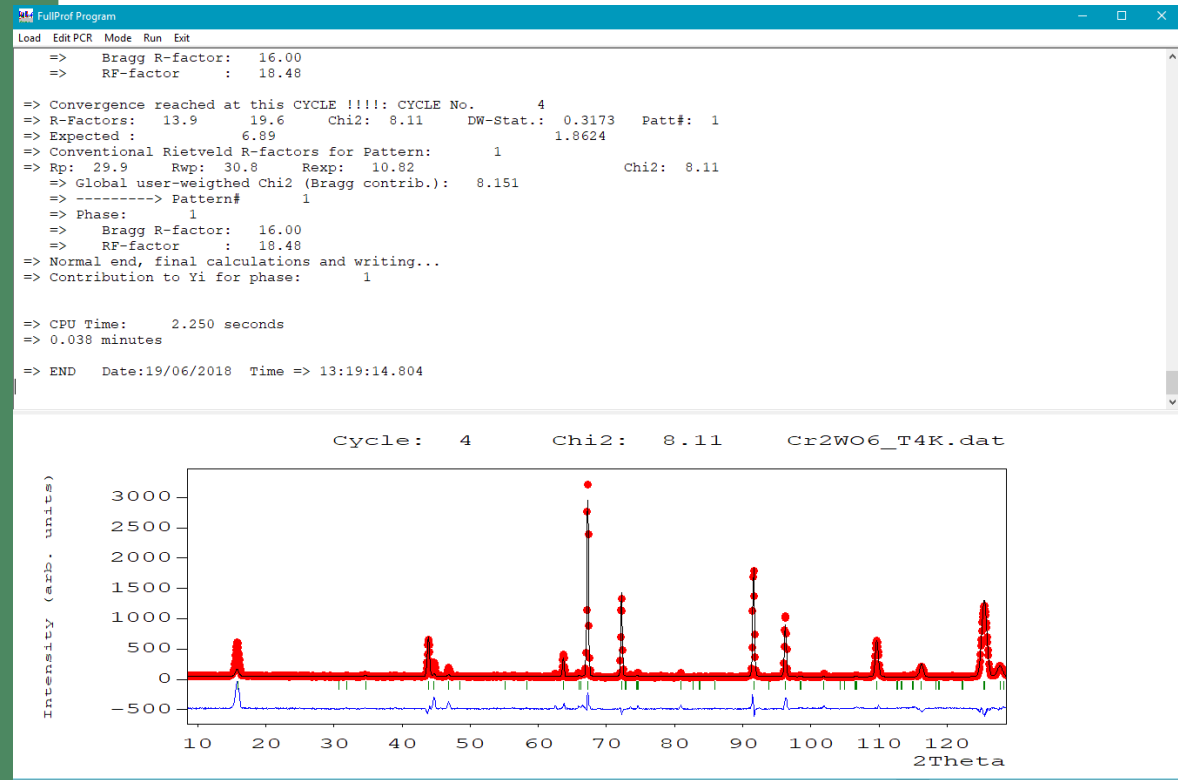
- Run the refinement.
- Select the “Cr2WO6_T4K.dat” neutron data file.
- Refinement captures most of the data well.

Cr2WO6_T4K.dat



Step 3: Refining the magnetic structure with Fullprof

- **[1]** Select the U,V,W,X to refine
- This improves refinement and leads to convergence.



```
? Data for PHASE number: 1 ==> Current R_Bragg for Pattern# 1: 0.00
-----
Nuclear and Magnetic Structure of: /srv/www/bcs_branch_www//bcs/www/tmp//bcs_fil
?
?Nat Dis Ang Pr1 Pr2 Pr3 Jbt IrF Isy Str Furth ATZ Nvk Npr More
4 0 0 0.0 0.0 1.0 10 0 2 0 0 767.677 0 7 0
?
Pn'nm number:58.395 <--Magnetic Space group symbol (BNS symbol & number)
Transform to standard:
Parent Space Group: IT_number: 136
Transform from Parent:
? Nsym Cen N_Clat N_Ant
8 0 0 0
?
? Symmetry operators
1 x,y,z,+1
2 -x,-y,-z,-1
3 x,y,-z,+1
4 -x,-y,z,-1
5 -x+1/2,y+1/2,-z+1/2,-1
6 x+1/2,-y+1/2,-z+1/2,+1
7 x+1/2,-y+1/2,z+1/2,+1
8 -x+1/2,y+1/2,z+1/2,-1
?
?Atom Typ Mag Uek X Y Z Biso Occ N_type Spc/Fftype
? Rx Ry Rz Ix Iy Iz / Line below:Codes
? beta11 beta22 beta33 beta12 beta13 beta23 / Line below:Codes
W1 W 1 0 0.00000 0.00000 0.00000 0.30000 0.25000 0 0 #
Cr1 MCR2 1 0 0.00000 0.00000 0.33300 0.30000 0.50000 1 0 #
1.00000 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 <--MagPar
0 0.00 0.00 0.00 0.00 0.00 0.00
01 0 1 0 0.30800 0.30800 0.00000 0.30000 0.50000 0 0 #
0.00 0.00 0.00 0.00 0.00 0.00
02 0 1 0 0.30800 0.30800 0.33300 0.30000 1.00000 0 0 #
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
1.5720 0.00000 0.00000 0.00000 0.00000 0.00000 0
21.00000 0.000 0.000 0.000 0.000 0.000
U U W X Y GauSiz LorSiz Size-Model
0.440000 -0.700000 0.350000 0.040000 0.000000 0.000000 0.000000 0
1 1 1 1 0.000 0.000 0.000
? h k l alpha beta gamma #Cell Info
4.582274 4.586748 8.856397 90.000000 90.000000 90.000000 #
61.00000 31.00000 51.00000 0.00000 0.00000 0.00000
? Pref1 Pref2 Asy1 Asy2 Asy3 Asy4 S_L D_L
1.00000 0.00000 0.00000 0.00000 0.00000 0.00000 0.03300 0.03300
0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00
? 2Th1/TOF1 2Th2/TOF2 Pattern to plot
8.500 128.694 1
```

Step 3: Refining the magnetic structure with Fullprof

- [1] Can now select the magnetic moment to refine. (Either in GUI or text file)

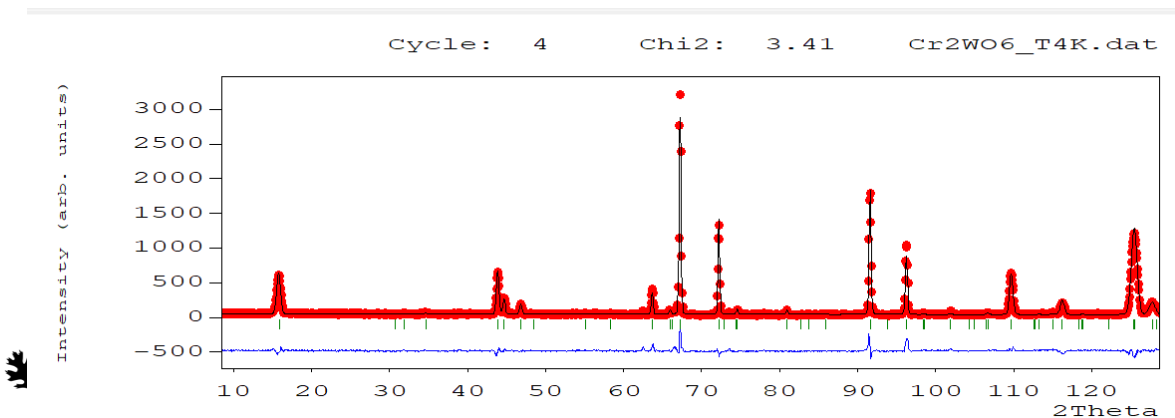
All peaks now fit well

```
FullProf Program
Load Edit PCR Mode Run Exit
=> Bragg R-factor: 9.668
=> RF-factor : 14.36

=> Convergence reached at this CYCLE !!!!: CYCLE No.      4
=> R-Factors: 9.93 12.7 Chi2: 3.41 DW-Stat.: 0.6310 Patt#: 1
=> Expected : 6.89 1.8636
=> Conventional Rietveld R-factors for Pattern: 1
=> Rp: 20.9 Rwp: 20.0 Rexp: 10.80 Chi2: 3.41
=> Global user-weighted Chi2 (Bragg contrib.): 3.418
=> -----> Pattern# 1
=> Phase: 1
=> Bragg R-factor: 9.668
=> RF-factor : 14.36
=> Normal end, final calculations and writing...
=> Contribution to Yi for phase: 1

=> CPU Time: 1.938 seconds
=> 0.032 minutes

=> END Date:19/06/2018 Time => 13:22:15.777
```



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)

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...

Refinement Information

Cycles of Refinement: 10

Stop Criterion of Convergence

Forced Termination when shifts < 0.02 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

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

Refinement weighting model

OK Atoms Prop. Vectors

Atoms Information: Phase 1

List of Atoms

Number of Atoms: 4

Atom #	Label	Ntyp	Mag. Rot.	Prog. V...	X	Y	Z	B	Occ
Atom # 1	W1	W	1	0	0.00000	0.00000	0.00000	0.30000	0.25000
Atom # 2	Cr1	MCR2	1	0	0.00000	0.00000	0.33300	0.30000	0.50000
Atom # 3	O1	O	1	0	0.30800	0.30800	0.00000	0.30000	0.50000
Atom # 4	O2	O	1	0	0.30800	0.30800	0.33300	0.30000	1.00000

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

1

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

Refine Positions

Refine B_iso

Refine B_aniso

Fix All

Cancel

OK

Step 3: Refining the magnetic structure with Fullprof

- Check magnetic structure in .fst file
- Check moment size is reasonable ($\sim 2\mu_B/\text{Cr}$)
- Refinement can be further improved by altering the thermal parameters, asymmetry, more background, atomic positions, zero, etc.

