

# From mCIF to refinement: Using the Bilbao Crystallographic Server and fullprof to refine $\text{Cr}_2\text{WO}_6$ data

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

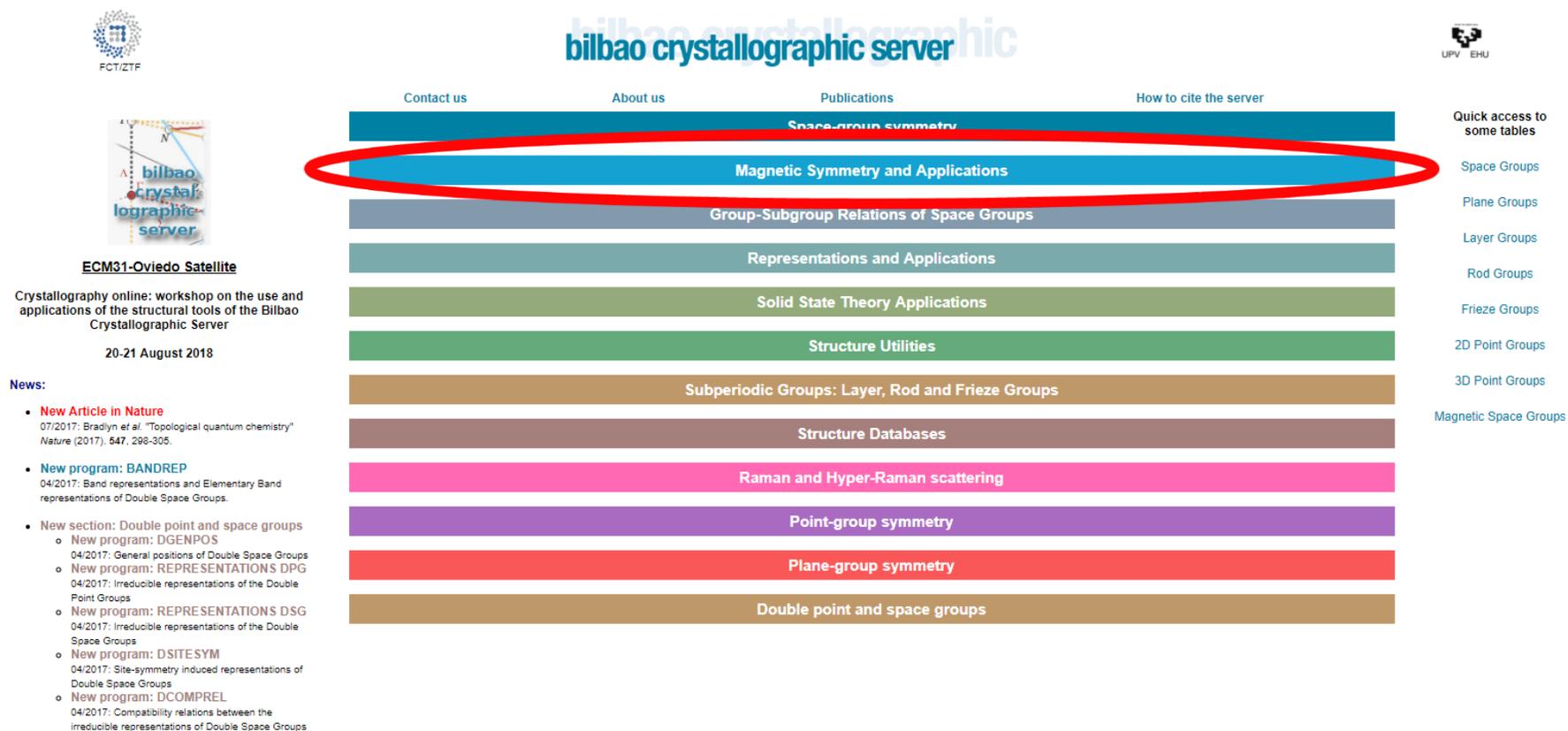
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# Cr<sub>2</sub>WO<sub>6</sub>

- There is a Cr<sub>2</sub>WO<sub>6</sub> 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<sub>2</sub>WO<sub>6</sub> 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



The screenshot shows the Bilbao Crystallographic Server homepage. The navigation menu includes: Contact us, About us, Publications, and How to cite the server. A dropdown menu is open, listing various categories: Space-group symmetry, **Magnetic Symmetry and Applications** (highlighted with a red oval), 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, and Double point and space groups. On the right side, there is a 'Quick access to some tables' section with links to Space Groups, Plane Groups, Layer Groups, Rod Groups, Frieze Groups, 2D Point Groups, 3D Point Groups, and Magnetic Space Groups. On the left side, there is a logo for FCT/ZTF, a logo for the Bilbao Crystallographic Server, and a news section titled 'ECM31-Oviedo Satellite' with a date of 20-21 August 2018. Below the news section, there are several news items:

**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

# Step 1: Creating mCIF file

- Then select “MAXMAGN”

Contact us      About us      Publications      How to cite the server

## Space-group symmetry

### Magnetic Symmetry and Applications

<a href="#">MGENPOS</a>	General Positions of Magnetic Space Groups
<a href="#">MWYCKPOS</a>	Wyckoff Positions of Magnetic Space Groups
<a href="#">MNORMALIZER</a>	Normalizers of Magnetic Space Groups
<a href="#">IDENTIFY MAGNETIC GROUP</a>	Identification of a Magnetic Space Group from a set of generators in an arbitrary setting
<a href="#">BNS2OG</a> 	Transformation of symmetry operations between BNS and OG settings
<a href="#">mCIF2PCR</a> 	Transformation from mCIF to PCR format (FullProf).
<a href="#">MPOINT</a> 	Magnetic Point Group Tables
<a href="#">MAGNEXT</a>	Extinction Rules of magnetic Space Groups
<a href="#">MAXMAGN</a> 	Maximal magnetic space groups for a given space group and a propagation vector
<a href="#">MAGMODELS</a>	Magnetic structure models for any given magnetic symmetry
<a href="#">k-SUBGROUPSMAG</a> 	Magnetic subgroups consistent with some given propagation vector(s) or a supercell
<a href="#">MAGNDATA</a> 	A collection of magnetic structures with transportable cif-type files
<a href="#">MVISUALIZE</a> 	3D Visualization of magnetic structures with Jmol
<a href="#">MTENSOR</a> 	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  $\text{Cr}_2\text{WO}_6$ ). For  $\text{Cr}_2\text{WO}_6$  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 constrains for the different possible symmetries and cif-like files can be produced. These files permit the different alternative models to be analyzed, refined, shown graphically, transported to ab-initio codes etc., with programs as ISODISTORT, JANA2006, StrConvert, VESTA, etc. These cif-like files can also be submitted to the program 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. Eloroo, 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

Submit

1



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

# 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<sub>2</sub>/mnm*)

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

2

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

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 <sub>x</sub> , M <sub>y</sub> , M <sub>z</sub>
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 <sub>x</sub> ,m <sub>y</sub> ,0) (1/2,1/2,z+1/2   -m <sub>x</sub> ,m <sub>y</sub> ,0) (1/2,1/2,-z+1/2   m <sub>x</sub> ,-m <sub>y</sub> ,0) (0,0,-z   -m <sub>x</sub> ,-m <sub>y</sub> ,0)	4	(M <sub>x</sub> ,M <sub>y</sub> ,0)	M <sub>x</sub> = 1 M <sub>y</sub> = 0.00000
3	O1 O 0.30800 0.30800 0.00000	(x,x,0   0,0,m <sub>z</sub> ) (-x,-x,0   0,0,-m <sub>z</sub> ) (-x+1/2,x+1/2,1/2   0,0,m <sub>z</sub> ) (x+1/2,-x+1/2,1/2   0,0,-m <sub>z</sub> )	4	-	-
4	O2 O 0.30800 0.30800 0.33300	(x,x,z   m <sub>x</sub> ,m <sub>y</sub> ,m <sub>z</sub> ) (-x,-x,z   m <sub>x</sub> ,m <sub>y</sub> ,-m <sub>z</sub> ) (-x+1/2,x+1/2,z+1/2   -m <sub>x</sub> ,m <sub>y</sub> ,m <sub>z</sub> ) (x+1/2,-x+1/2,z+1/2   -m <sub>x</sub> ,m <sub>y</sub> ,-m <sub>z</sub> ) (-x+1/2,x+1/2,-z+1/2   m <sub>x</sub> ,-m <sub>y</sub> ,m <sub>z</sub> ) (x+1/2,-x+1/2,-z+1/2   m <sub>x</sub> ,-m <sub>y</sub> ,-m <sub>z</sub> ) (x,x,-z   -m <sub>x</sub> ,-m <sub>y</sub> ,m <sub>z</sub> ) (-x,-x,-z   -m <sub>x</sub> ,-m <sub>y</sub> ,-m <sub>z</sub> )	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 MVISUALIZE”

## mCIF file of the structure

Submit this mcif file to MVISUALIZE for 3D visualization of the estructure using Jmol

**2** [Submit to MVISUALIZE](#)

**1** [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_5y0htAoR
_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: 3D Visualization of magnetic structures with Jmol

MVISUALIZE Main Page

Show/Hide File

The screenshot shows the MVISUALIZE web interface. The main window displays a 3D visualization of a magnetic structure within a unit cell. The structure consists of blue spheres (atoms) and grey arrows (magnetic moments) arranged in a lattice. The unit cell axes are labeled a, b, and c. The interface includes a toolbar with various controls for viewing and manipulating the structure.

Toolbar controls include:

- Select cell... (dropdown)
- Toggle Parent Cell
- Toggle Standard Cell
- Export PNG Image
- Save PNG-3D
- Save ZIP file
- View Along Axis... (dropdown)
- Unit Cell Info
- Show unit cell a,b,c
- Add 1 cell along x
- Remove 1 cell along x
- Add 1 cell along y
- Remove 1 cell along y
- Add 1 cell along z
- Remove 1 cell along z
- All / Magnetic Atoms
- Show/Hide Labels
- Larger / Smaller (for Vectors and Atoms)
- Window Size (with directional arrows)
- Bigger / Smaller
- Background Color (dropdown)
- Toggle Quality
- Center
- X=1, Y=1, Z=1 (input fields)
- Choose supercell
- Draw bonds & polyhedra (with Join and with dropdowns)
- from 0.75 to 2.75 Å (range input)
- Draw Bonds Polyhedra
- Delete Bonds Polyhedra
- Clear all drawings

At the bottom of the interface, there are buttons for help, console, and Execute.

# Step 2: Creating a pcr from an mcif file

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

Contact us      About us      Publications      How to cite the server

**Space-group symmetry**

**Magnetic Symmetry and Applications**

<a href="#">MGENPOS</a>	General Positions of Magnetic Space Groups
<a href="#">MWYCKPOS</a>	Wyckoff Positions of Magnetic Space Groups
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**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 2

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

The file has been successfully converted.

Click to download it

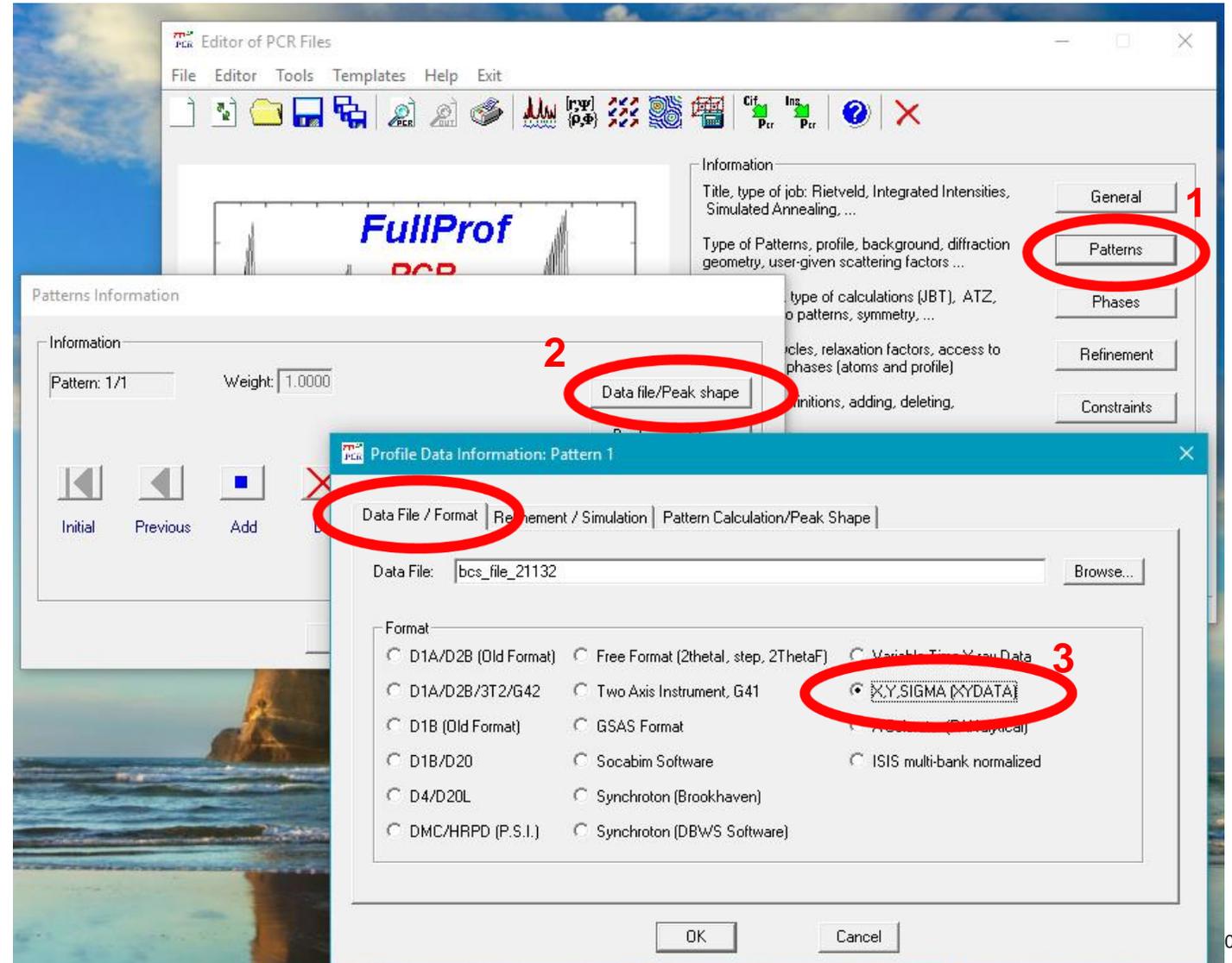
- This will convert the file and “Click to download it” will download the .pcr file
- We will use this .pcr file in Step 3. Put it in the same folder as the data file “Cr2WO6\_T4K.dat”

# 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.

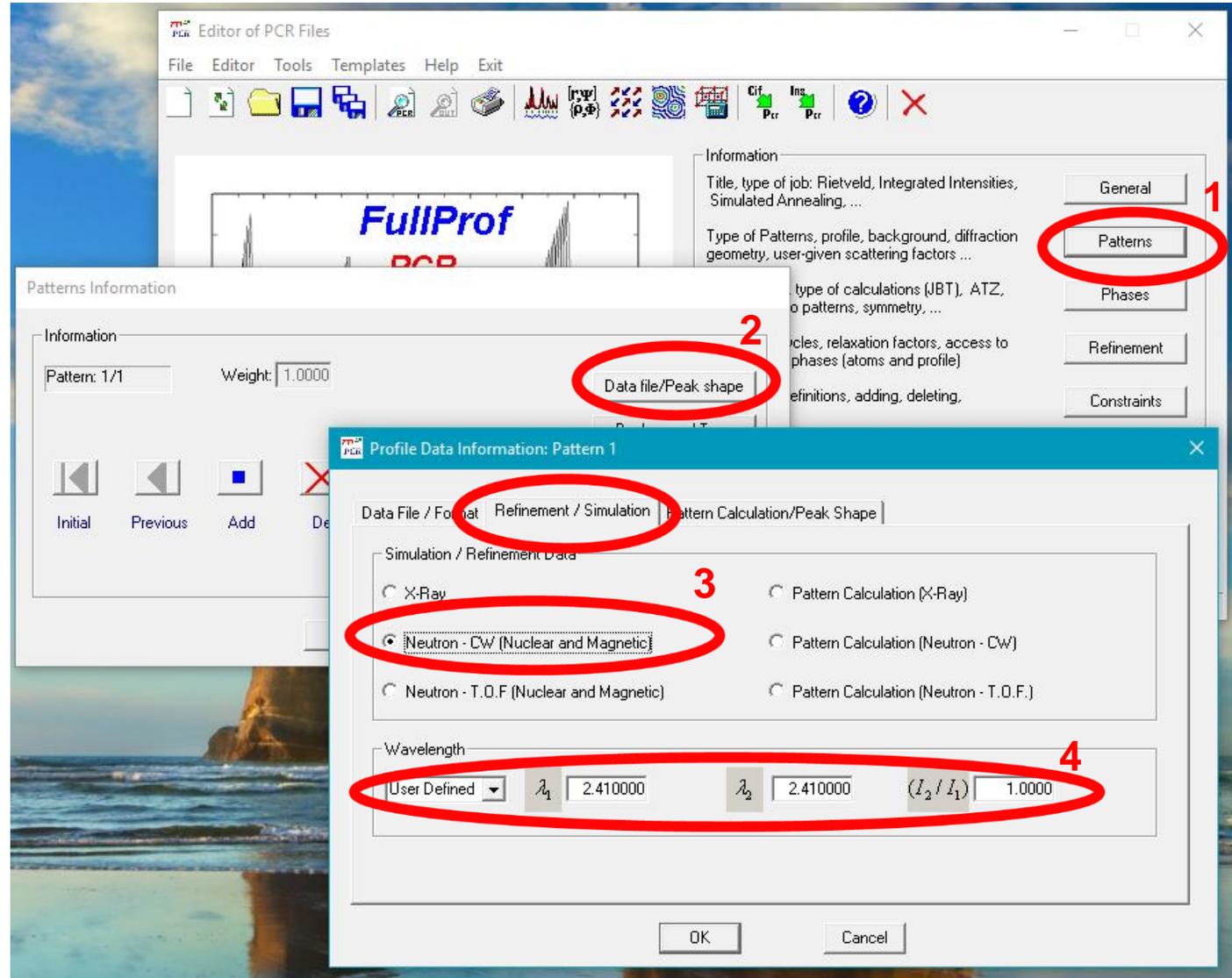
# Step 3: Refining the magnetic structure with Fullprof

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



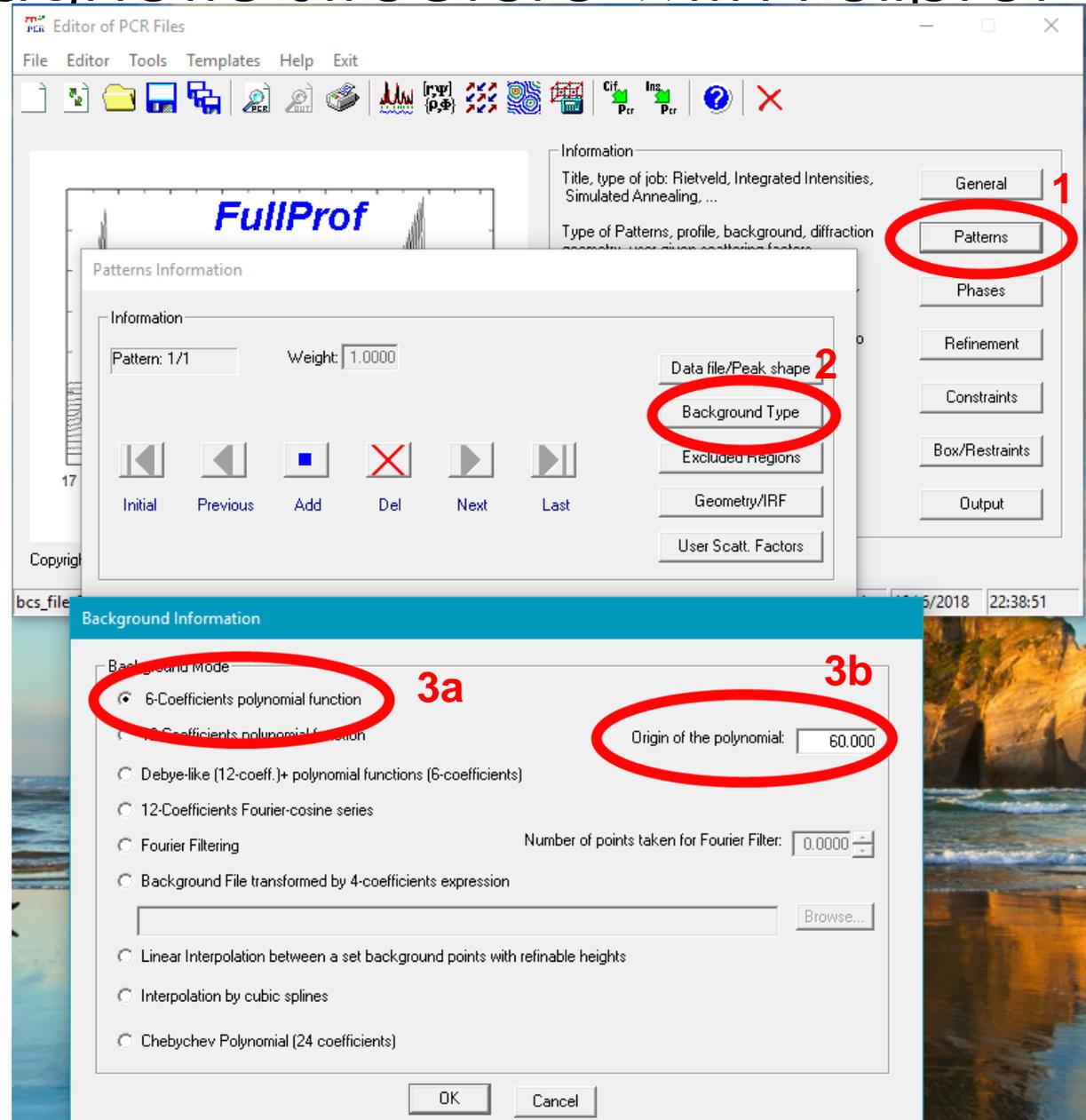
# Step 3: Refining the magnetic structure with Fullprof

- 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) (constant wavelength)
- Peak shape should be “Thompson-Cox...”

The screenshot displays the Fullprof software interface. The 'Pattern Contribution Information for Phase 1' dialog box is open, showing the 'Type of Pattern' section with 'Neutron (Constant Wavelength)' selected. The 'Peak shape' dropdown is set to 'Thompson-Cox-Hastings pseudo-Voigt \* Axial divergence asymmetry'. The 'Information' dialog box is also open, showing the 'Contribution to Patterns' button circled in red. The sidebar on the right has the 'Phases' button circled in red. The background shows a scenic view of a beach with waves and a rock formation.

# Step 3: Refining the magnetic structure with Fullprof

- Setting starting values for refinements
- Starting background values

The screenshot displays the Fullprof software interface. The main window is titled 'Editor of PCR Files'. A 'Refinement Information' dialog box is open, showing various settings. A red circle labeled '1' highlights the 'Refinement' button in the right-hand sidebar. In the 'Refinement Information' dialog, the 'Background' button under the 'Refinement weighting model' section is circled in red and labeled '2'. Below this, a '6 Coefficients Polynomial Background: Pattern 1' dialog box is open, showing a table of coefficients. A red circle labeled '3' highlights the 'd\_0' coefficient field, which contains the value '50.000'. The table has 6 columns (d\_0 to d\_5) and 4 rows of coefficient fields. The 'd\_0' field is highlighted with a red circle.

Refinement Information dialog box settings:

- Cycles of Refinement: 1
- Stop Criterion of Coverage: 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
- Refinement weighting model: Background (selected)
- Reduction factor of number of data points: 0

6 Coefficients Polynomial Background: Pattern 1 dialog box table:

	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
Coefficients						
Coefficients						
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.

Refinement Information

Cycles of Refinement: 1

Stop Criterium of Coverage  
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 | 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-Absorption

Reduction factor of number of data points: 0

Instrumental Parameters Refinement: Pattern 1

	Zero	Displacement	Transparency	Wavelength
Coefficients	0.230000	0.000000	0.000000	0.000000

# 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 diffraction patterns with the text "FullProf PCR Editor" overlaid. The "Refinement Information" panel is visible, showing "Cycles of Refinement" set to 1. The "Profile Parameters: Phase 1 Pattern 1" dialog box is open, showing various parameters. The "Factors" section has "Scale" set to 2.0000 and "Overall B-factor" set to 0.0000. The "Cell Parameters" section shows lattice parameters a, b, c, alpha, beta, and gamma. The "FWHM / Shape Parameters" section has "U" set to 0.440000, "V" set to -0.700000, "W" set to 0.350000, and "IG" set to 0.000000. The "Shape Parameters" section has "X" set to 0.040000, "Y" set to 0.000000, and "SZ" set to 0.000000. The "Refinement Information" panel has the "Refinement" button circled in red with a "1" next to it. The "Profile Parameters" dialog box has the "Profile" button circled in red with a "2" next to it.

Information

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

General

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

Patterns

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

Phases

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

Refinement

Refinement Information

Cycles of Refinement: 1

Profile Parameters: Phase 1 Pattern 1

Factors

	Scale	Overall B-factor
Coefficients	2.0000	0.0000

Cell Parameters

	a	b	c	alpha	beta	gamma
Coefficients	4.571000	4.571000	8.853001	90.000	90.000	90.000

FWHM / Shape Parameters

	U	V	W	IG
Coefficients	0.440000	-0.700000	0.350000	0.000000

Shape Parameters

	X	Y	SZ
Coefficients	0.040000	0.000000	0.000000

Refine All

Fix All

Cancel

OK

Relaxation Factors for Shifts

Atomic 1.00 Anisotropic 1.00 Profile 1.00 Global 1.00

Bragg R-Factor excluding reflections limiting excluded regions

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

Atoms Prop. Vectors

Patterns

Profile

Micro-Structure

HKL Shifts Further Parameters

# Step 3: Refining the magnetic structure with Fullprof

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

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)

General

Patterns

Phases

Refinement

Refinement Information

Cycles of Refinement: 1

Stop Criterion of Coverage

Relaxation Factors for Shifts

1.00 Anisotropic 1.00 Profile 1.00 Global 1.00

Bragg R-Factor excluding reflections limiting excluded regions

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

Atoms Prop. Vectors

1 2 3 4 5 6 7

Profile Micro-Structure

HKL Shifts Further Parameters

Atoms Information: Phase 1

List of Atoms

Number of Atoms: 4

	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

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

# 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.

```
! 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 Nvk 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:
! 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 /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 #
! 0.00 0.00 0.00 0.00 0.00 0.00
! HCR2 1 0 0.00000 0.00000 0.33300 0.30000 0.50000 1 0 #
! 0.00 0.00 0.00 0.00 0.00 0.00
! 1.00000 0.00000 0.00000 0.00000 0.00000 0.00000 <--MagPar
! 11.00 21.00 0.00 0.00 0.00 0.00
! 0 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 Bwv Str1 Str2 Str3 Strain-Model
! 2.0000 0.00000 0.00000 0.00000 0.00000 0.00000 0
! 0.00000 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
! 0.000 0.000 0.000 0.000 0.000 0.000 0.000
```

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).

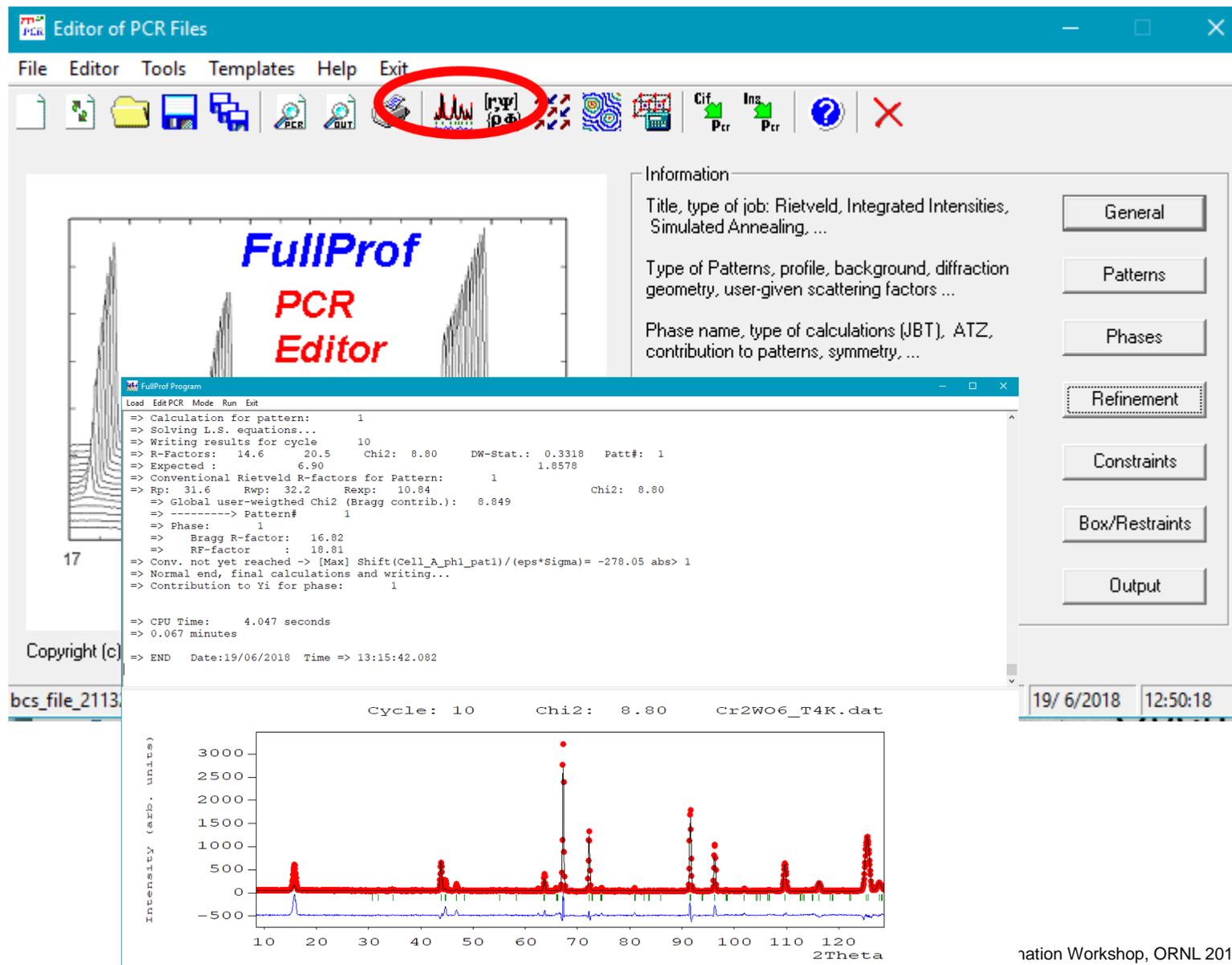
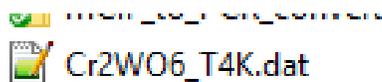
```

2.410000 2.410000 1.000000 60.000 15.0000 0.0000 0.0000 180.00 0.0000 0.0000
!NCY 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 0.000000 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 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'm 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 Ueq X Z Biso Occ N_type Spc/Fftype /Line t
! Rx Ry Rz Ix Iy Iz MagPh / Line below:Codes
W1 W 1 0 0.00000 0.00000 0.00000 0.30000 0.25000 0 0 #
! beta11 beta22 beta33 beta12 beta13 beta23 / Line below:Codes
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 <-MagPar
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
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
!-----> 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 0.000 0.000
! U W X Y GauSiz LorSiz Size-Model
0.440000 -0.700000 0.350000 0.040000 0.000000 0.000000 0
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

```

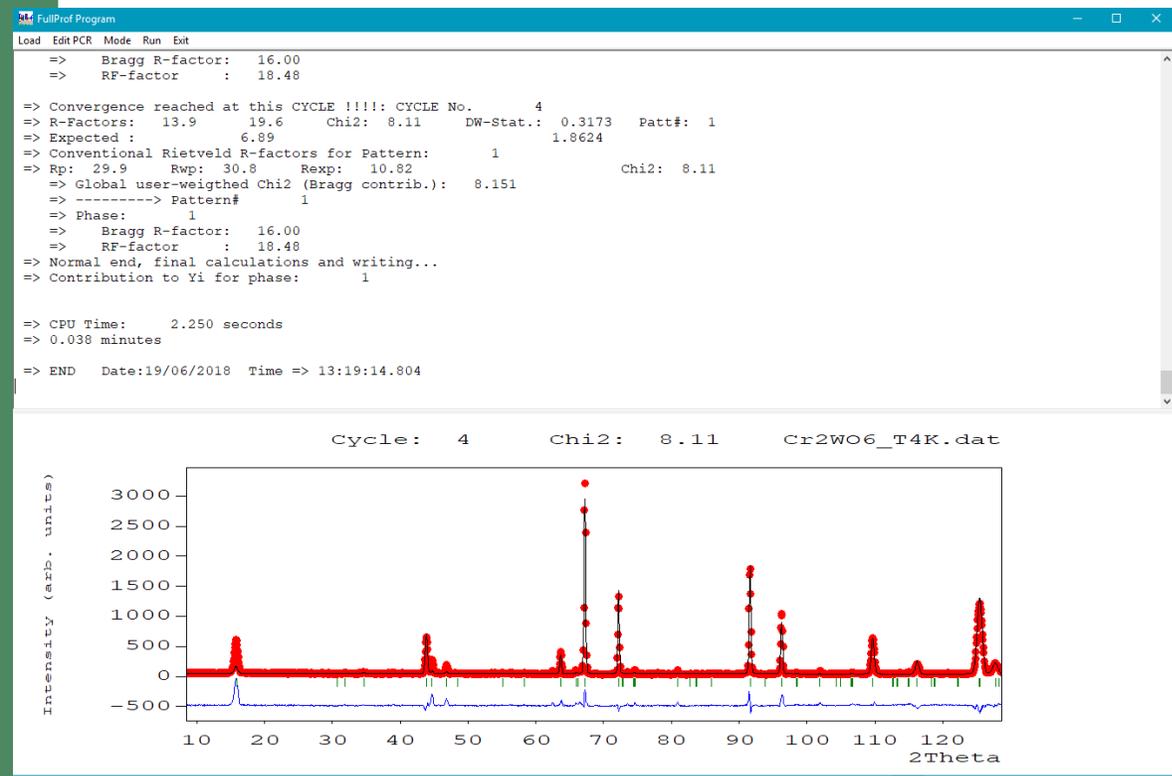
# Step 3: Refining the magnetic structure with Fullprof

- Run the refinement.
- Select the “Cr2WO6\_T4K.dat” neutron data file.
- Refinement captures most of the data well.



# Step 3: Refining the magnetic structure with Fullprof

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



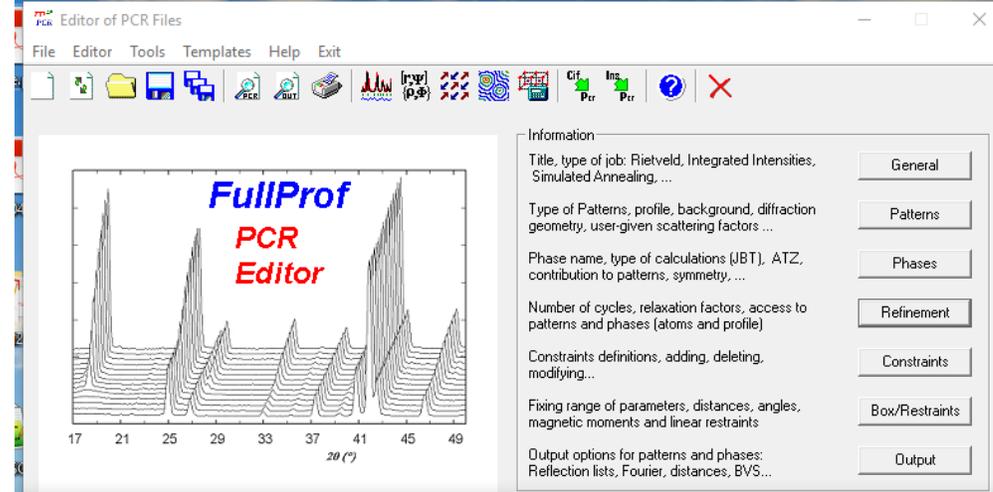
```

! 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 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 #
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 0.30000 0.30000 0.00000 0.30000 0.50000 0 0 #
  0.00 0.00 0.00 0.00 0.00 0.00
02 0 1 0 0.30000 0.30000 0.33300 0.30000 1.00000 0 0 #
  0.00 0.00 0.00 0.00
!-----> Profile Parameters for Pattern # 1
! Scale Shape1 B0v Str1 Str2 Str3 Strain-Model
1.5720 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 0
  21.00000 0.000 0.000 0.000 0.000 0.000 0.000
  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
  
```

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
    
```

## Atoms Information: Phase 1

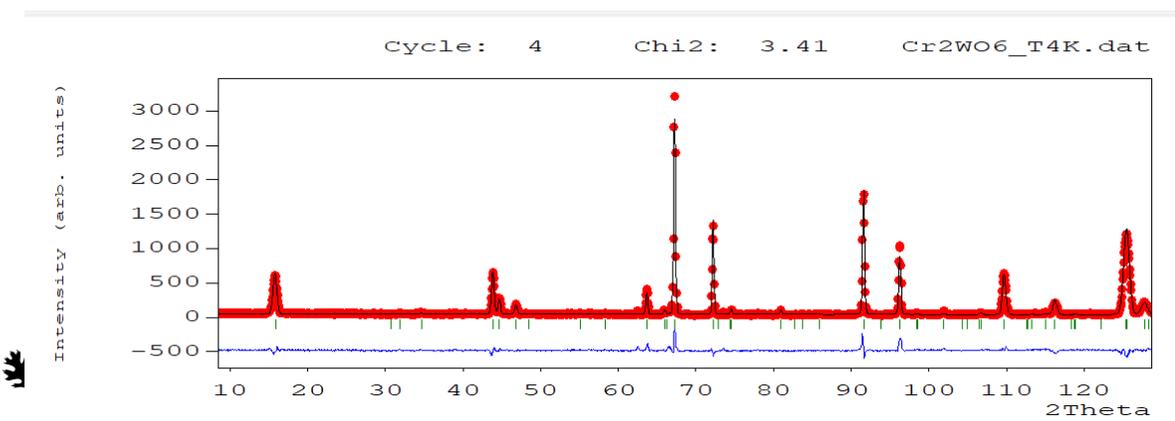
List of Atoms  
Number of Atoms: 4

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

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



# 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.

