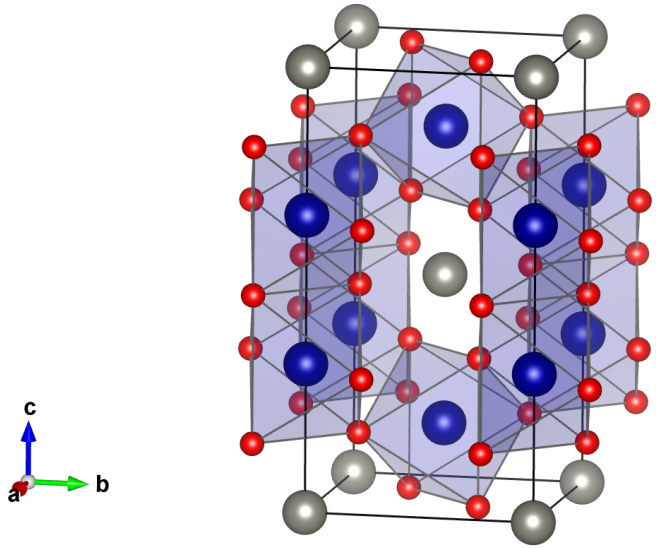
Example Cr2WO6 (magndata #0.75)
commensurate magnetic structure – modulation (propagation) vector: (0,0,0)

Neutron powder data measured at 4K on HB2A at HFIR

λ= 2.4097 A

References:
Zhu et al., PRL 113, 076406 (2014)
PHYSICAL REVIEW B **92**, 094419 (2015)

Input files: Cr2WO6_T4K.dat (powder profile data)
Cr2WO6.cif (nuclear structure)



Parent space group:

P4$_2$/mnm (N. 136)

Propagation vector:

k= (0,0,0)

Magnetic site:

Cr 4e (0,0,z)

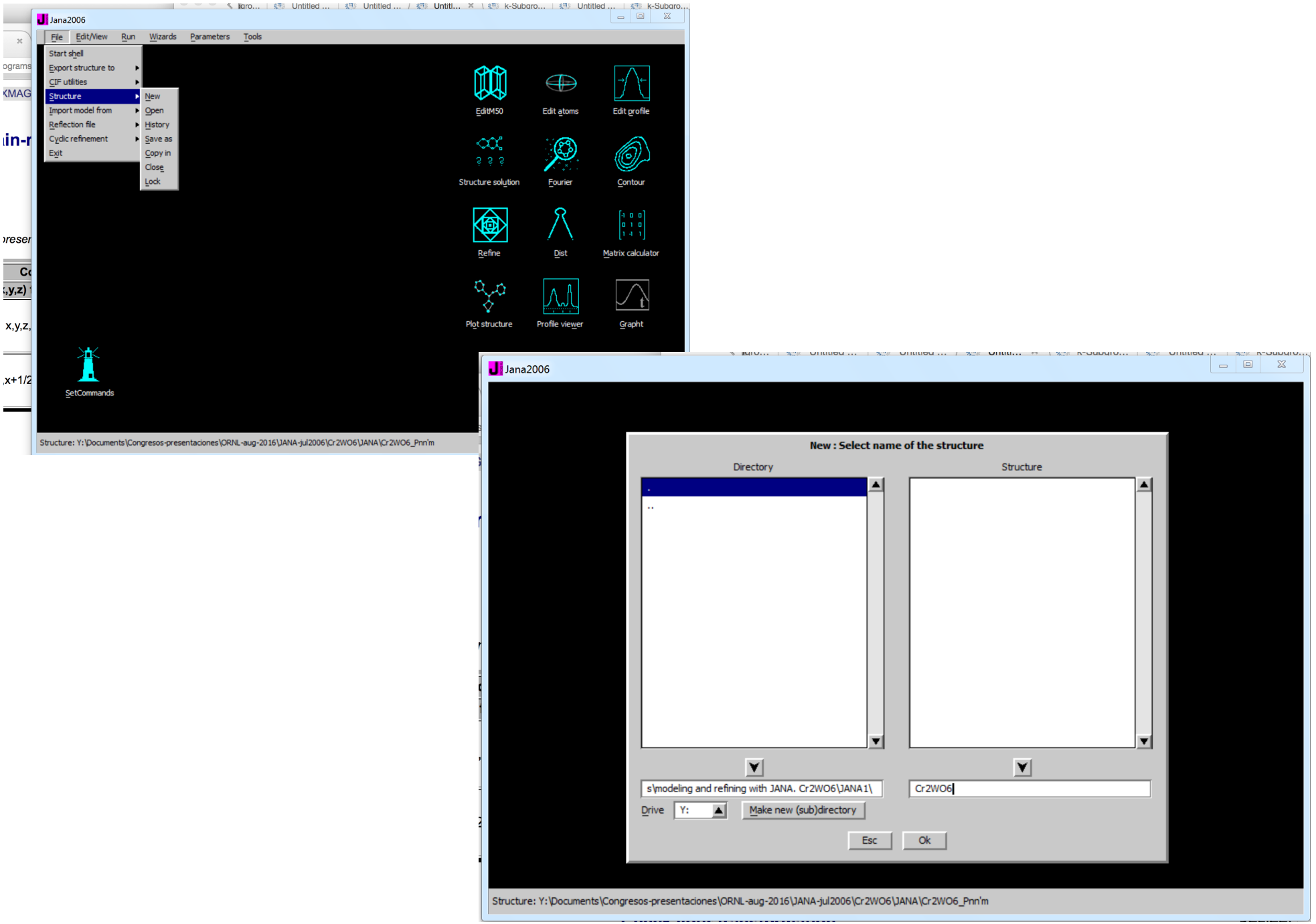
1. Creating new jobname

Start Jana2006

"**File → Structure → New**" opens a file manager

Left panel: locate directory with input files

Right panel: double-click at Cr2WO6


Jana2006
File
Edit/View
Run
Wizards
Parameters
Tools
Start shell
Export structure to
CIF utilities
Structure
Import model from
Reflection file
Cyclic refinement
Exit
New
Open
History
Save as
Copy in
Close
Lock
EditM50
Edit atoms
Edit profile
Structure solution
Fourier
Contour
Refine
Dist
Matrix calculator
Plot structure
Profile viewer
Grapht
SetCommands
Structure: Y:\Documents\Congresos-presentaciones\ORNL-aug-2016\JANA-jul2006\Cr2WO6\JANA\Cr2WO6_Pnn'm
Jana2006
New : Select name of the structure
Directory
Structure
s\modeling and refining with JANA. Cr2WO6\JANA1\
Cr2WO6
Drive
Y:
Make new (sub)directory
Esc
Ok
Structure: Y:\Documents\Congresos-presentaciones\ORNL-aug-2016\JANA-jul2006\Cr2WO6\JANA\Cr2WO6_Pnn'm

2. Import Wizard

Select "Magnetic parent structure: nuclear model from CIF"; NEXT

[On the screen: Select input CIF file]

Right panel: select the input file Cr2WO6.cif; OK

3. Define magnetic propagation vector and form factors

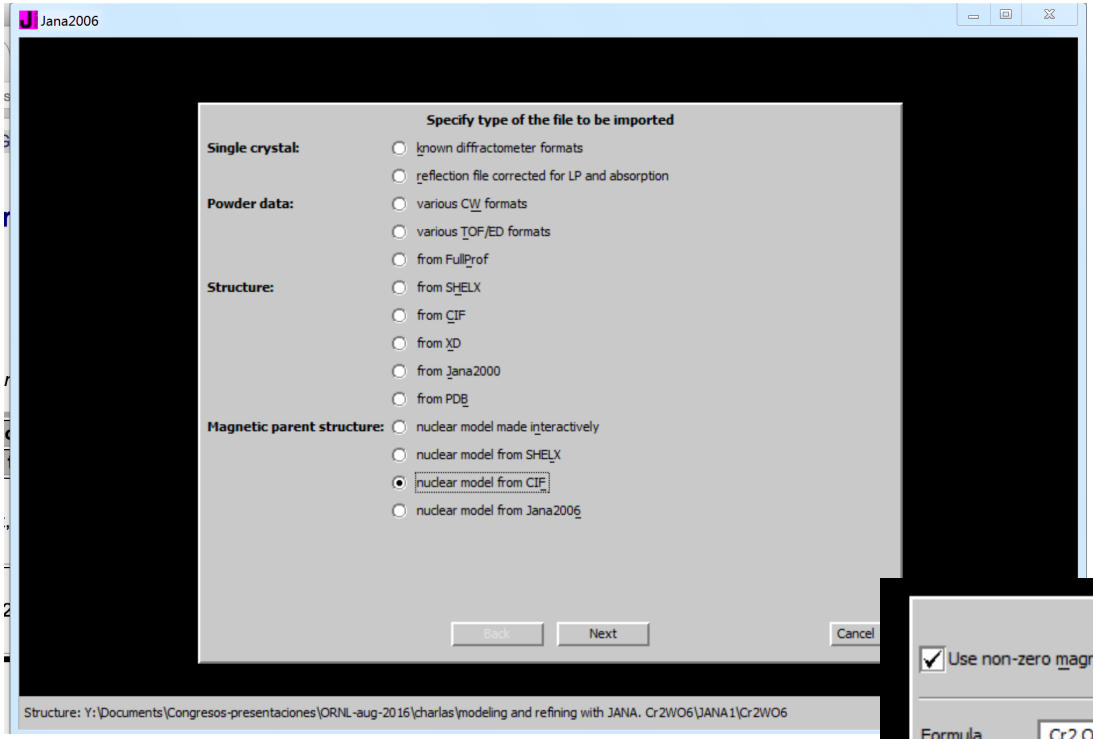
[On the screen: Define magnetic propagation vector and form factors]

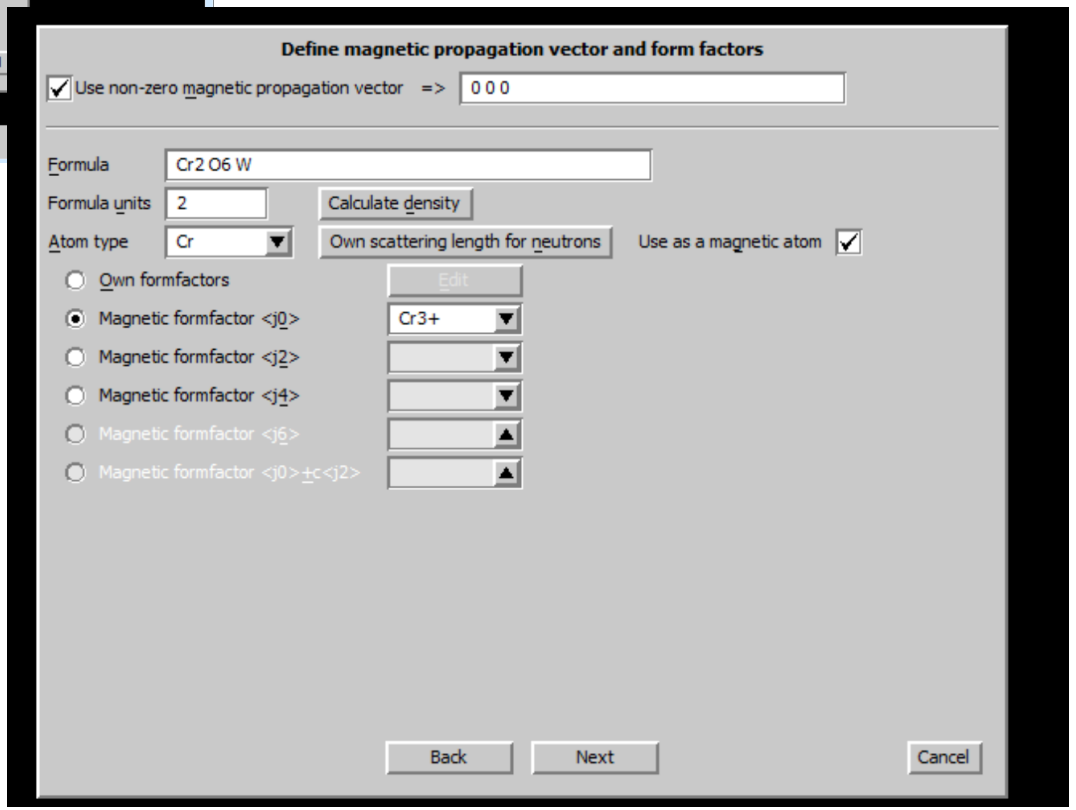
Check "Use non-zero magnetic propagation vector" and fill its value: (0, 0, 0)

For "Atom type" select "Cr"; check "Use as a magnetic atom"

For "Magnetic formfactor <j0>" select Cr3+;

The same for Cr3+; NEXT; FINISH


Jana2006
Specify type of the file to be imported
Single crystal:
known diffractometer formats
reflection file corrected for LP and absorption
Powder data:
various CW formats
various TOF/ED formats
from FullProf
Structure:
from SHELX
from CIF
from XD
from Jana2000
from PDB
Magnetic parent structure:
nuclear model made interactively
nuclear model from SHELX
nuclear model from CIF
nuclear model from Jana2006
Back
Next
Cancel
Structure: Y:\Documents\Congresos-presentaciones\ORNL-aug-2016\charlas\modeling and refining with JANA. Cr2WO6\JANA1\Cr2WO6


Define magnetic propagation vector and form factors
Use non-zero magnetic propagation vector =>
0 0 0
Formula
Cr2 O6 W
Formula units
2
Calculate density
Atom type
Cr
Own scattering length for neutrons
Use as a magnetic atom
Own formfactors
Edit
Magnetic formfactor <j0>
Cr3+
Magnetic formfactor <j2>
Magnetic formfactor <j4>
Magnetic formfactor <j6>
Magnetic formfactor <j0>+c<j2>
Back
Next
Cancel

4. Import the reflection file

[On the screen: Specify type of the file to be imported]

Select "Various CW format"; NEXT

Check that the name of the input file is "Cr2WO6_T4K.dat" and the format is **"Free format of 2th, I, [sig(I)]"; "Debye-Scherrer Method"**, NEXT

Choose OK if the program finds problems with the file format and requests to change the format of the file.

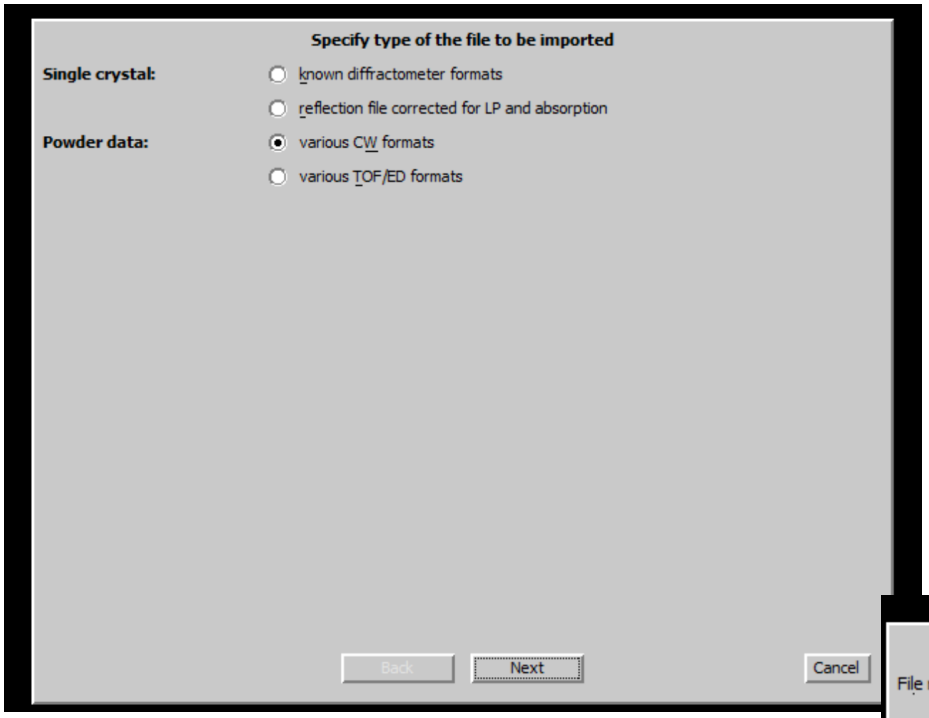
[On the screen: Complete/correct experimental parameters];

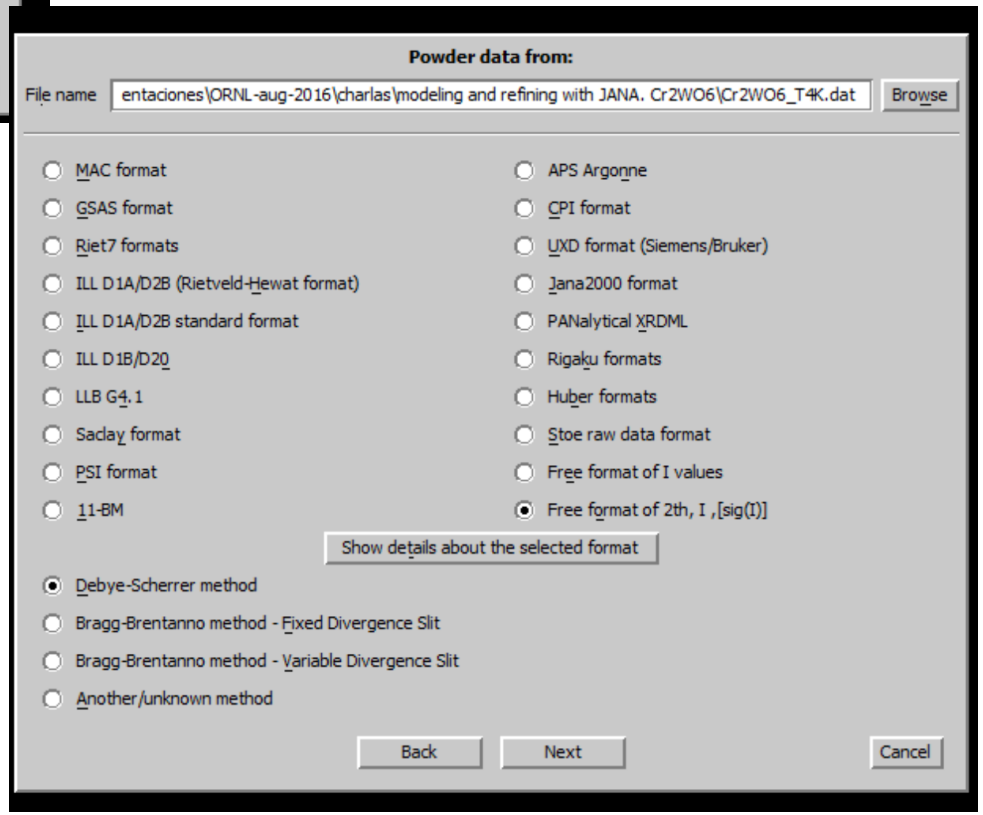
For "Wave length" type 2.4097 ; NEXT; FINISH

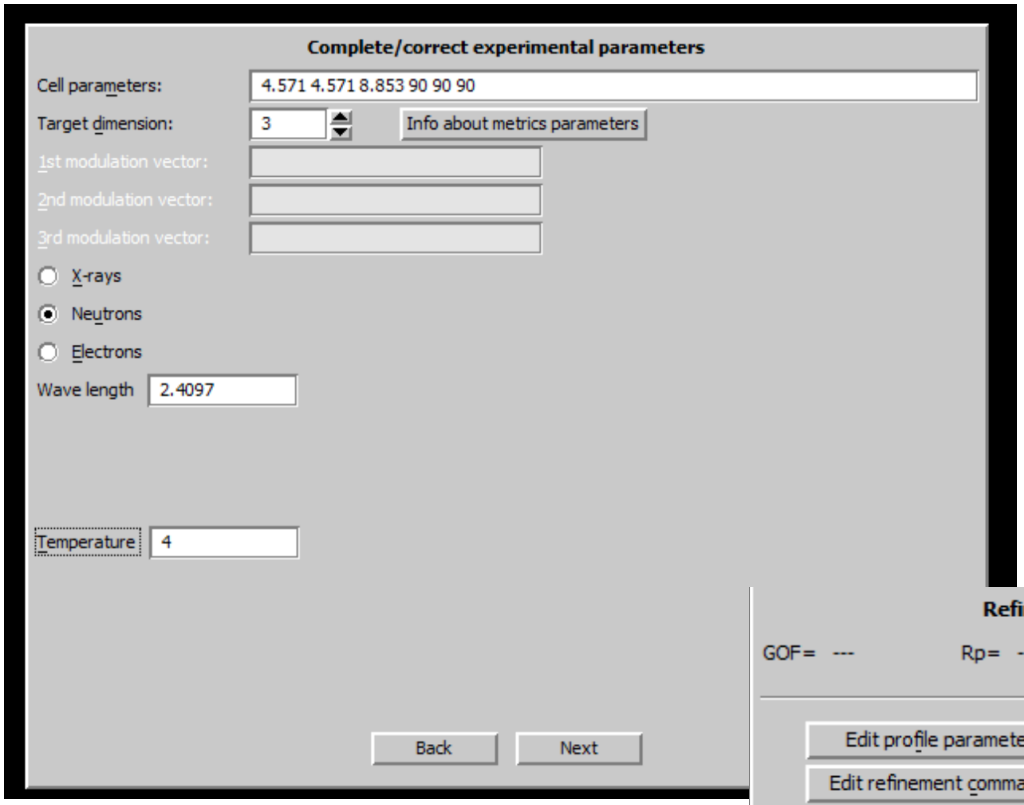
[On the screen: Data repository]

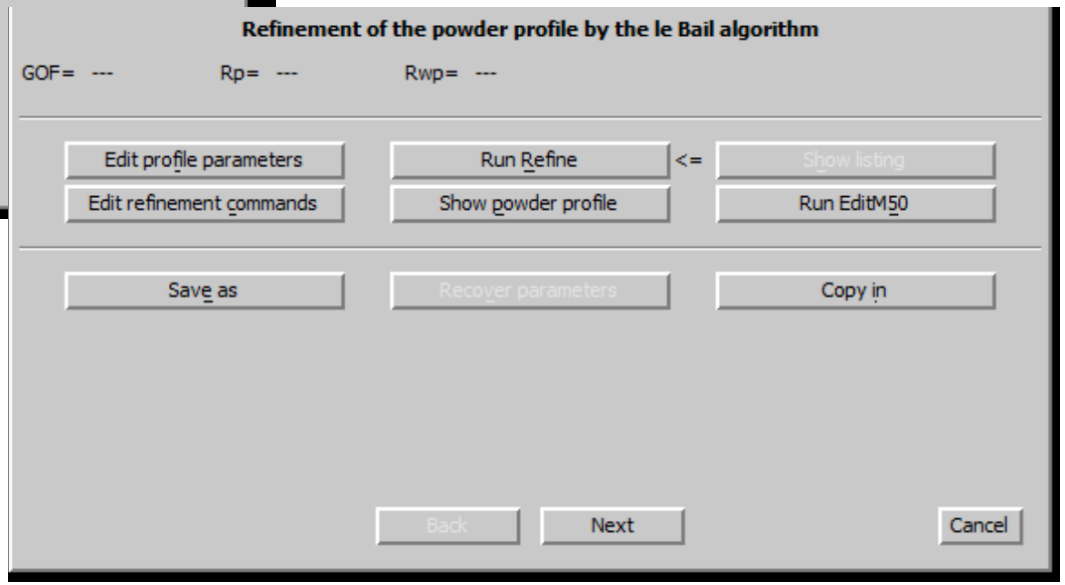
Optionally write the data temperature (?)

OK; YES to "Do you want to accept made changes?";


Specify type of the file to be imported
Single crystal:
known diffractometer formats
reflection file corrected for LP and absorption
Powder data:
various CW formats
various TOF/ED formats
Back
Next
Cancel


Powder data from:
File name
entaciones\ORNL-aug-2016\charlas\modeling and refining with JANA. Cr2WO6\Cr2WO6_T4K.dat
Browse
MAC format
GSAS format
Riet7 formats
ILL D1A/D2B (Rietveld-Hewat format)
ILL D1A/D2B standard format
ILL D1B/D20
LLB G4.1
Saclay format
PSI format
11-BM
APS Argonne
CPI format
UXD format (Siemens/Bruker)
Jana2000 format
PANalytical XRDML
Rigaku formats
Huber formats
Stoe raw data format
Free format of I values
Free format of 2th, I ,[sig(I)]
Show details about the selected format
Debye-Scherrer method
Bragg-Brentanno method - Fixed Divergence Slit
Bragg-Brentanno method - Variable Divergence Slit
Another/unknown method
Back
Next
Cancel


Complete/correct experimental parameters
Cell parameters:
4.571 4.571 8.853 90 90 90
Target dimension:
3
Info about metrics parameters
1st modulation vector:
2nd modulation vector:
3rd modulation vector:
X-rays
Neutrons
Electrons
Wave length
2.4097
Temperature
4
Back
Next


Refinement of the powder profile by the le Bail algorithm
GOF= ---
Rp= ---
Rwp= ---
Edit profile parameters
Run Refine
<=
Show listing
Edit refinement commands
Show powder profile
Run EditM50
Save as
Recover parameters
Copy in
Back
Next
Cancel

5. Refining of profile parameters by le Bail technique.

[On the screen: Information]

NEXT;

[On the screen: Refinement of the powder profile by the le Bail algorithm]

“Show powder profile”

The background is relatively smooth and it can be described without introducing manual background.

“Quit”;

“Edit profile parameters”;

[On the screen: Powder options]

I**n page “Cell**” **activate refinement of a, b and c parameters**;

In the page “Profile” change the starting value of GW to 50 and activate its refinement;

In the page “Corrections” activate refinement of “shift” parameter and change the number of used Legendre polynomials to 8

OK; YES to rewrite the changes

“Edit refinement command”; In the page “Basic” change the number of cycles to 20

OK; “Run Refine”;

The refinement should end with Rp~17.5%.

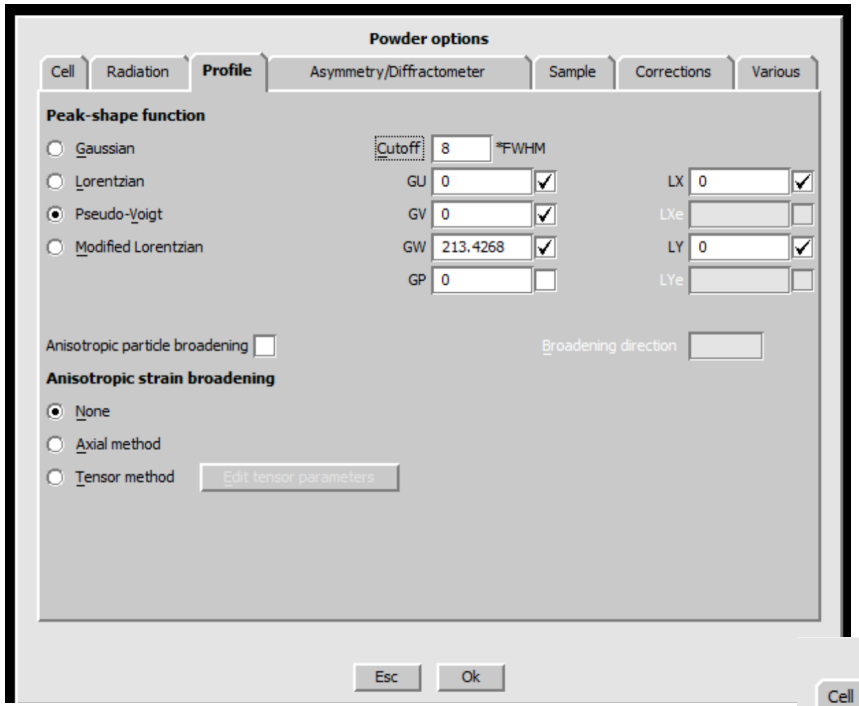
“Edit profile parameters”;

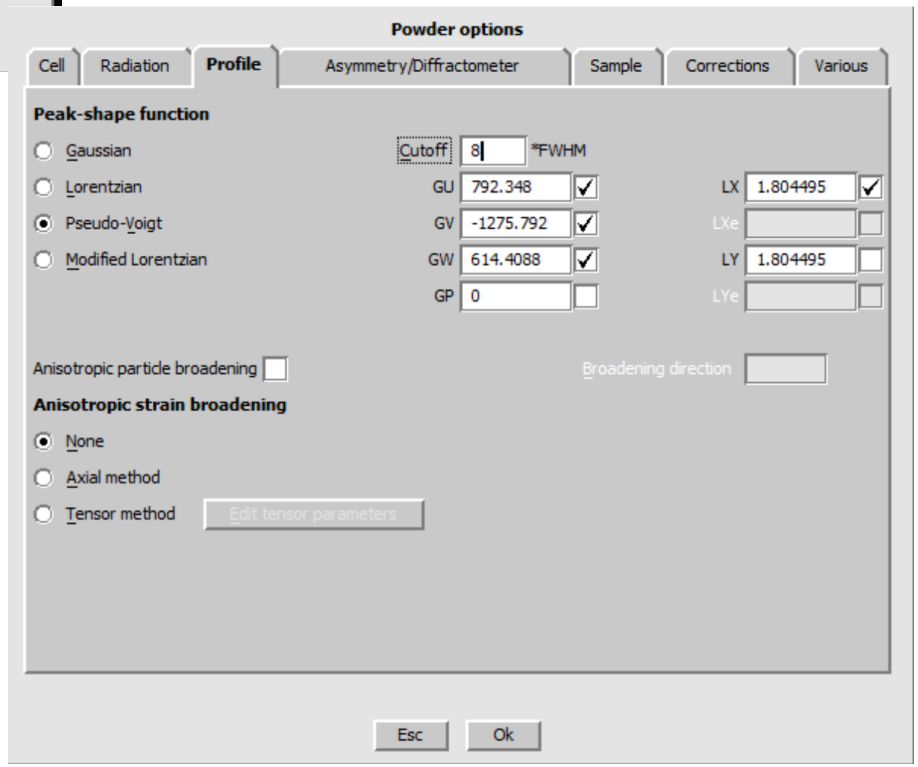
[On the screen: Powder options]

In the page “Profile” activate refinement of GU and GV; Change the peak-shape function to pseudo-Voigt and activate refinement of LX; OK; YES to the question about rewriting;

“Run Refine”

The refinement should converge to Rp~7.2%.


Powder options
Cell
Radiation
Profile
Asymmetry/Diffractometer
Sample
Corrections
Various
Peak-shape function
Gaussian
Lorentzian
Pseudo-Voigt
Modified Lorentzian
Cutoff
8
*FWHM
GU
0
GV
0
GW
213.4268
GP
0
LX
0
LXe
LY
0
LYe
Anisotropic particle broadening
Broadening direction
Anisotropic strain broadening
None
Axial method
Tensor method
Edit tensor parameters
Esc
Ok


Powder options
Cell
Radiation
Profile
Asymmetry/Diffractometer
Sample
Corrections
Various
Peak-shape function
Gaussian
Lorentzian
Pseudo-Voigt
Modified Lorentzian
Cutoff
8
*FWHM
GU
792.348
GV
-1275.792
GW
614.4088
GP
0
LX
1.804495
LXe
LY
1.804495
LYe
Anisotropic particle broadening
Broadening direction
Anisotropic strain broadening
None
Axial method
Tensor method
Edit tensor parameters
Esc
Ok

"Show powder profile"; *Some peaks are observed that should correspond to some impurity.*
The high Rp is probably due to these unadjustable peaks.
Select the region 2θ from 10 to 20 degs – use the button "X exactly".
The peak (0 0 1) has some asymmetry.
QUIT
"Edit profile parameters" -> Asymmetry/Difractometer-> correction by divergence
Activate refinement of HpS/L and HmS /L keeping their default values
OK; YES to save changes
"Run Refine";
The refinement should end with a similar Rp, but the asymmetry of the peak is well fit
NEXT; YES to leave the form for le Bail refinement

6. Refinement of the scale parameter for the nuclear structure
[On the screen: Refinement of the nuclear structure by the Rietveld method]
"Edit refinement commands"; On the page "Various" **select "Fixed commands"**;
Fix all parameters for all atoms ("*" for "Atoms/Parameters" textbox); "Add"; OK; OK;
*The command will appear in the panel as "fixed all *"*
"Run Refine"
The refinement should converge to Rp~14.4%, R(obs)~6.9%.
NEXT; YES to leave the form for Rietvel refinement

7. Testing different irreps to get the best single irrep model

[On the screen: Information]

NEXT*[On the screen: Representation analysis]*

This window has an information character. You can see here all irreps leading to Magnetic ordering. The "details" buttons provide more information about an individual irrep and its connection to the kernel magnetic symmetry.

NEXT

[On the screen: List of kernels and epikernels]

NEXT

[On the screen: Select Shubnikov superspace group]

Select the first group in the lis*t, one can see that this group forces cero magnetic moments and therefore is not possible*

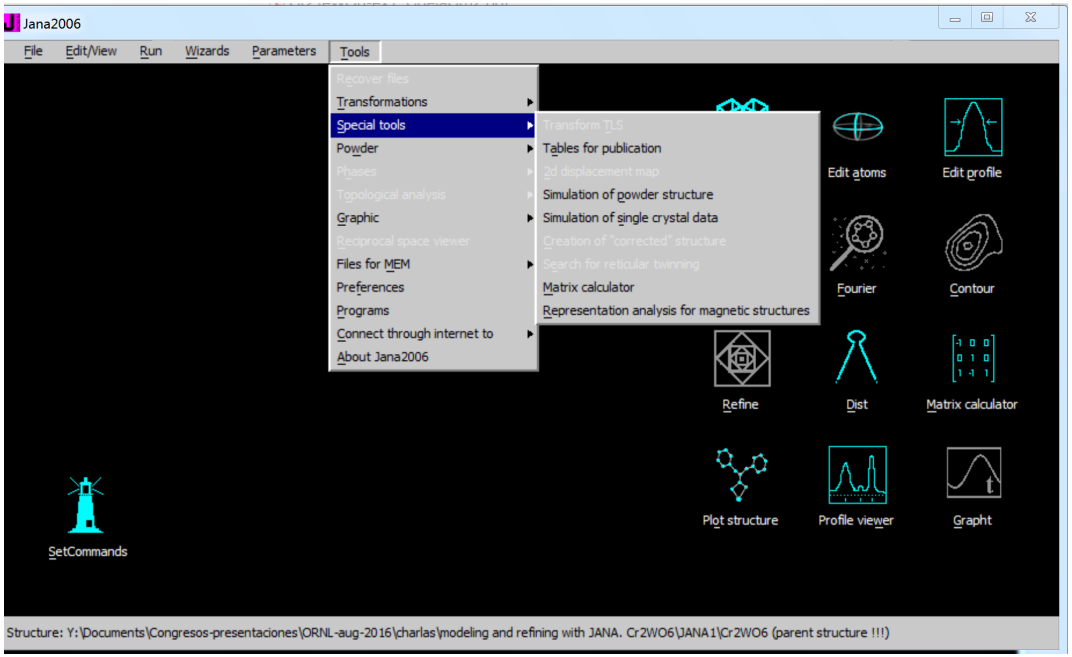
Select the second group in the lis*t, the spin modulations are forced to be along z. With the* ***Option "details****" one gets information about the group and the resulting constraints on the spin modulation parameters.*

Use the option "start profile simulation" to observe that the tetragonal groups in the list would not explain some of the magnetic peaks.

"Back"

Select some of the orthorhombic groups, and use the option "Start graphic simulation*", visualize the different models with different orthorhombic MSGs.*

Use also "start profile simulation" *to check that the group Pnn'm is a candidate to describe the symmetry of the phase. Select this group, which is one of the possible epikernels of mGM5-, together with Cmm'm. (JANA uses non-standard labels for the MSGs adapted to the basis used)*



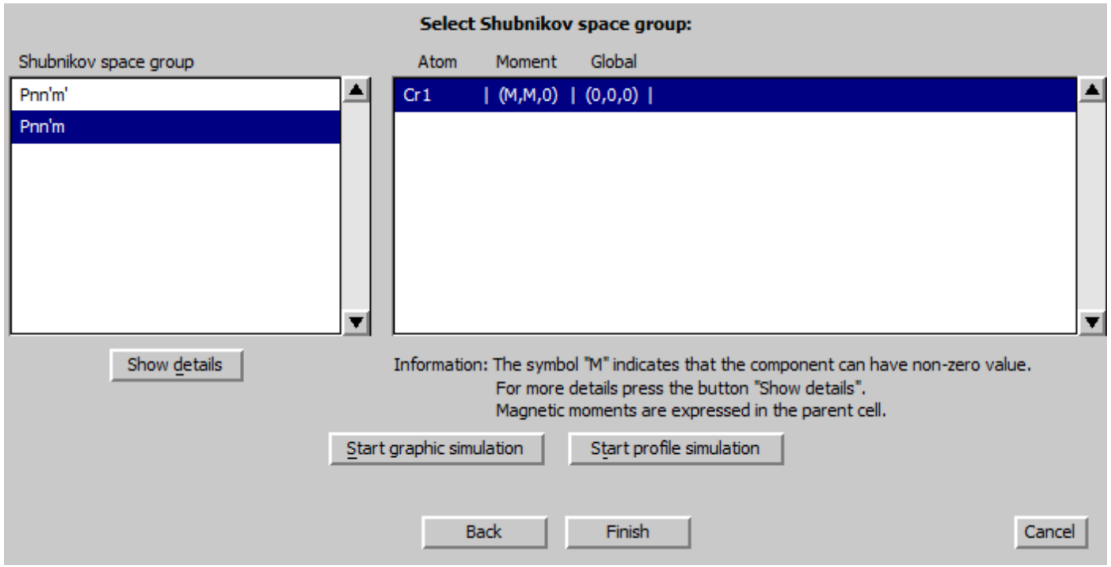
List of kernels and epikernels:

Shubnikov space group	Axes	Origin shift	Representation
P42'/mn'm	(1,0,0|0,1,0|0,0,1)	(0,0,0)	mGM4+
P42/m'n'm'	(1,0,0|0,1,0|0,0,1)	(0,0,0)	mGM1-
P42'/m'n'm	(1,0,0|0,1,0|0,0,1)	(0,0,0)	mGM2-
P42/m'nm	(1,0,0|0,1,0|0,0,1)	(0,0,0)	mGM3-
P42'/m'nm'	(1,0,0|0,1,0|0,0,1)	(0,0,0)	mGM4-
Cm'mm'	(1,1,0|-1,1,0|0,0,1)	(0,0,0)	mGM5+
Cmm'm	(1,1,0|-1,1,0|0,0,1)	(0,0,0)	mGM5-
Pnn'm'	(1,0,0|0,1,0|0,0,1)	(0,0,0)	mGM5+
Pnn'm	(1,0,0|0,1,0|0,0,1)	(0,0,0)	mGM5-
P2'/m'	(0,1,0|0,0,1|1,0,0)	(0,0,0)	mGM5+

Show details

Select from above kernels/epikernels one representative of a family of Shubnikov space groups for testing.

Back Next Cancel


Select Shubnikov space group:
Shubnikov space group
Pnn'm'
Pnn'm
Atom Moment Global
Cr1 | (M,M,0) | (0,0,0) |
Show details
Information: The symbol "M" indicates that the component can have non-zero value.
For more details press the button "Show details".
Magnetic moments are expressed in the parent cell.
Start graphic simulation
Start profile simulation
Back
Finish
Cancel

8. Refinement in the superspace group – Pnn'm

[On the screen: Select Shubnikov space group]

Select in the left panel the group Pnn'm; FINISH

YES to the question "Do you want to test structure for the last epikernel?";

[On screen: Select structure name]

Use the default name i.e. Cr2WO6_01 or any other; OK

Right click on the icon "Refine"; Select the page "Basic" and change the "Number of cycles" to 100 and the "Damping factor" to 0.1;

Select the page "Various". Use the button "Restrictions". All atoms are restricted to have identical ADP parameters;

Use button "Fixed commands" and fix all coordinates and delete or disable fixing of ADP's (if present).

OK; OK; "Yes+start";

The refinement should end with Rp~8.8% and Bragg R factors R(obs) ~4.5%, 4.7% and 3.1% for all, nuclear and magnetic reflections, respectively. ***(Edit/View → "View of Refine")***

Use "**Tools → Powder → Profile**" viewer and check fit of the calculated powder profile :

Select the page of Refine "Various". Use the button "fixed commands" and disable the command: "fixed xyz". Run refine again. Now the atom coordinates will also be refined.

The refinement should end with Rp~7.4% and Bragg R factors R(obs) ~2.3%, 2.2% and 2.0% for all, main and satellite reflections, respectively. ("View of Refine").

Go to "EditProfile" and activate the cell parameter b to be refined. Run Refine. R values Similar R values…

Edit/View → View of Refine: Mxo= 0.19(15) Myo=2.12(2)

9. Visualize the refined model with VESTA or MVISUALIZE (Jmol)

Tools -> Graphic -> Run Viewer -> Draw+return

File -> CIF utilities -> make CIF file
Save the file. It is in magCIF format an includes the list of reflections. With an editor delete this last part of the file, and produce a file with extension .mcif

This file can be used to visualize the structure with VESTA, Jmol or MVISUALIZE.

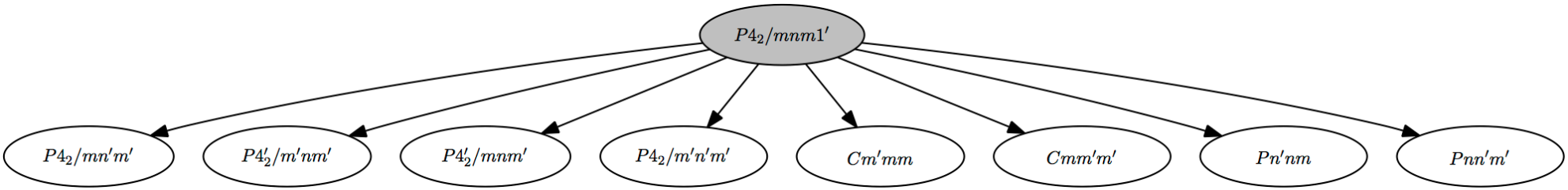
10. The weak canting Mx0 is negligible

Edit atoms -> Cr1 -> Edit
Fix to zero Mx0;
Right click Refine -> Basic -> desactive "Automatic refinement keys";
Refine -> Rp ~7.35, R= 2.19, 2.22, 1.88

Cr mx,my,0 (0 , 2.12(2), 0)

From FP tutorial of C. Del Cruz: (0, 2.04(2), 0)

K-SUBGROUPSMAG:



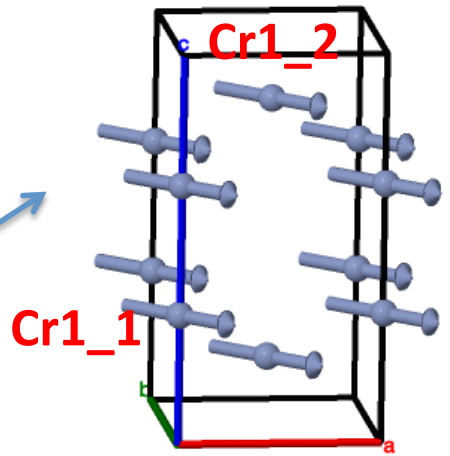
Maximal magnetic space groups for the parent space group 136 ($P4_2/mnm$) and the propagation vector k = (0, 0, 0)

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

N	Group (BNS)	Transformation matrix	General positions	Properties	Magnetic structure
1	$P4_2/m'n'm'$ (#136.503) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
2	$P4_2'/m'nm'$ (#136.502) 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	$P4_2/mn'm'$ (#136.501) 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	$P4_2'/m'n'm$ (#136.500) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
5	$P4_2'/mnm'$ (#136.499) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
6	$P4_2'/mn'm$ (#136.498) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
7	$P4_2/m'nm$ (#136.497) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
8	$P4_2/mnm$ (#136.495) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
9	$Cmm'm'$ (#65.486) Go to a subgroup	$\begin{pmatrix} 1 & 1 & 0 & 0 \\ -1 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
10	$Cm'mm$ (#65.483) Go to a subgroup	$\begin{pmatrix} 1 & 1 & 0 & 0 \\ -1 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
11	$Pnn'm'$ (#58.398) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show

8	$P4_2/mnm$ (#136.495) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	MAGNEXT *Tensor properties* MTENSOR	Show
9	$Cmm'm'$ (#65.486) Go to a subgroup	$\begin{pmatrix} 1 & 1 & 0 & 0 \\ -1 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
10	$Cm'mm$ (#65.483) Go to a subgroup	$\begin{pmatrix} 1 & 1 & 0 & 0 \\ -1 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
11	$Pnn'm'$ (#58.398) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
12	$Pn'nm$ (#58.395) 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

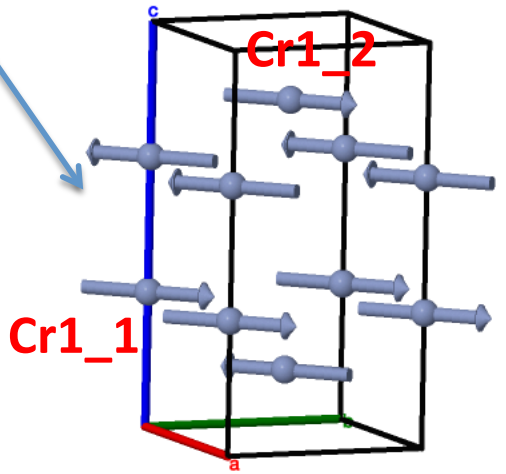
BNS:C m m 'm '
k1=0,0,0



Cr1_1 (mx,-mx,0)

Cr1_2 (mx',-mx',0)

BNS:C m 'm m
k1=0,0,0

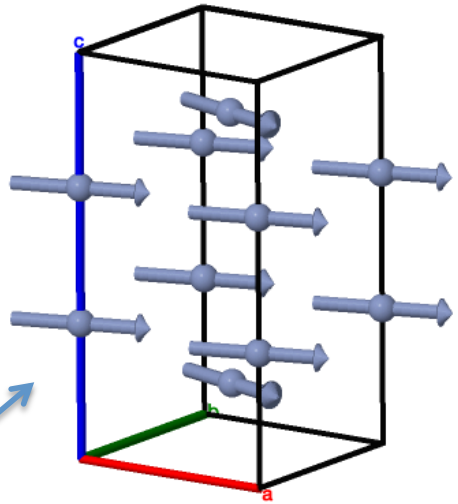


Magnetic site splits into two independent sites

Two spin parameters to be fit

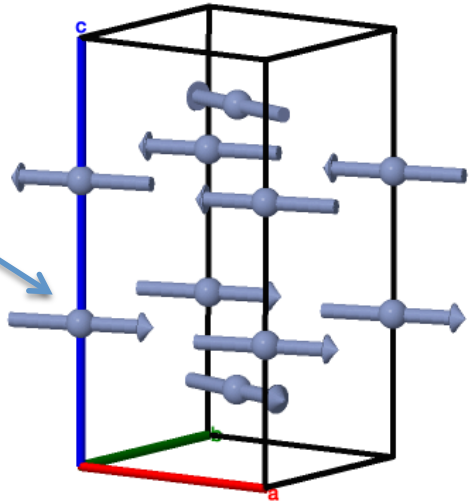
8	$P4_2/mnm$ (#136.495) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	MAGNEXT *Tensor properties* MTENSOR	Show
9	$Cmm'm'$ (#65.486) Go to a subgroup	$\begin{pmatrix} 1 & 1 & 0 & 0 \\ -1 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
10	$Cm'mm$ (#65.483) Go to a subgroup	$\begin{pmatrix} 1 & 1 & 0 & 0 \\ -1 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
11	$Pnn'm'$ (#58.398) Go to a subgroup	$\begin{pmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \end{pmatrix}$ Alternatives (domain-related)	Show	*Systematic absences* MAGNEXT *Tensor properties* MTENSOR	Show
12	$Pn'nm$ (#58.395) 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

BNS:P n n 'm '
k1=0,0,0



Cr1_1 (mx,my,0)

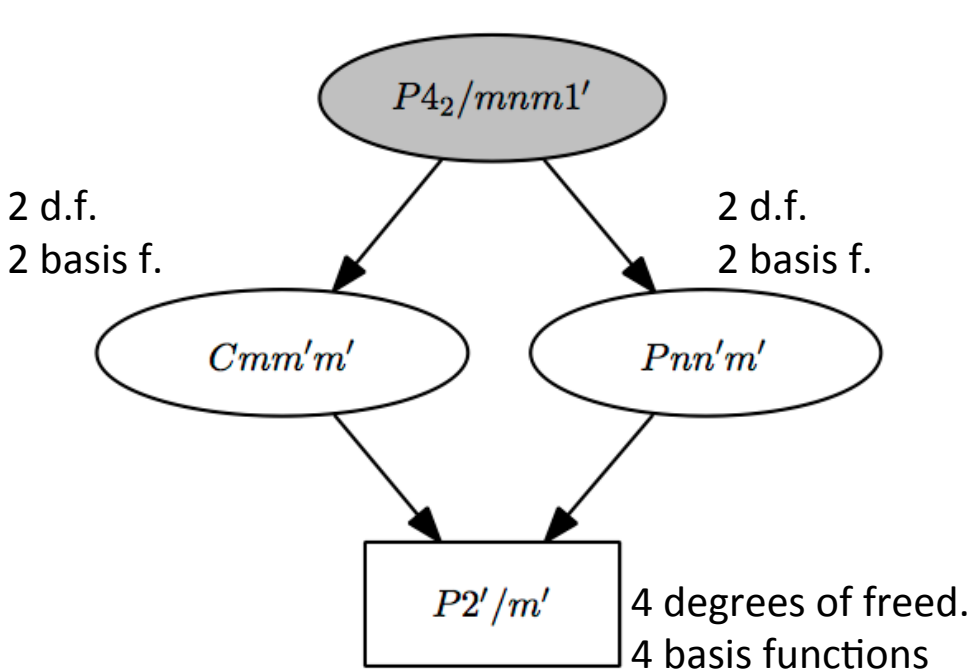
BNS:P n 'n m
k1=0,0,0



Only ONE independent magnetic site. But two independent spin components.
Spin canting symmetry allowed

Two spin parameters to be fit

Irrep GM5+ :



Irrep GM5-: