

Jana2006 Cookbook

Version April 2014

Solved examples on various topics covered by crystallographic computing system Jana2006. The cookbook cumulates examples from Jana workshops. The program Jana2006, the latest cookbook and data for examples are available in jana.fzu.cz.

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Example 12.1: Ba5Co5

Simple magnetic structure

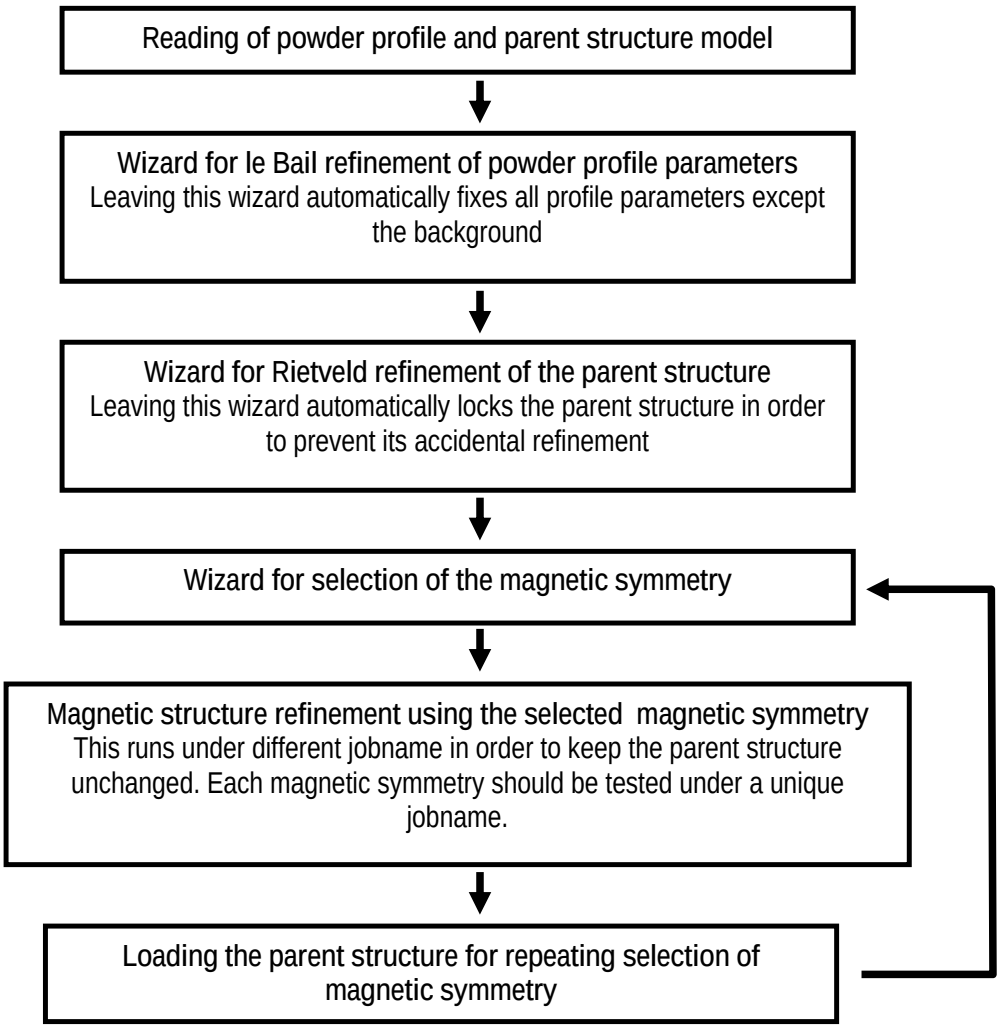
Revised: 30 July 2014

$Ba_5Co_5ClO_{13}$

Powder data measured with the ILL neutron source at 1.4 K
For more details: Mentré, O., Kauffmann, M., Ehora, G., Daviero-Minaud, S., Abraham F. & Roussel, P. (2008). *Solid State Sciences* **10**, 471-475.

Input files: Ba5Co5.dat (powder profile data)
Ba5Co5_nuclear.cif (nuclear structure)

In order to facilitate work with magnetic structures Jana2006 offers special wizards and protects the basic file - the parent structure. However, for experienced users of Jana, all operations offered by wizards can be also done manually by starting the tools separately from the basic user interface. The standard work with magnetic structures based on powder data follows this scheme:

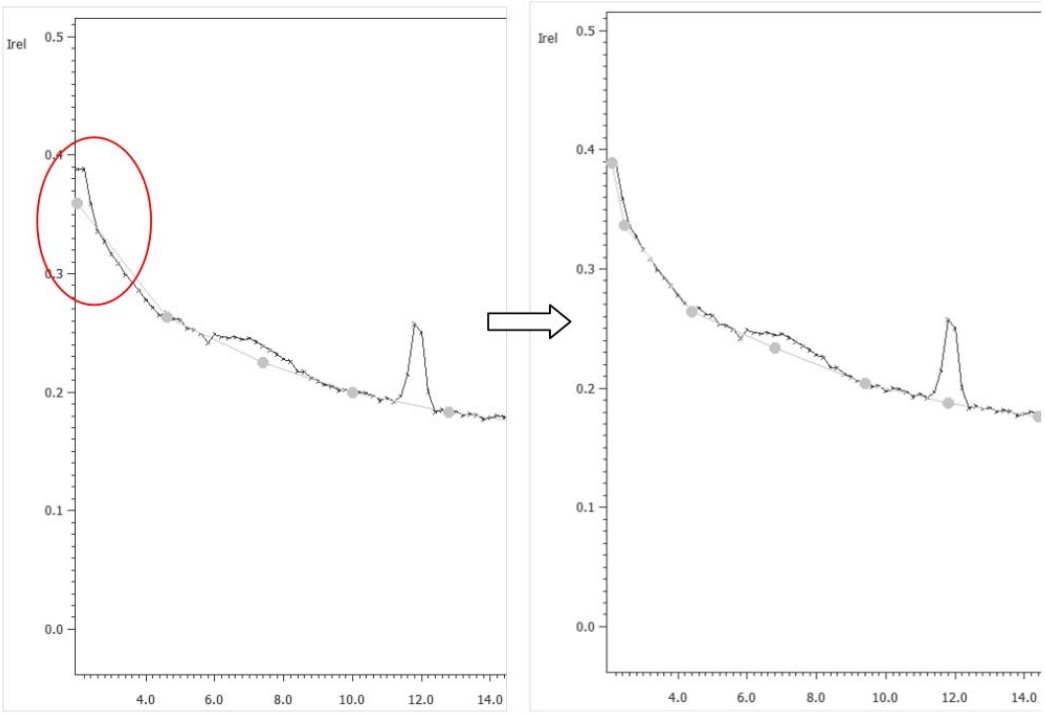


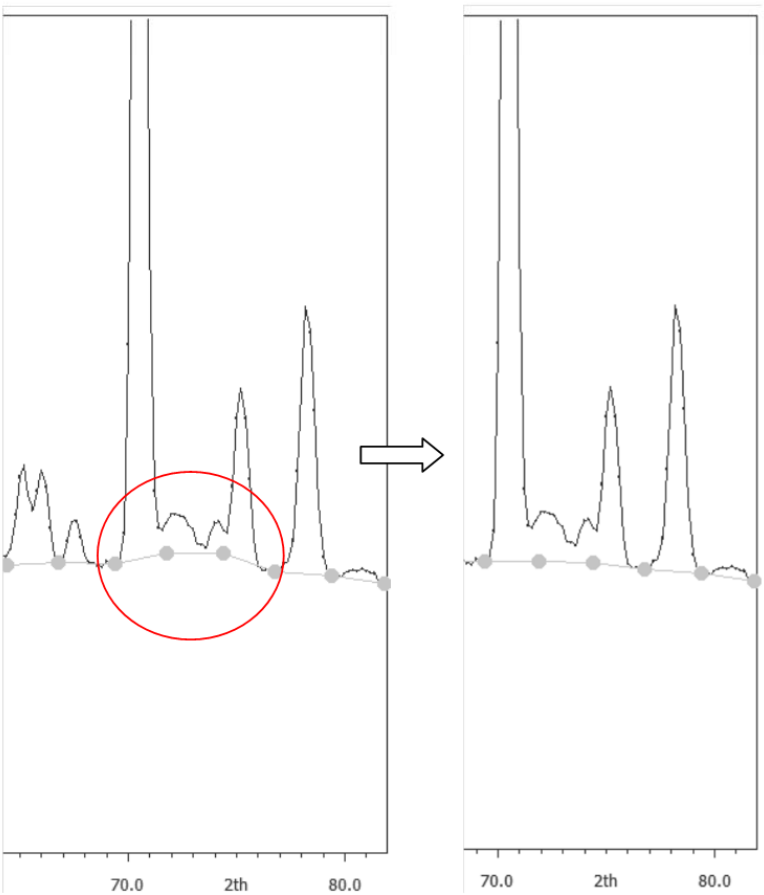
1. ***Creating new jobname***
   Start Jana2006
   "File → Structure → New" opens a file manager
   Left pane: locate directory with input files
   Right pane: double-click Ba5Co5
   Import Wizard
   [On the screen: Specify type of the file to be imported]
   Select "Magnetic: nuclear model from CIF"; NEXT
   [On the screen: Select input CIF file]
   Right pane: select (by the button with arrow) the input file Ba5Co5_nuclear.cif; OK

2. ***Define magnetic propagation vector and form factors***
   [On the screen: Define magnetic propagation vector and form factors]
   For "Atom type" select "Co"; check "Use as a magnetic atom"
   For "Magnetic formfactor <j0>" select Co4+; NEXT; Finish
   The parent structure has been created

3. ***Import the reflection file***
   [On the screen: Specify type of the file to be imported]
   Select "Powder data - Various CW formats"; NEXT
   Check that the name of the input file is "Ba5Co5.dat" and the format is "Free format of 2th, I, [sig(I)]"; NEXT
   [On the screen: Complete/correct experimental parameters];
   For "Wave length" type 2.52; NEXT; Finish
   [On the screen: Data repository]
   OK; YES to "Do you want to accept made changes?"

   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"
   As common for powder neutron data the background is strongly varying with the diffraction angle. It is worthwhile to estimate it manually and use just a few additional parameters.
   Manual background: "Create new"; Use default parameters; OK
   The automatic procedure has created a first estimation of the manual background, which can be modified manually: the points can be dragged by the left mouse button; dragging a point to the position of neighboring point deletes the point; clicking between two points creates a new point.
   Using "X+", "Y+", enlarge the profile plot
   Modify background point at high and low angles as indicated in the next figures:

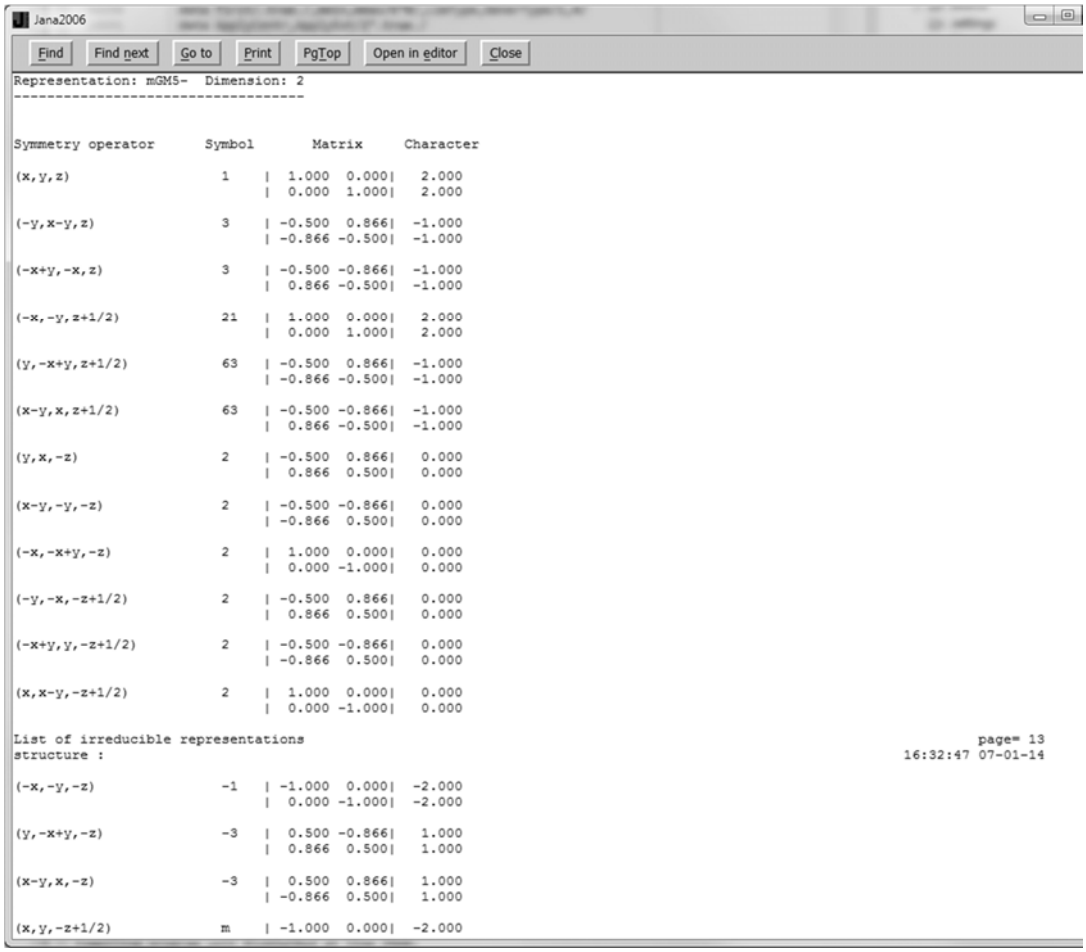




"QUIT"; Select "Accept the new manual background" and "Reset polynomial coefficients to zeros"; OK

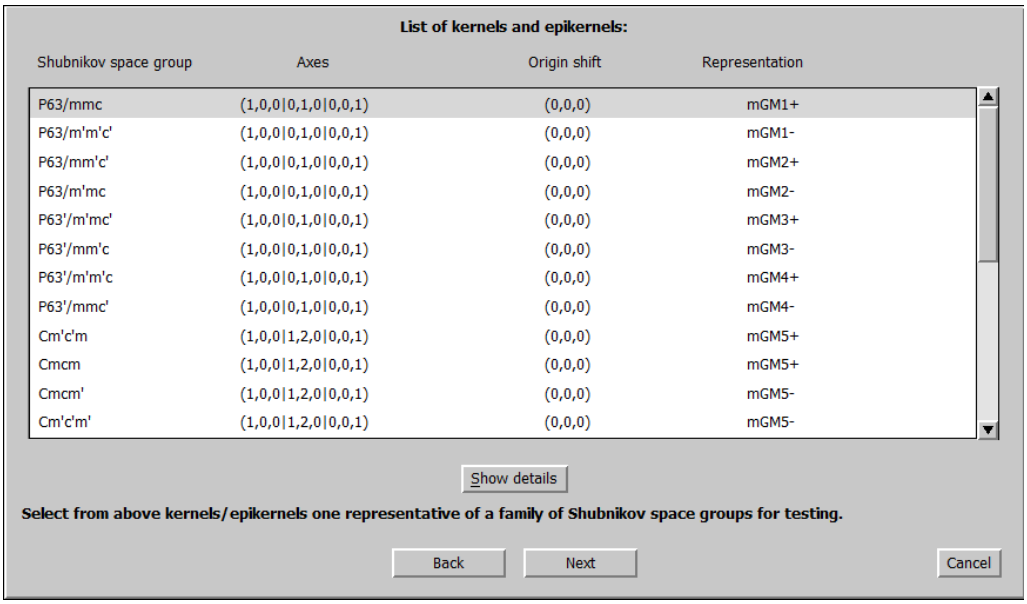
This means that the polynomial background will be refined and combined with the manual background
"Edit profile parameters";
[On the screen: Powder options]
In page "Cell" activate refinement of a and c parameters;
In the page "Profile" change the starting value of GW to 50 and activate its refinement;
In the page "Corrections" activate refinement of "shift" parameter; OK; YES to save changes
"Run Refine";
The refinement should converge to Rp~2.7%.
"Edit profile parameters";
[On the screen: Powder options]
In the page "Profile" change activate refinement of GU and GV; OK; YES to save changes
"Run Refine";
The refinement should converge to Rp~2.1%.
"Show powder profile" to see the fit of the calculated profile; QUIT
NEXT;
YES to "Do you really want to leave the form for le Bail refinement?"
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);
Press "Add";
*The command will appear in the panel as "fixed all *"*
OK; OK;
"Run Refine";
The refinement should converge to Rp~3.4%.
NEXT;
YES to "Do you really want to leave the form for Rietveld refinement?"
NEXT to accept the Information

4. ***Testing different irreps to get the best single irrep model***
[On the screen: Representation analysis]
This window has an informative character. You can see here all irreps leading to non-paramagnetic ordering. The "details" buttons provide more information about an individual irrep and its connection to the kernel magnetic symmetry.
Press "Display representations" and look at the representation "mGM5-".
Individual 2x2 representation matrices of each of 24 operations:

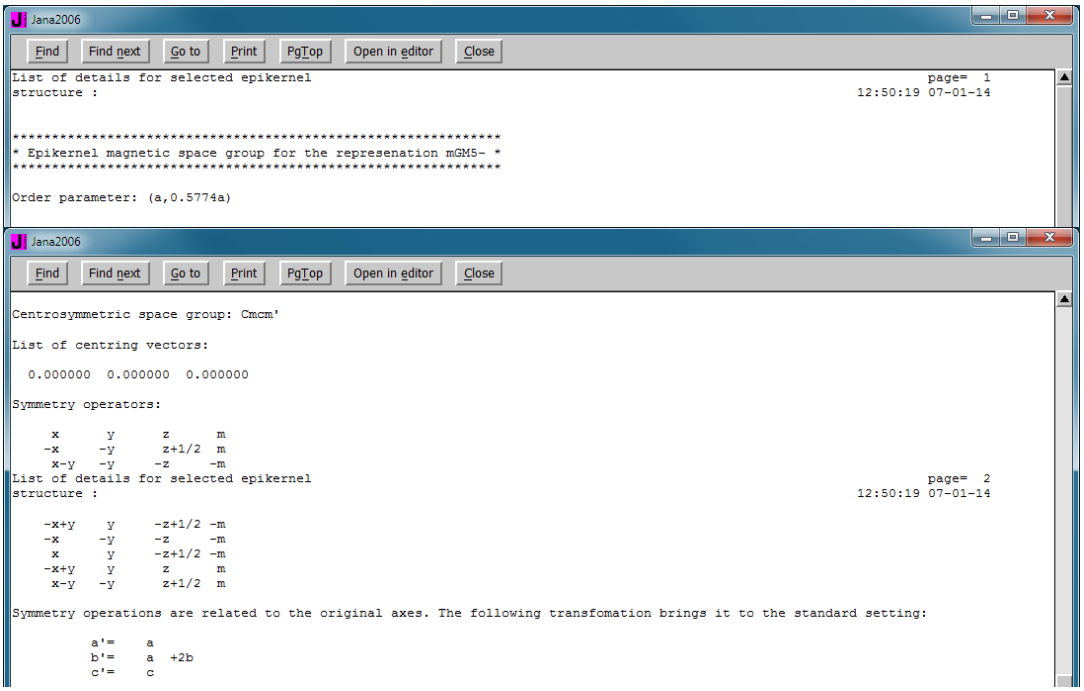


Close the window; NEXT;

[On the screen: List of kernels and epikernels]



Select e.g. magnetic group Cmcm' and press "Show details".



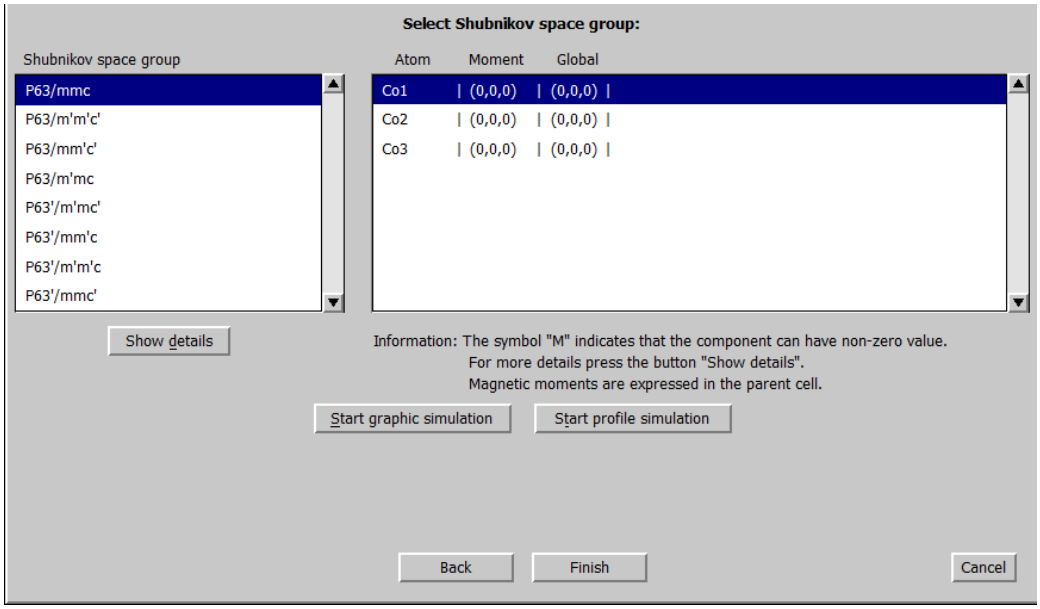
It shows symmetry operators of the selected magnetic group, transformation to the standard setting and the relevant order parameter.

Close;

Select one of the hexagonal magnetic space groups; NEXT

This will pass all present hexagonal magnetic space groups to further testing.

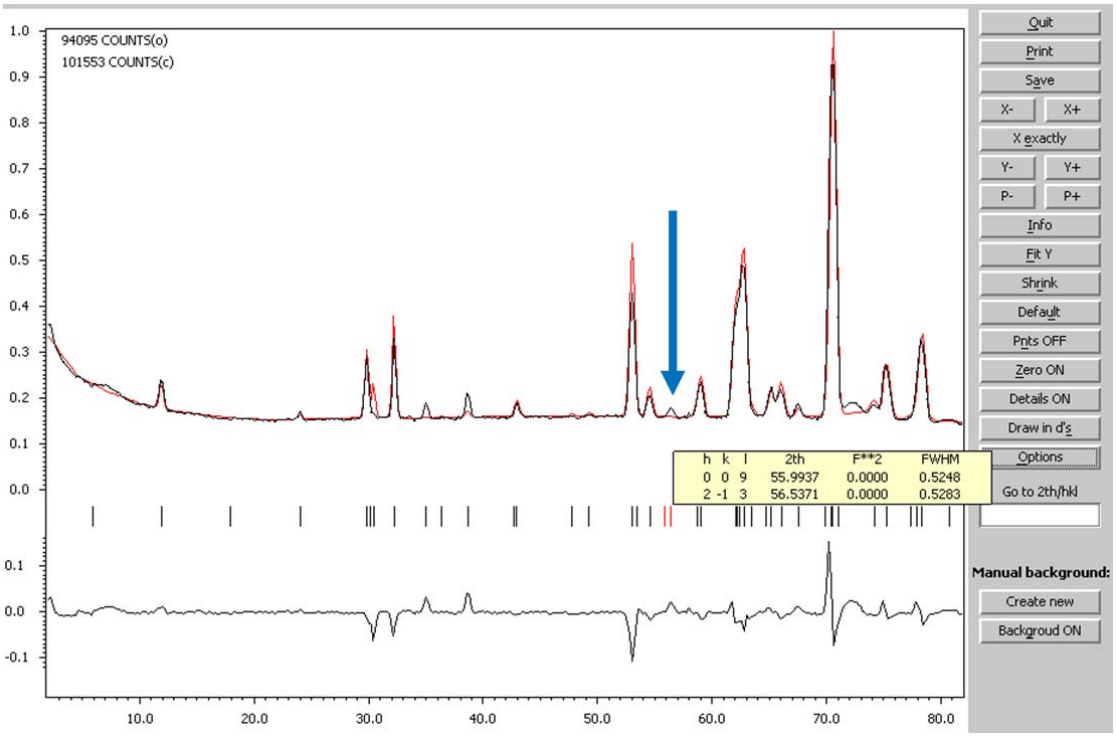
[On the screen: Select magnetic space group]



For an individual Shubnikov space group the tool shows components of the magnetic vector of all magnetic atoms in the structure. The button "Show details" gives more detailed

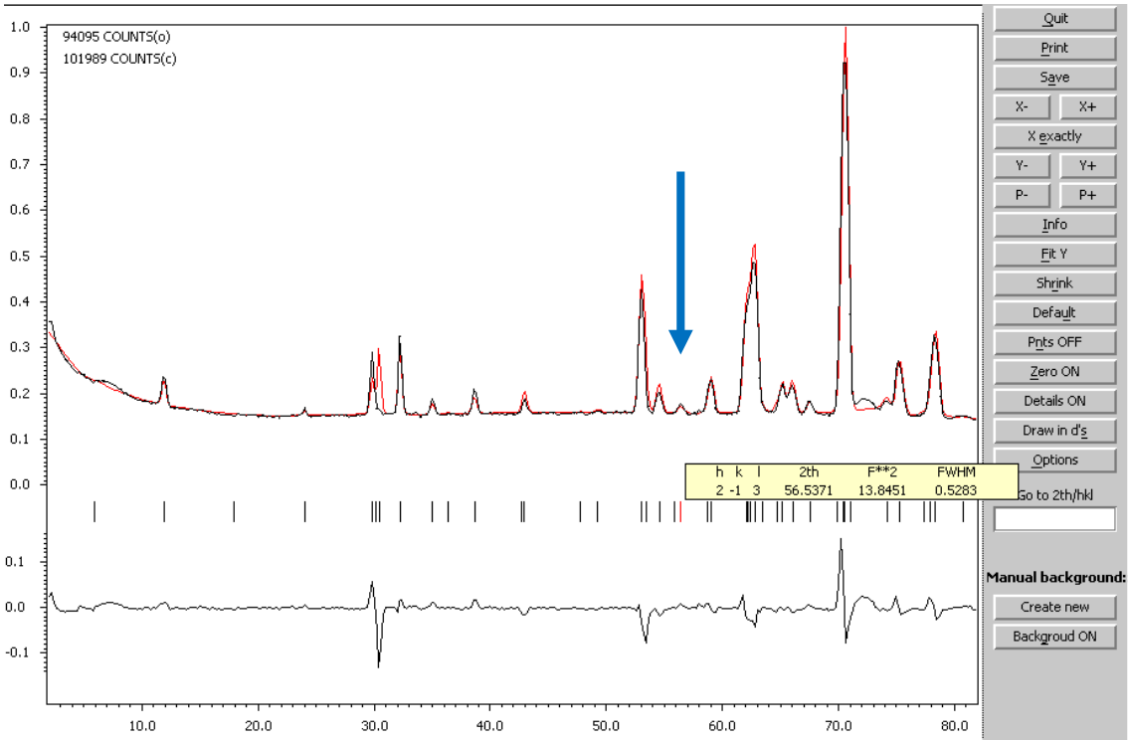
analysis. In our case only two Shubnikov space groups – P6$_3$/mm'c' and P6$_3$' /m'm'c – allow magnetic ordering for all Co atoms. Note that for the first one the global magnetic moment for is generally non-zero which means that this arrangement allows a ferromagnetic ordering. As ferromagnetism has not been detected the first model can be excluded from the analysis. However we shall test all models to check if such a decision can also be made on the base of diffraction experiment.

Select the Shubnikov space group *P6$_3$/mm'c'* and press "Start profile simulation". Use "Go to 2th/hkl" textbox and find position of the reflection (2,-1,3). Click on the tick corresponding to the reflection (2,-1,3):



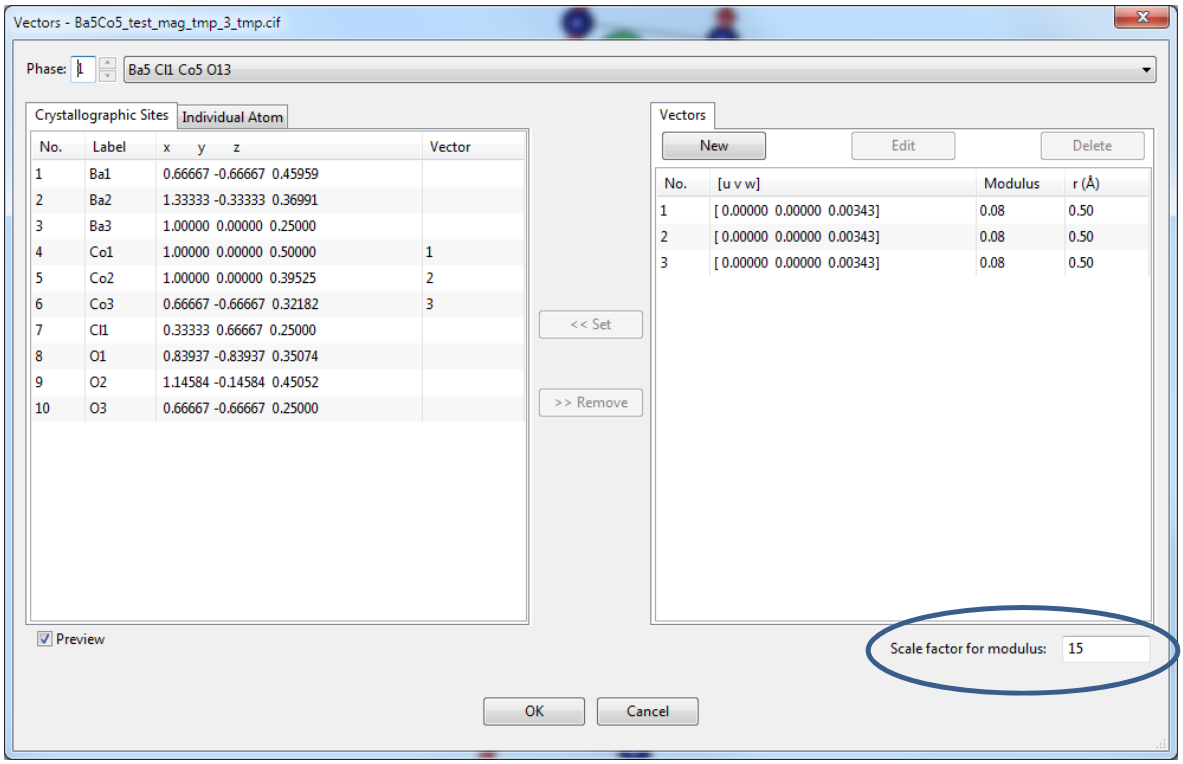
Although the peak is clearly visible, the calculated structure factor is zero.

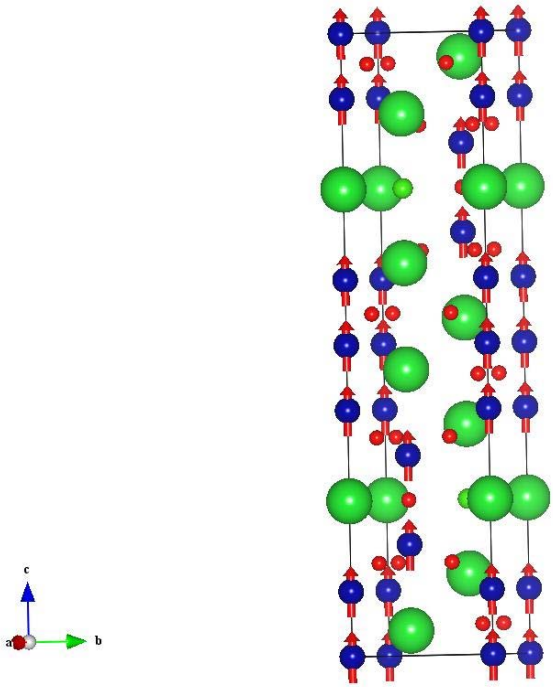
ESC
Press "Quit" and the select Shubnikov space groups *P6$_3$'/m'm'c* and investigate again the reflection (2 -1 3):



For this Shubnikov space group the intensity of (2 -1 3) is non-zero in accordance with the diffraction pattern. As this reflection is systematically absent for the nuclear structure (c glide plane) this is clear that it originates from magnetic scattering. As apparent from the the Shubnikov space group test all magnetic moments are directed along the z-axis. This leads to the systematic extinctions $l = 2n+1$ *for reflections* $(h,h,-2h,l)$ *but only in the case that the glide plane "c" is combined with the time inversion operation The fact that the peak (2 -1 3) is clearly detectable gives us another support for the Shubnikov space group* $P6_3'/m'm'c$.

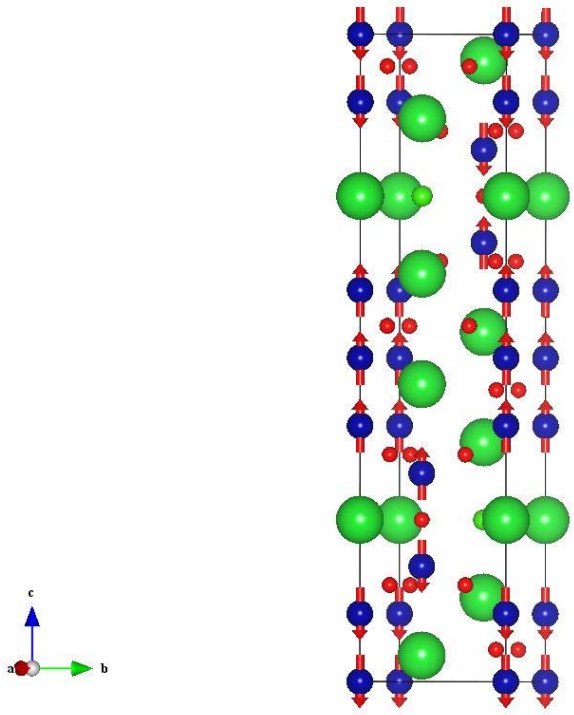
Select again the Shubnikov space group *$P6_3/mm'c'$* and press "Start graphic simulation";
Select "Use Vesta to draw the magnetic structure";
Press "Draw+return" or "Draw+continue";
In the VESTA program you can modify the length of the displayed vectors by "Edit→Vectors…"





You can see that in this case the magnetic moments, which are in the simulation chosen identical for the independent magnetic atoms, are not compensated in the unit cell.

Repeat this procedure for *P6₃'/m'm'c*



5. ***Refinement of the magnetic structure in P6₃/mm'c'(GM2+)***

   [On the screen: Select Shubnikov space group]

   Select the Shubnikov space group *P6₃/mm'c'*; Finish; YES to the question "Do you want to continue with the last kernel/epikernel?";

   [On the screen: Select structure name]

   Use the default name "Ba5Co5_01"; OK

   The test will be done under the new job name in order to keep the parent structure unchanged

   [On the screen: Basic window]

   Right-click the icon of Refine and select the page "Various".

   Click "Fixed commands" and make sure the coordinates of all atoms are fixed:

   fixed xyz *

   Click "Restrictions" and make sure ADP parameters all atoms are restricted to be the same:

   restric * 12

   The "Fixed commands" and "Restrictions" have been set automatically by the program when creating the testing structure.

   OK; select the page "Basic"

   Change the "Number of cycles" to 100 and the "Damping factor" to 0.1;

   OK; Start refinement;

   Refinement converges with Rp~3.3%, R(obs)~8.4%.

6. ***Refinement of the magnetic structure in P6₃'/m'm'c (GM4+)***

   [On the screen: Basic window]

   "File → Structure → History" and select the parent structure Ba5Co5; OK;

[On screen: Basic window]
"Tools → Special tools → Representation analysis for magnetic structures";
[On the screen: Representation analysis]
NEXT; Select one of hexagonal Shubnikov space groups; NEXT; Select *P6₃'/m'm'c*; Finish;
YES to the question "Do you want to continue with the last kernel/epikernel?";
[On the screen: Select structure name]
Use the default name; OK
[On the screen: Basic window]
Right-click the icon of Refine and select the page "Various".
Click "Fixed commands" and make sure the coordinates of all atoms are fixed:

```
fixed xyz *
```

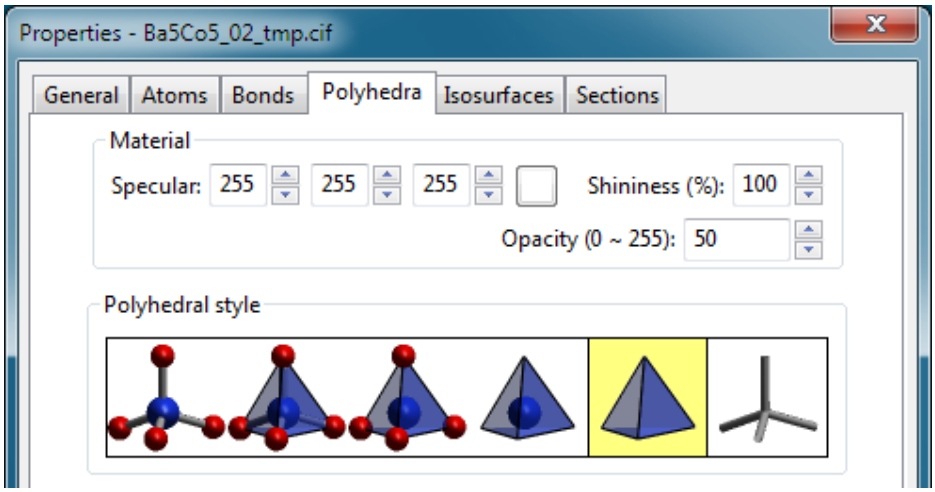
Click "Restrictions" and make sure ADP parameters all atoms are restricted to be the same:

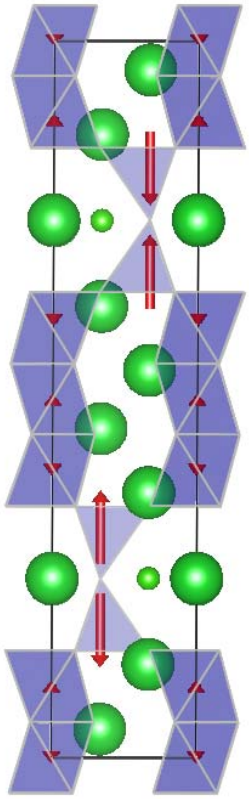
```
restric * 12
```

OK; select the page "Basic"
Check that the "Number of cycles" is 100 and the "Damping factor" to 0.1;
OK; Start refinement;
Refinement converges with Rp~3%, R(obs)~6%.

7. ***Drawing of the magnetic structure***

[On the screen: Basic window]
Double click on the icon "Plot structure";
Press "Draw+return" or "Draw+continue";
Select "Objects→Structural models" and choose "Polyhedral"
Select "Edit→Bonds"; New; Define A1 as Co, A2 as O and Max.length 2.3; Apply;OK;
Select "Objects→Properties→Polyhedra";
Choose new polyhedral style and reduce the opacity to 50:





The magnetic moments of atoms Co4 and Co5 are negligible. The atom Co6 is tetrahedrally coordinated by O atoms while the others have octahedral coordination.

Example 12.2: Ba6Co6

Commensurate magnetic structure – modulation (propagation) vector (0,0,1/2)
Revised: 30 July 2014

$Ba_6Co_6ClO_{16}$

Powder data measured with the ILL neutron source at 1.4 K

For more details: Mentré, O., Kauffmann, M., Ehora, G., Daviero-Minaud, S., Abraham F. & Roussel, P. (2008). *Solid State Sciences* **10**, 471-475.

Input files: Ba6Co6.dat (powder profile data)
Ba6Co6_nuclear.cif (nuclear structure)

1. ***Creating new jobname***
   Start Jana2006
   "File → Structure → New" opens a file manager
   Left pane: locate directory with input files
   Right pane: double-click Ba6Co6

2. ***Import Wizard***
   Select "Magnetic: nuclear model from CIF"; NEXT
   Right pane: select the input file Ba6Co6_nuclear.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 1/2
   Select atom type Co; check "Use as a magnetic atom"; select "Magnetic formfactor <j0>" Co4+; NEXT; FINISH
   The parent structure is created

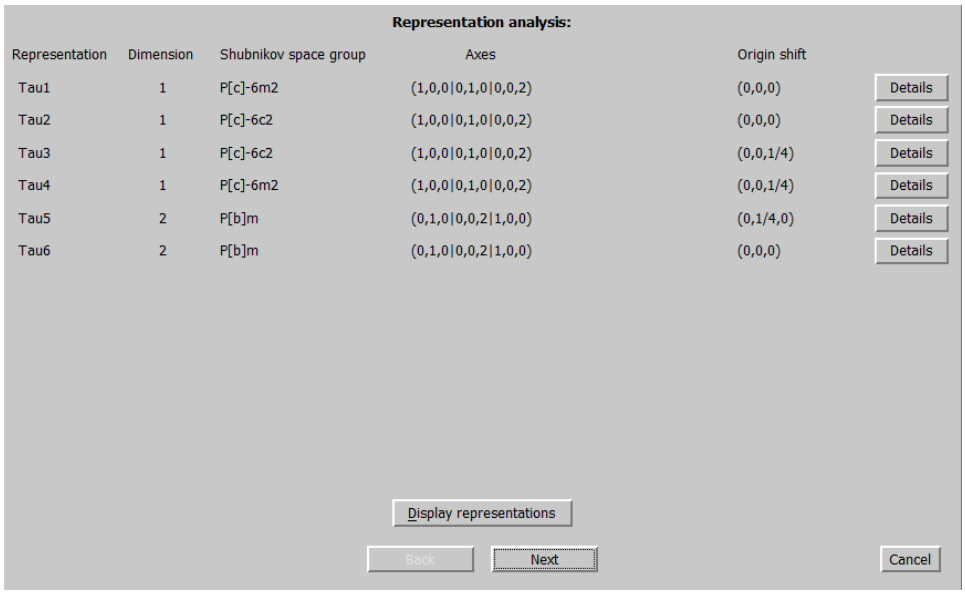
4. ***Import the reflection file***
   [On the screen: Specify type of the file to be imported]
   Select "Various CW format"; NEXT;
   Check that the name of the input file is "Ba6Co6.dat" and the data format is "Free format of 2th, I, [sig(I)]"; NEXT
   [On the screen: Complete/correct experimental parameters];
   Fill used wavelength 2.394; NEXT; FINISH
   [On the screen: Data repository]
   OK; YES to "Do you want to accept made changes?"

5. ***Refining of profile parameters by the le Bail technique.***
   [On the screen: Information]
   NEXT
   [On the screen: Refinement of the powder profile by the le Bail algorithm]
   "Show powder profile"

Although the background is often strongly varying with the diffraction angle for powder neutron data, in this case it is relatively smooth and it can be described without introducing a manual background.
"Quit"; "Edit profile parameters";
[On the screen: Powder options]
In page "Cell" activate refinement of a 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; for background enlarge the number of used Legendre polynomials to 10
OK; YES to save the changes
"Edit refinement commands"; In the page "Basic" change the number of cycles to 100; OK
"Run Refine";
The refinement should converge to Rp~9.3%.
"Edit profile parameters";
[On the screen: Powder options]
In the page "Profile" change activate refinement of GU and GV
Change the peak-shape function to pseudo-Voigt and activate refinement LY
OK; YES to save the changes
"Run Refine"
The refinement should converge to Rp~5.5%.
NEXT

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 (wild character *); "Add"; OK; OK
   "Run Refine"
   The refinement should converge to Rp=9.7%, R(obs)=6.5%
   NEXT

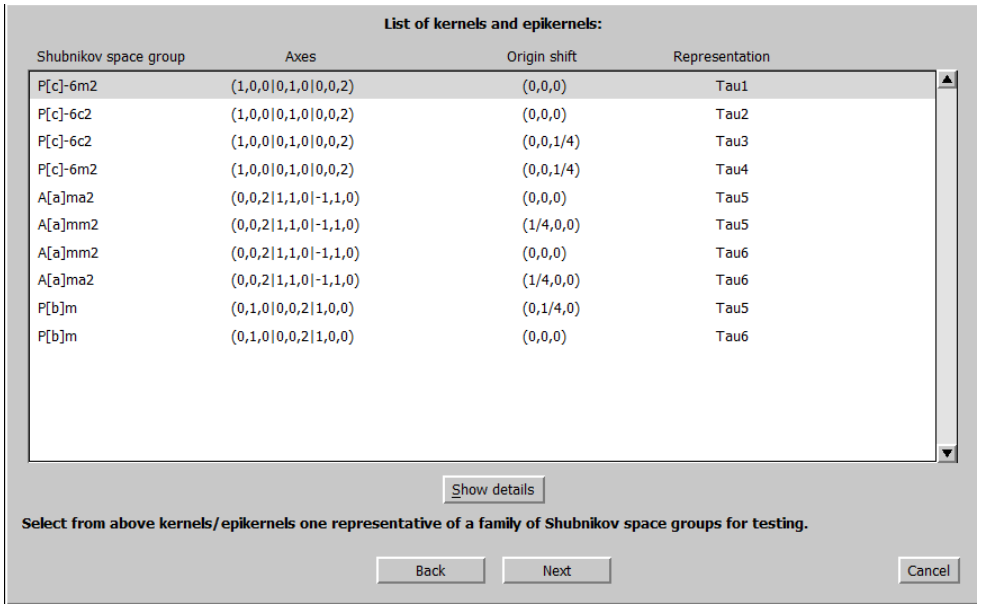
7. ***Testing different irreps to get the best single irrep model***
   [On the screen: Information]
   NEXT
   [On the screen: Representation analysis]



This window has an informative character and shows all irreps leading to non-paramagnetic ordering. The "Details" buttons provides more information about an individual irrep. There are four highest symmetrical hexagonal Shubnikov space groups; two of them differ only in the origin position which may lead for commensurate cases to different solutions.

NEXT

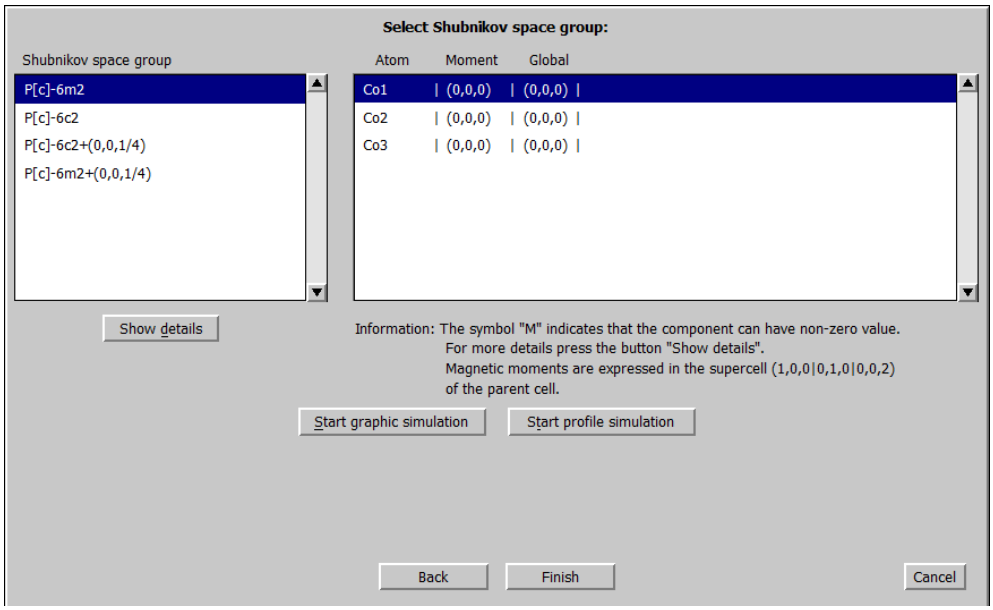
[On screen: List of kernels and epikernels]



Select one of the hexagonal Shubnikov groups; NEXT

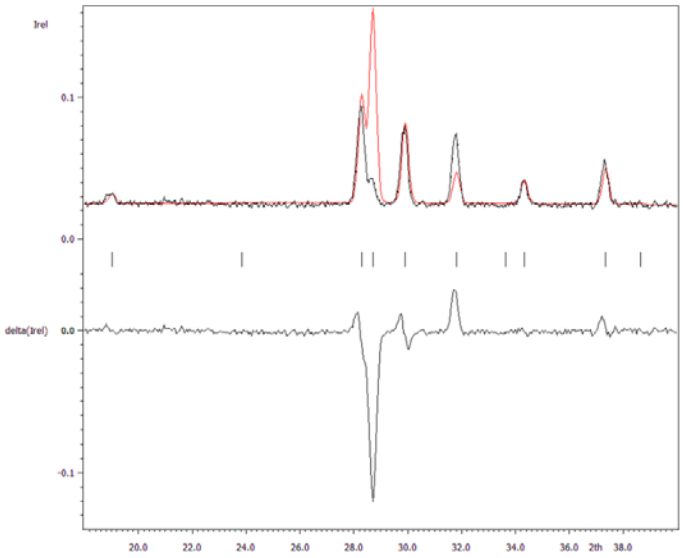
This will pass all present hexagonal Shubnikov groups to further testing.

[On the screen: Select Shubnikov space group]

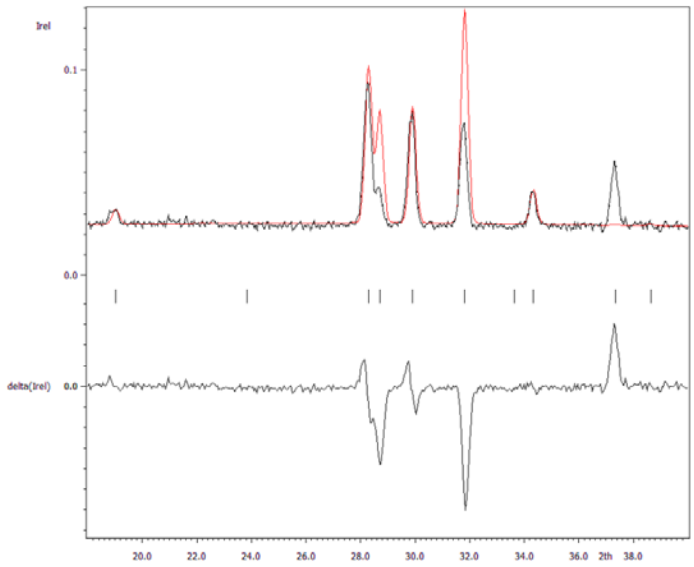


Only the second and the third Shubnikov group allows for non-zero magnetic moments of Co atoms.

Select the second Shubnikov group:
P[c]-6c2
Press “Start profile simulation”;
Press "X exactly" and focus the region 18-40 degrees; Press “Fit Y”:



Press “Quit” and repeat this procedure for the next magnetic Shubnikov group *P[c]-6c2 with origin shifted by (1/4,0,0), which is listed in the third line*:



At this stage none of them can be excluded.

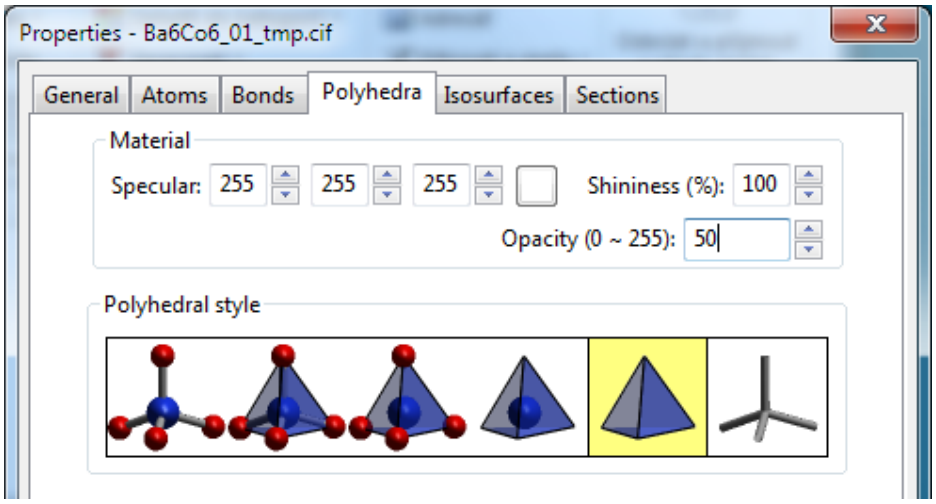
8. ***Refinement of the magnetic structure – P[c]-6c2***

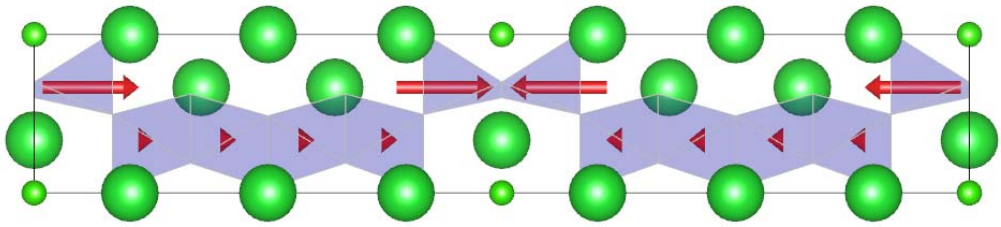
[On the screen: Select Shubnikov space group]
Select the Shubnikov space grou*p P[c]-6c2* from the second line of the list
FINISH; YES to the question "Do you want to continue with the last kernel/epikernel?";
[On the screen: Select structure name]
Use the default name; OK
[On the screen: Basic window of Jana2006]
Right-click the icon of Refine and select the page "Various"
Use the button "Restrictions" and ensure that all atoms (i.e. *) have been restricted to have identical ADP parameters.
*The restriction "restric * 12" has been set automatically when the program created the testing structure. To see the meaning of the restriction focus it by mouse and press "Load". "Load" opens the restriction for editing. Make sure that "Coordinates" and "Modulations" boxes are cleared and press "Rewrite" to close the editing mode.*
OK
Select the page "Fixed commands" and (similarly like in the previous step) ensure that all atomic positions are fixed in their current values ("fixed xyz *").
OK
Select the page "Basic" and change the "Number of cycles" to 100 and the "Damping factor" to 0.1; OK; Start refinement
Refinement converges with Rp~8.8%, R(obs)~ 6.5%, 6.5%, 6.5% for all, nuclear and magnetic reflections, respectively.

9. ***Drawing the resulting magnetic structure***

[On the screen: Basic window]
Double click on the icon "Plot structure"
Select "Use Vesta to draw the magnetic structure"
Press "Draw+return" or "Draw+continue"
Select "Objects→Structural models" and choose "Polyhedral"

Select “Edit→Bonds”; New; Define A1 as Co, A2 as O and Max.length 2.3; Apply;OK;
Select “Objecs→Properties→Polyhedra”;
Choose new polyhedral style and reduce the opacity to 50:





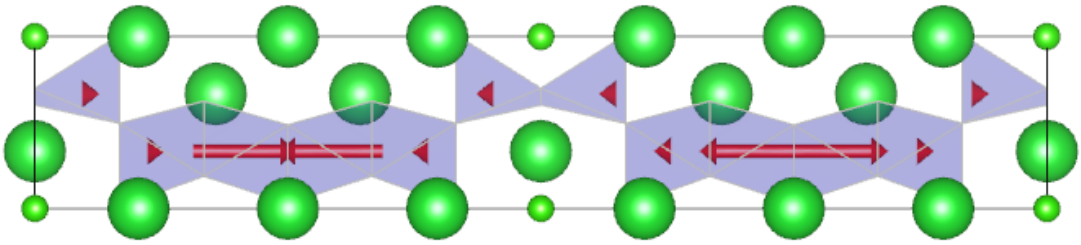
10. Refinement of the magnetic structure – P[c]-6c2 origin shifted by (0,0,1/4)

[On the screen: Basic window]
“File → Structure → History” and select the parent structure Ba6Co6; OK;
[On screen: Basic window]
“Tools → Special tools → Representation analysis for magnetic structures”;
[On the screen: Representation analysis]
NEXT; Select one of hexagonal magnetic groups; NEXT; Select the third Shubnikov space group; FINISH
YES to the question “Do you want to continue with the last kernel/epikernel?”;
[On the screen: Select structure name]
Use the default name; OK
[On the screen: The following transformation can bring the structure to the standard setting]
NO to reject the origin shift
[On the screen: Basic window]
Right-click the icon of Refine and select the page “Various”.
Similarly like in the previous test ensure (under “Restrictions”) that all atoms are restricted

to have identical ADP and ensure (under "Fixed commands") that coordinates of all atoms are fixed.
Select the page "Basic" and change the "Number of cycles" to 100 and the "Damping factor" to 0.1; OK; Start refinement;
Refinement converges with Rp~8.9%, R(obs)~ 6.6%, 6.5%, 7.7% for all, nuclear and magnetic reflections, respectively.

11. *Comparing of two different results*

[On screen: Basic window]
Double click on the icon "Plot structure";
Select "Use Vesta to draw the magnetic structure"; OK
Press "Draw+return" or "Draw+continue"
In VESTA select "Objects→Structural models" and choose "Polyhedral"
Select "Edit→Bonds"; New; Define A1 as Co, A2 as O and Max.length 2.3; Apply;OK;
Select "Objecs→Properties→Polyhedra";
Choose new polyhedral style and reduce the opacity to 50;



The models are very different. In the first model the only significant magnetic moment was localized at the tetra hedrally coordinated Co atom while in the second one the significantly non-zero magnetic moment was localized at the octahedrally coordinated site.

The first structure model has better R value. Moreover the presence of magnetic moments at the tetrahedral site corresponds to the result in Example 12.1

Example 12.3: PrSrMnO

Commensurate magnetic structure – modulation (propagation) vector (1,0,0)

Revised: 30 July 2014

Powder data measured the ILL neutron source at 10 K

For more details: K. Knížek, J. Hejtmánek, and Z. Jirák, C. Martin, M. Hervieu, and B. Raveau, G. André and F. Bourée, Chemistry of Materials, (2004), 1104-1110.

Input files: PrSrMnO.dat (powder profile data)
PrSrMnO_nuclear.cif (nuclear structure)

1. ***Creating new jobname***
   Start Jana2006
   "File → Structure → New" opens a file manager
   Left pane: locate directory with input files
   Right pane: double-click PrSrMnO

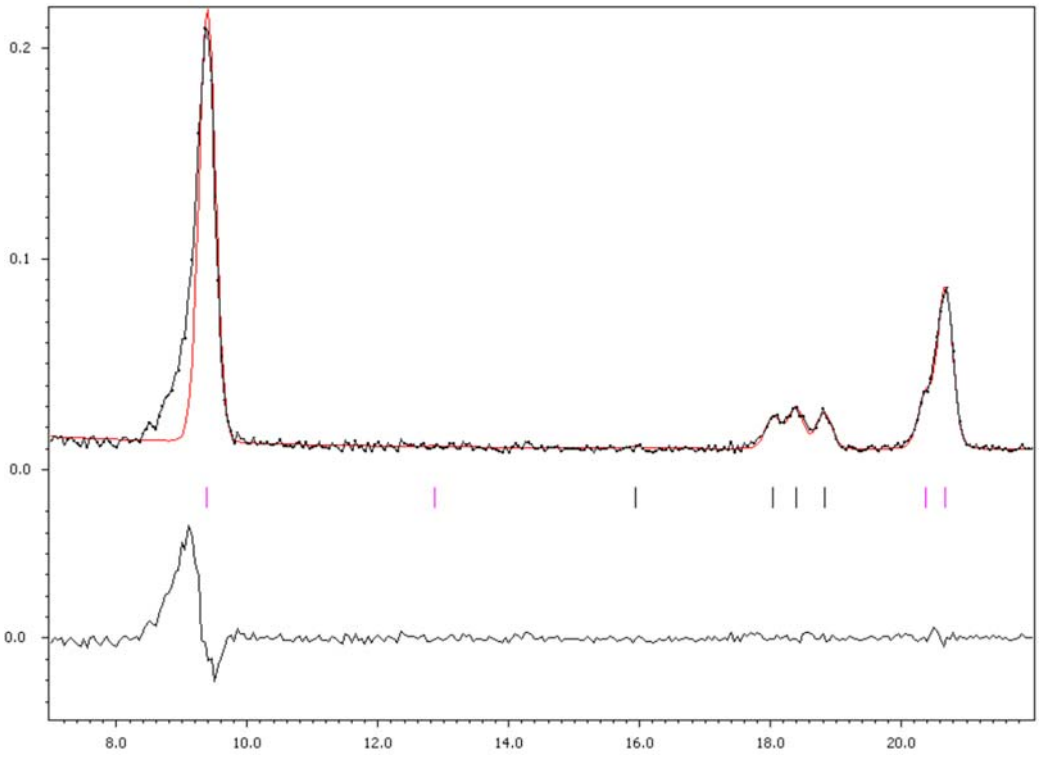
2. ***Import Wizard***
   Select "Magnetic: nuclear model from CIF"; NEXT
   [On the screen: Select input CIF file]
   Right pane: select the input file PrSrMnO_basic.cif; OK

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: 1 0 0
   For "Atom type" select "Mn"; check "Use as a magnetic atom"
   For "Magnetic formfactor <j0>" select Mn3+; NEXT; FINISH
   The parent structure is created

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 "PrSrMnO.dat" and the format is "ILL D1A/D2B (Rietveld-Hewat format)"; NEXT
   [On the screen: Complete/correct experimental parameters];
   For "Wave length" type 1.227; NEXT; FINISH
   [On the screen: Data repository]
   OK; YES to "Do you want to accept made changes?"

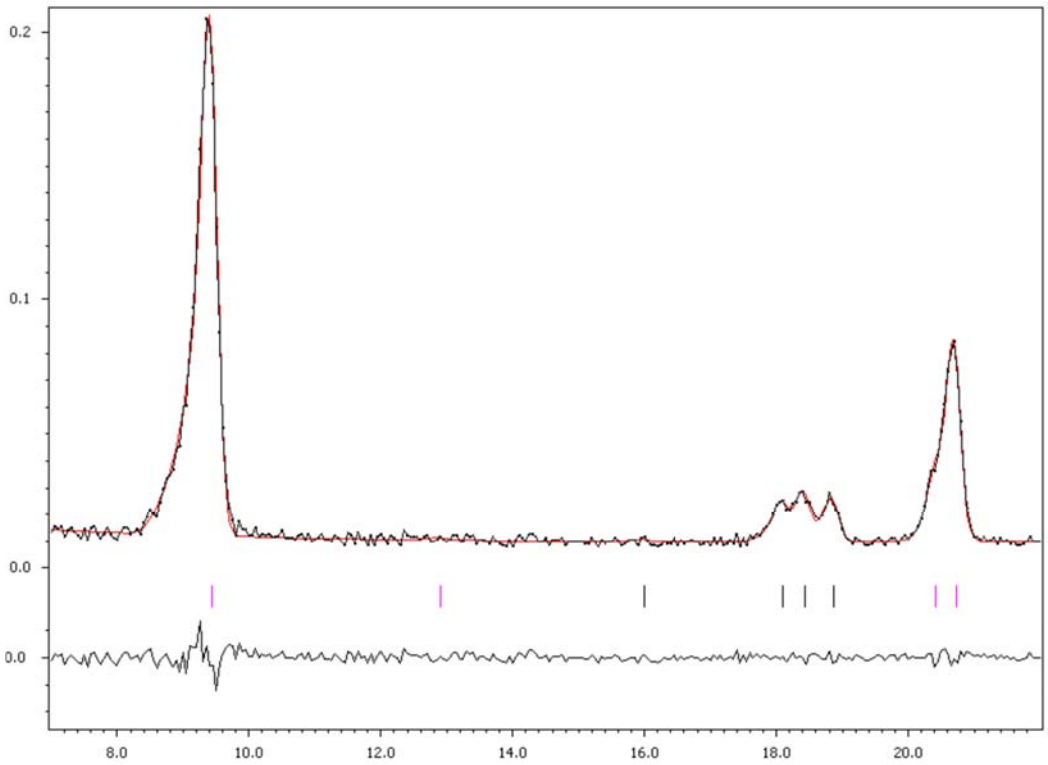
5. ***Refining of profile parameters by le Bail technique.***
   [On the screen: Information]
   NEXT;
   [On the screen: Refinement of the powder profile by the le Bail algorithm]
   "Show powder profile"
   The background is relatively smooth and it can be described without introducing manual

background.
"Quit";
"Edit profile parameters";
[On the screen: Powder options]
In 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 10
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~7.8%.
"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 LY; OK; YES to the question about rewriting;
"Run Refine"
The refinement should converge to Rp~5.9%.
"Show powder profile"; Select the region 2θ from 7 to 22 degs – use the button "X exactly".



*The first peak has a strong asymmetry which for neutron and synchrotron cases can be described by the correction for divergence as introduced by Finger-Cox-Jephcoat, J. Appl. Cryst. (1994). **27**, 892-900.*
"Quit"
"Edit profile parameters"
[On the screen: Powder options]

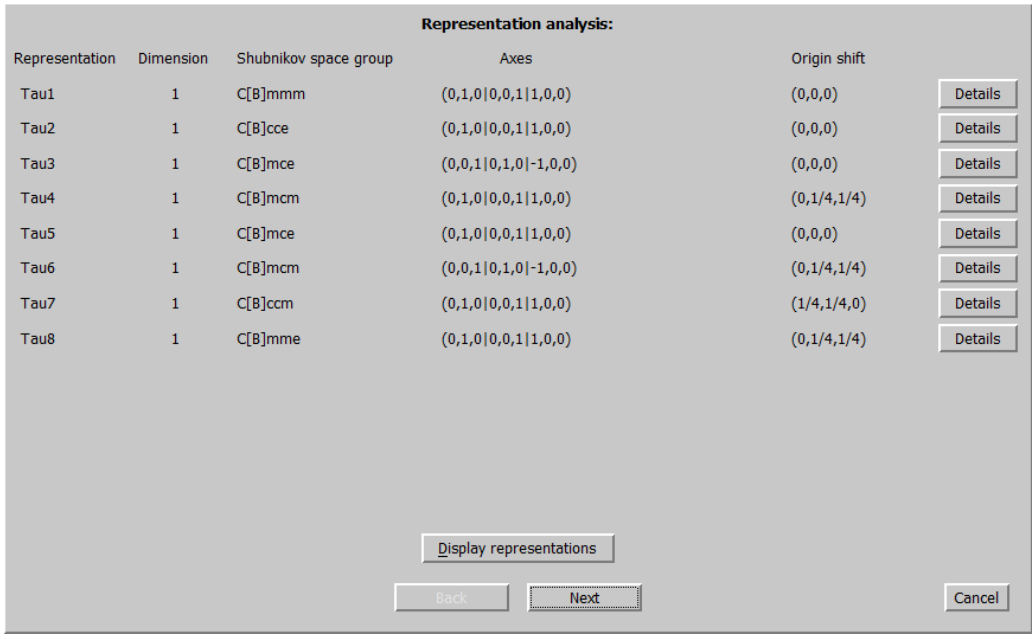
In the page “Asymmetry” select “correction by divergence”
Activate refinement of HpS/L and HmS /L keeping their default values
OK; YES to save changes
“Run Refine”;
The refinement should end with Rp~4.7%.
“Show powder profile”; Select the region 2θ from about 7 to 22 degs.



The correction considerably improved the profile
“Quit”; NEXT

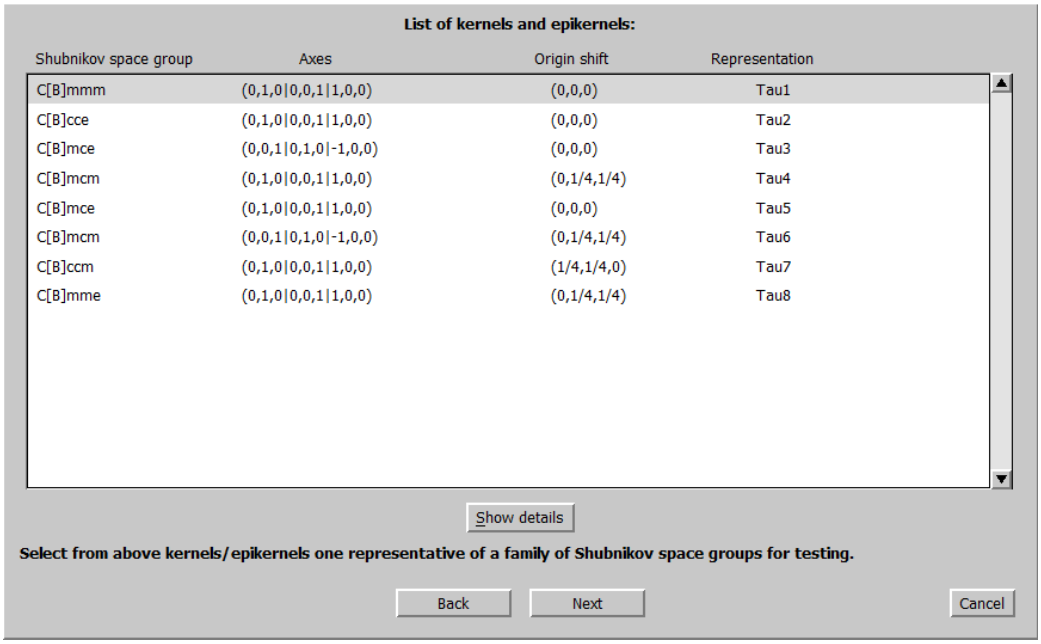
6. ***Refinement of the scale parameter for the nuclear structure***
[On the screen: Refinement of the nuclear structure by the Rietveld method]
“Edit refinement commands”; On the page “Various” select “Fixed commands”;
Fix all parameters for all atoms (“*” for “Atoms/Parameters” textbox); “Add”; OK; OK;
*The command will appear in the panel as “fixed all *”*
“Run Refine”
The refinement should converge to Rp~11.6%, R(obs)~3.6%.
NEXT

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 non-paramagnetic ordering. The “details” buttons provide more information about an individual irrep and its connection to the kernel magnetic symmetry.



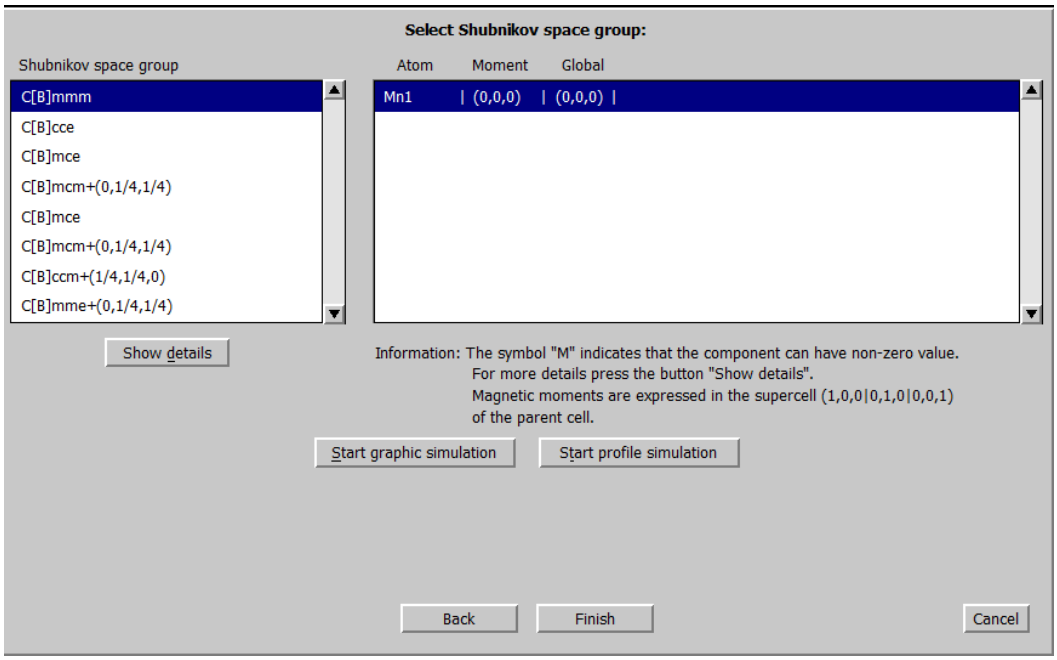
NEXT

[On the screen: List of kernels and epikernels]



NEXT

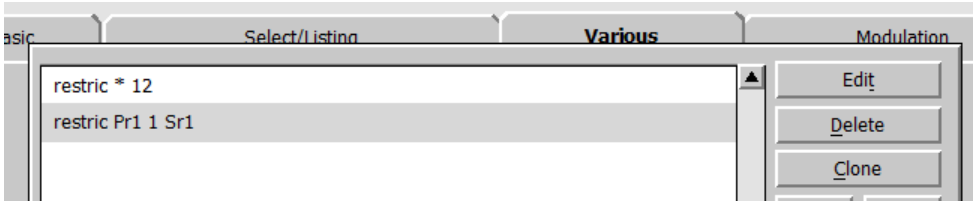
[On the screen: Select Shubnikov space group]



*This form shows that only for Shubnikov space group **№** 2, **№** 4, **№** 6 and **№** 8 allows non-zero magnetic moment for the Mn1 atom.In the following we shall test all four possibilities. Pr1 Sr1*

7. ***Refinement in the Shubnikov space group C[B]cce (№ 2 in the list)***

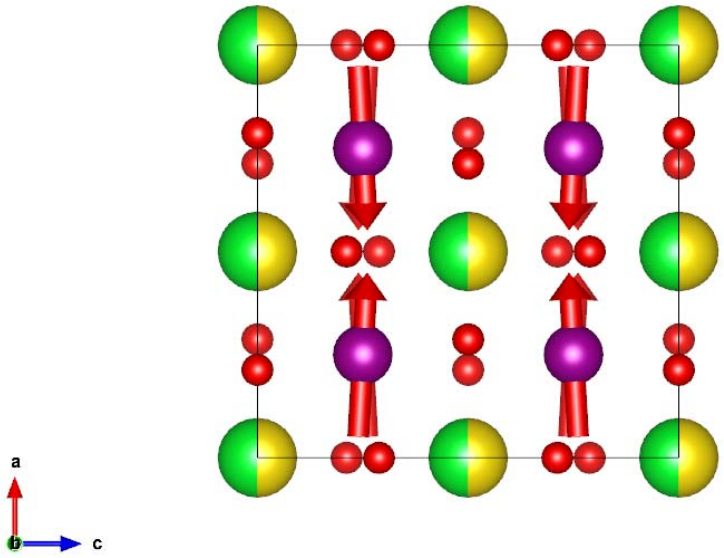
[On the screen: Select Shubnikov space group]
Select the second line in the left panel; FINISH
YES to the question "Do you want to continue with the last kernel/epikernel";
[On screen: Select structure name]
Use the default name i.e. PrSrMnO_01; OK
After selection of the actual Shubnikov group you can use either the cell and origin selection as follows from the setting of the parent structure or you can apply transformation to get a standard setting.
NO to the question "Do you want to apply this transformation?".
Right click on the icon "Refine"; Select the "Basic" and change the "Number of cycles" to 100 and the "Damping factor" to 0.1;
Select the page "Various" and use the button "Restrictions"
Check that all atoms are restricted to have identical ADP parameters and not restricted occupancies;
Restrict atoms Pr1 and Sr1, occupying the same position in the nuclear structure: identical Coordinates ADP parameters and "keep overall sum" for occupancies; Add;



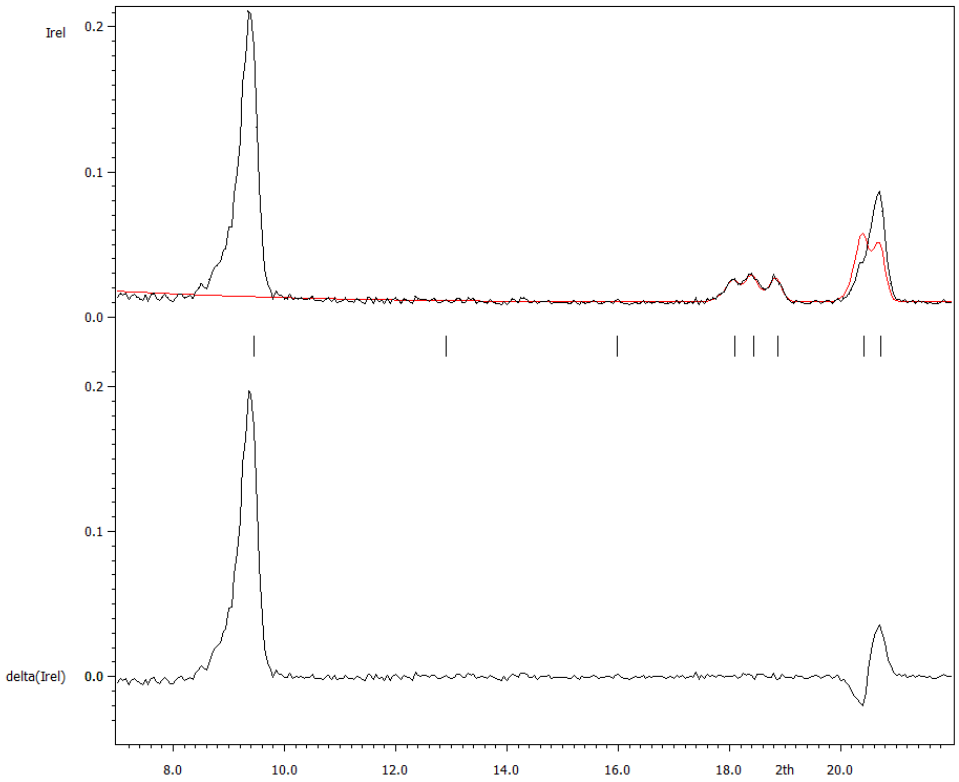
OK; "Yes+start";

Refinement converges with Rp~10.1%, R(obs)~3.7%.
This refinement is very unstable.

Double click on the icon "Plot structure" and select Vesta to draw the structure:

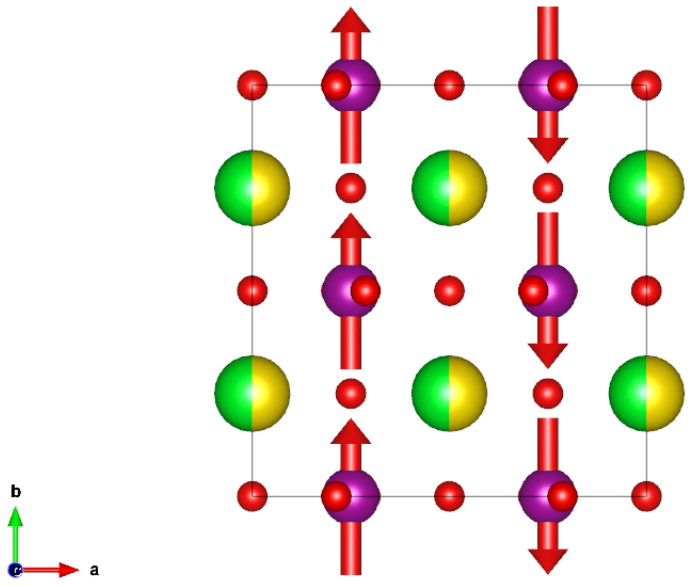


Use "Tools → Powder → Profile viewer" and check fit of the calculated powder profile:

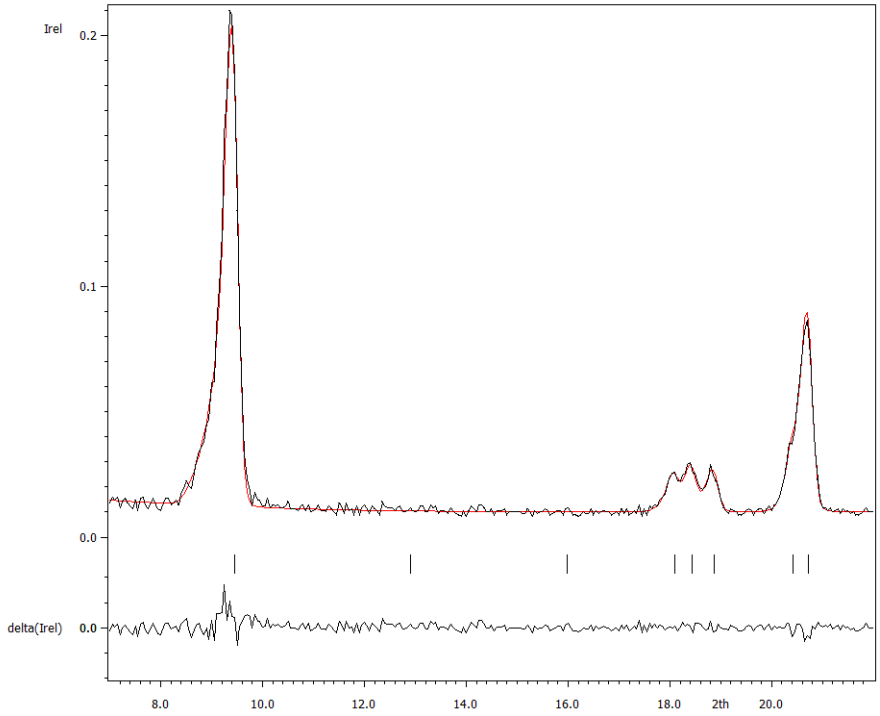


The model cannot explain the peak at (1,0,0,0)
"Quit"

8. ***Refinement in the Shubnikov superspace group C[B]mcm with the origin shift (0,1/4,1/4) (№ 4 in the list)***
[On screen: Basic window]
"File → Structure → History" and select the parent structure PrSrMnO; OK;
[On screen: Basic window]
"Tools → Special tools → Representation analysis for magnetic structures";
[On screen: Representation analysis]
NEXT; NEXT; Select the fourth Shubnikov superspace group "*C[B]mcm+(0,1/4,1/4)";* FINISH
YES to the question "Do you want to continue with the last kernel/epikernel?";
[On screen: Select structure name]
Use the default name i.e. PrSrMnO_02; OK
No to the question "Do you want to apply this transformation".
Right click on the icon "Refine"; Select the "Basic" and change the "Number of cycles" to 100 and the "Damping factor" to 0.1;
Select the page "Various" and use the button "Restrictions"
Check that all atoms are restricted to have identical ADP parameters and not restricted occupancies;
Restrict atoms Pr1 and Sr1, occupying the same position in the nuclear structure: identical Coordinates ADP parameters and "keep overall sum" for occupancies; Add;
OK; OK; "Yes+start";
Refinement converges with Rp~6.0%, R(obs)~3.2%.
Double click on the icon "Plot structure" and select Vesta to draw the structure::



Use “Tools → Powder → Profile” viewer and check fit of the calculated powder profile:



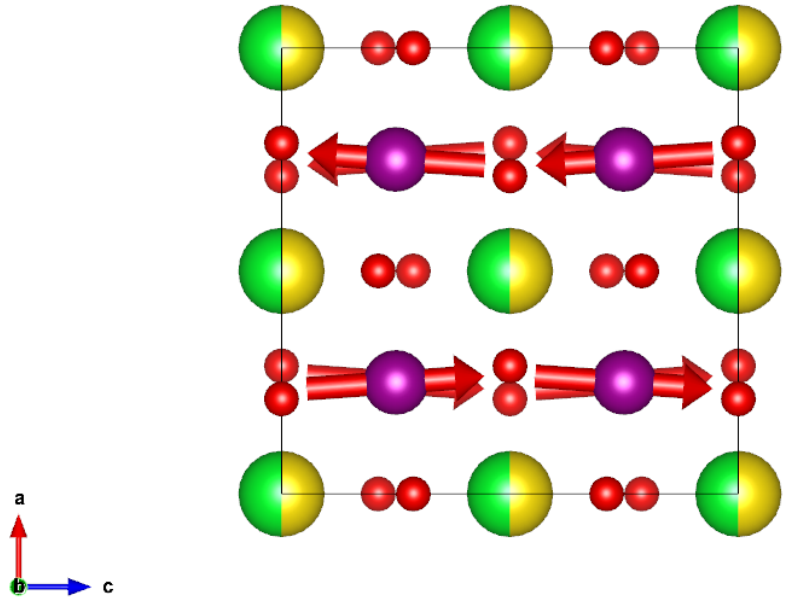
The profile fit is very good.

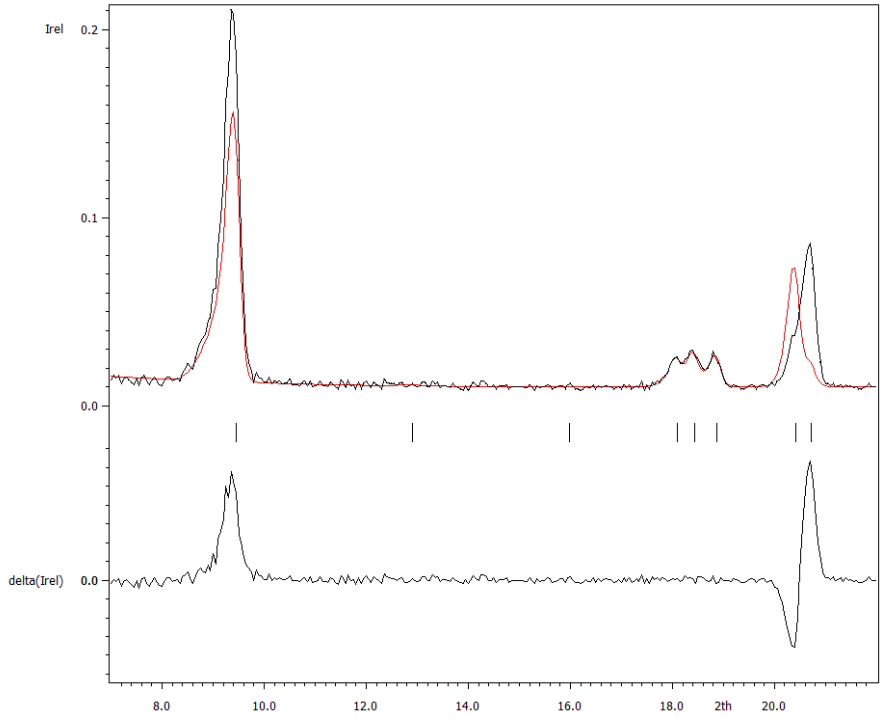
9. ***Refinement in the Shubnikov superspace group C[B]mcm with the origin shift (0,1/4,1/4) (№ 6 in the list)***

   The symbol in the table is the same as for the № 4 but both epikernels are generally different. Compare details for both. The first one has in the original setting the symbol A[C]mma and the second one A[C]mam. Both give the same symbol when transformed to the standard setting.

   Make the same steps and calculation as in the previous test.

   Rp~8.1%, R(obs)~ 3.9%.



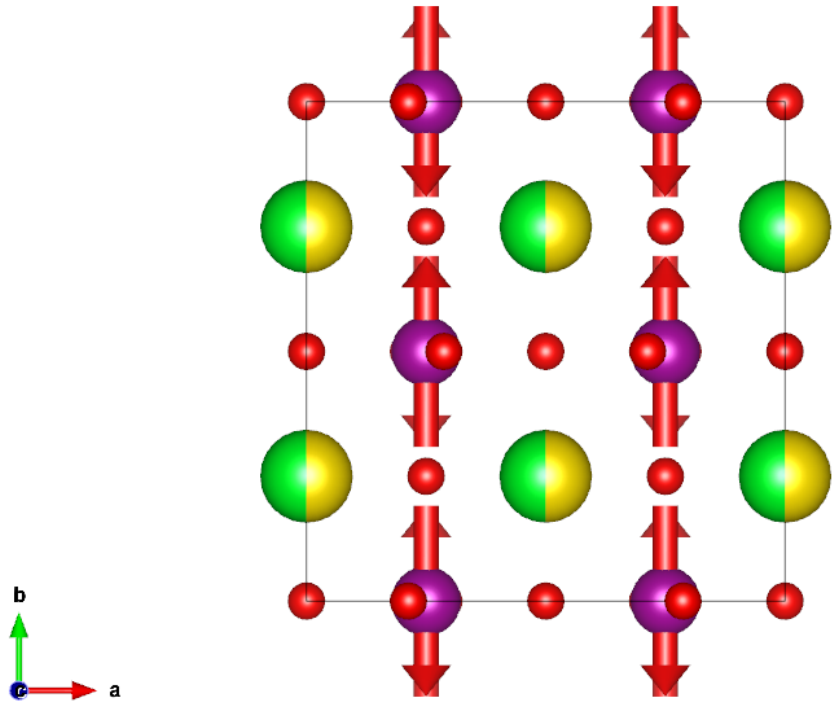


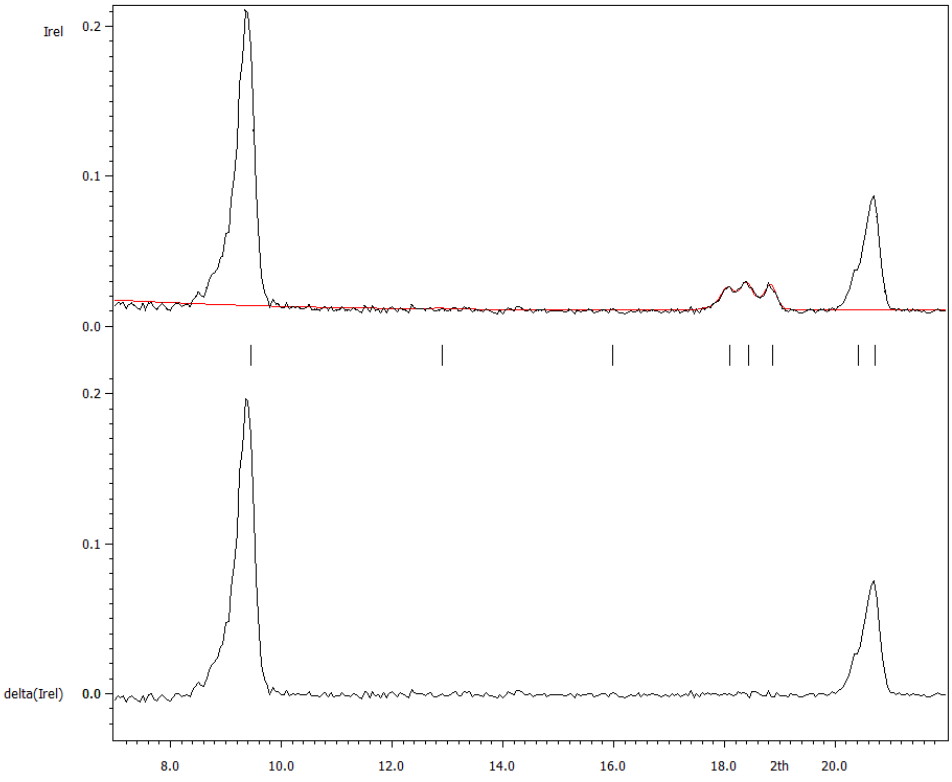
10. Refinement in the Shubnikov superspace group C[B]mme+(0,1/4,1/4) (№ 8 in the list)

Make the same calculation for the last Shubnikov superspace group with sin wave (Line eight in "Select Shubnikov space group" dialogue).

Rp~11.5%, R(obs)~ 3.1%.

This refinement is very unstable.


b
a


Irel
0.2
0.1
0.0
0.2
0.1
delta(Irel)
0.0
8.0
10.0
12.0
14.0
16.0
18.0
2th
20.0

Example 12.4: $MnWO_4$

Incommensurate magnetic structure

Revised: 30 July 2014

Powder data measured the ILL neutron source at 10 K

For more details: L. Meddar, M. Josse, P. Deniard, C. La, G. Andre, F. Damay, V. Petříček, S. Jobic, M.H. Whangbo, M. Maglione & C. Payen: *Effect of Nonmagnetic Substituents Mg and Zn on the Phase Competitionin the Multiferroic Antiferromagnet $MnWO_4$*
Chemistry of Materials, **21**, (2009), 5203-5214.

Input files: MnWO4.dat (powder profile data)
MnWO4_nuclear.cif (nuclear structure)

1. ***Creating new jobname***
   Start Jana2006
   "File → Structure → New" opens a file manager
   Left pane: locate directory with input files
   Right pane: double-click MnWO4

2. ***Import Wizard***
   Select "Magnetic: nuclear model from CIF", NEXT
   Right pane: select the input file MnWO4_nuclear.cif

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: -0.209 0.5 0.453
   Select atom type Mn; check "Use as a magnetic atom"; select "Magnetic form factor <j0>" Mn2+; NEXT; FINISH
   The parent structure is created

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 "MnWO4.dat" and the data format is "Free format of 2th, I, [sig(I)]"; NEXT
   [On the screen: Complete/correct experimental parameters];
   Fill used wavelength 2.4226; NEXT; FINISH
   [On the screen: Data repository]
   OK; YES to "Do you want to accept made changes?"

5. ***Refining of profile parameters by the 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]
In page "Cell" activate refinement of a, b, c parameters and beta angle;
Activate refinement of component q1 and q3 of the modulation vector. The q2 component is fixed for planar monoclinic superspace groups to 0.5;
Check that the maximal satellite index is set to 1;
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 enlarge the number of used Legendre polynomials to 20
OK; YES to save the changes
"Edit refinement commands"; In the page "Basic" change the number of cycles to 100 and the damping factor to 0.5; OK
"Run Refine";
The refinement should converge to Rp~5.2%.
"Edit profile parameters";
[On the screen: Powder options]
In the page "Profile" change activate refinement of GU and GV
Change the peak-shape function to pseudo-Voigt and activate refinement LY
OK; YES to save the changes
"Run Refine"
The refinement should converge to Rp~3.4%.
NEXT; YES to leave the form for le Bail refinement

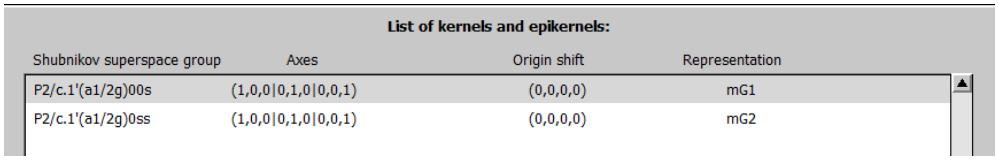
6. ***Refinement of the scale parameter for the nuclear structure***
[On the screen: Refinement of the nuclear structure by the Rietveld method]
"Edit refinement commands"; On the page "Various" select "Fixed commands";
Fix all parameters for all atoms (wild character *); "Add"; OK; OK
"Run Refine"
The refinement should converge to Rp=12.8%, R(obs)=5.5%.
NEXT; YES to leave the form for Rietveld 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 non-paramagnetic ordering. The "details" buttons provide more information about an individual irrep and its connection to the kernel magnetic symmetry.

Representation analysis:

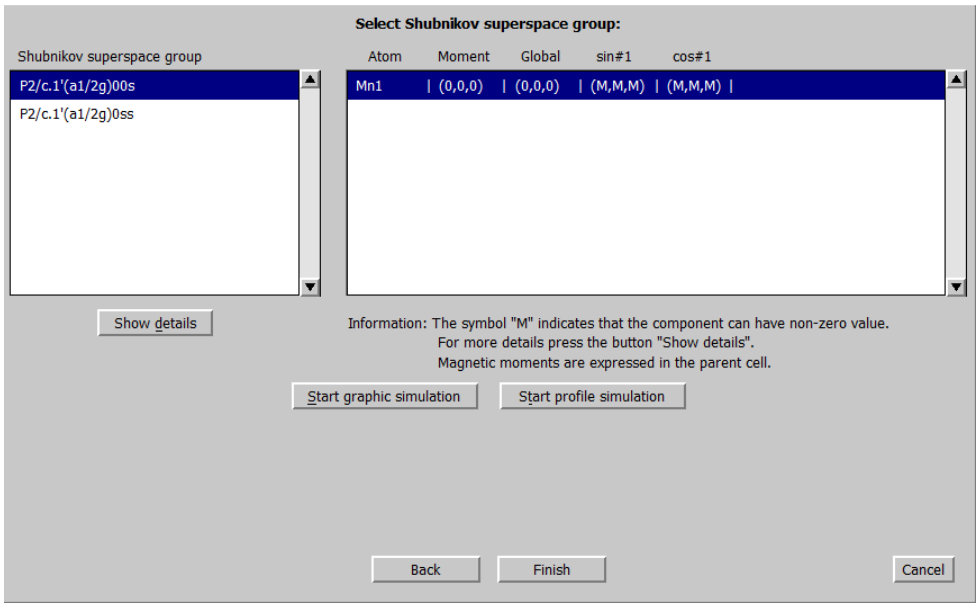
Representation	Dimension	Shubnikov superspace group	Axes	Origin shift	
mG1	2	P2/c.1'(a1/2g)00s	(1,0,0|0,1,0|0,0,1)	(0,0,0,0)	Details
mG2	2	P2/c.1'(a1/2g)0ss	(1,0,0|0,1,0|0,0,1)	(0,0,0,0)	Details

NEXT
[On the screen: List of kernels and epikernels]



NEXT

[On the screen: Select Shubnikov superspace group]



Notice that for both SSG's there are non-zero amplitudes for sine and cosine waves. However only three amplitudes are independent as can be found from "Details":

```
*****************************************************************************
* List of relationships between magnetic moments of individual atoms *
*****************************************************************************

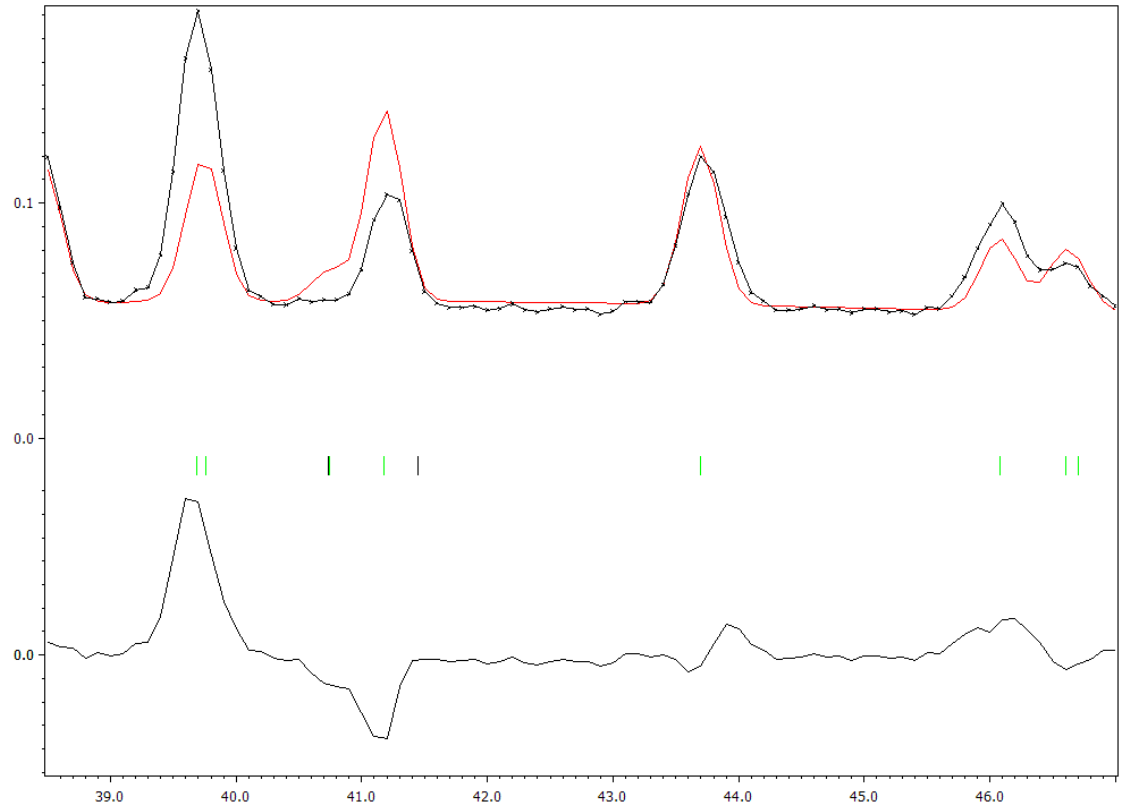
Atom: Mn1
=========
mx0=0                          mxsin1 ... free              mxcos1=1.6787*mxsin1
my0=0                          mysin1 ... free              mycos1=-0.59571*mysin1
mz0=0                          mzsin1 ... free              mzcos1=1.6787*mzsin1
```

The cosine amplitudes have generally non-zero values but they are related with the sine amplitudes. However the coefficients are functions of y coordinate of the Mn1 atom, which means that these restrictions are non-linear. Later we shall see how these restrictions can be simplified.
In this case both possibilities give similar diffraction patterns and we cannot decide at this stage which solution makes a better fit.

Note: The presence of a non-zero rational modulation vector leads to a specific problem of the origin selection. While for the space group $P\,2/c$ *the c glide planes located at y=0 and y=1/2 are equivalent, in the SSG* $P\,2/c\,.1'(\alpha\,1/2\,\gamma)00\,s$ *the first one is connected with zero shift along the internal space but the second one 1/2 shift. This means that for solution of modulated structure when starting from the model solved and refined from the main reflections, the origin selection must be reconsidered and two models shifted by 1/2 in y must be tested. Another possibility is to use non-standard SSG symbol* $P\,2/c\,.1'(\alpha\,1/2\,\gamma)0\,ss$ *which would generate reverse order of c glide planes. The second method is used in this example.*

8. ***Refinement in the first Shubnikov superspace group – P2/c.1'(a1/2g)00s***

[On the screen: Select Shubnikov space group]
Select the first line in the left panel; FINISH
YES to the question "Do you want to continue with the last kernel/epikernel";
[On screen: Select structure name]
Use the default name i.e. MnWO4_01; 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;
Restrict atoms Zn1 and Mn1, occupying the same position in the nuclear structure: identical Coordinates + Modulations + ADP parameters and "keep overall sum" for occupancies
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~5.6% and Bragg R factors R(obs)=5.9%, 3.5% and 9.6% for all, main and satellite reflections, respectively.
Use "Tools → Powder → Profile" viewer and check fit of the calculated powder profile and focus to the interval (38.5,47):



Quit

9. ***Refinement in the second Shubnikov superspace group – P2/c.1'(a1/2g)0ss***

[On the screen: Basic window]

"File → Structure → History" and select the parent structure MnWO4; OK;

[On screen: Basic window]

"Tools → Special tools → Representation analysis for magnetic structures";

Continue with representation analysis; OK;

[On the screen: Representation analysis]

NEXT; NEXT; Select P2/c.1'(a1/2g)0ss; FINISH;

YES to the question "Do you want to continue with the last kernel/epikernel?";

[On the screen: Select structure name]

Use the default name; OK

[On the screen: Basic window]

Right click on the icon "Refine"; Select the "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;

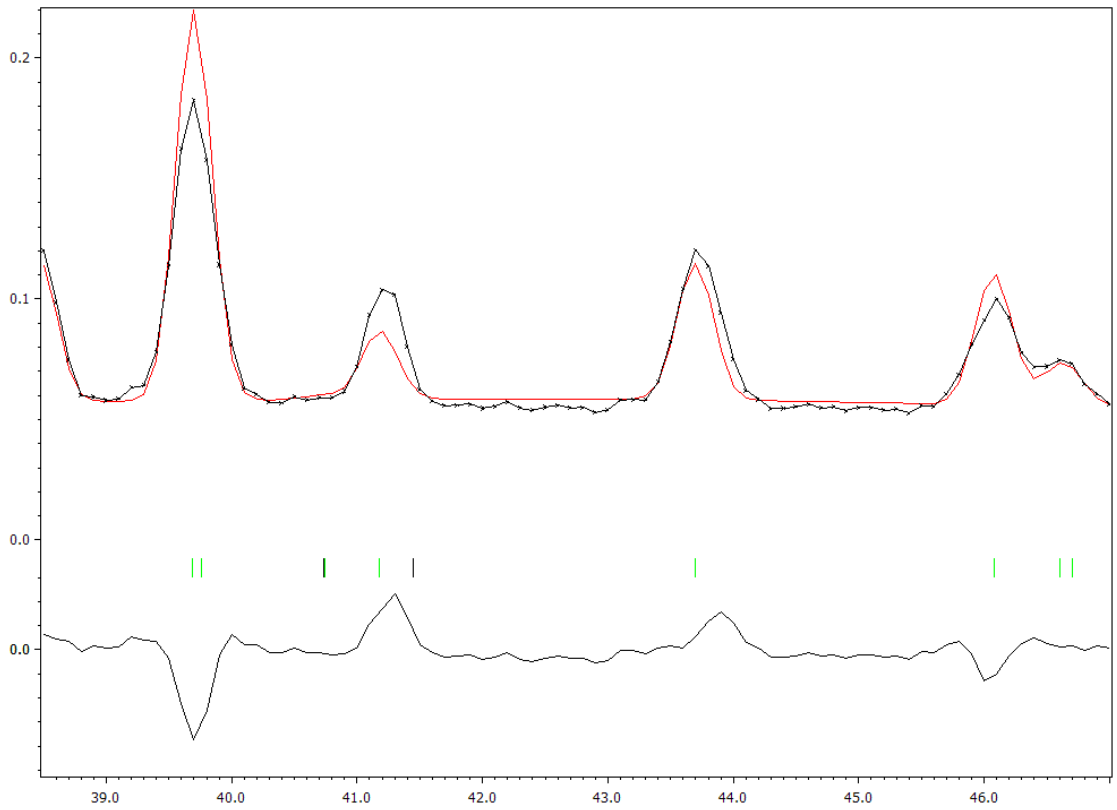
Restrict atoms Zn1 and Mn1, occupying the same position in the nuclear structure: identical Coordinates + Modulations + ADP parameters and "keep overall sum" for occupancies

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~5.0% and Bragg R factors R(obs)=4.3%, 3.0% and 6.4%

for all, main and satellite reflections, respectively.
Use "Tools → Powder → Profile" viewer and check fit of the calculated powder and focus to the interval (38.5,47):



The second solution gives significantly better profile fit and also better Bragg R values.
Quit

10. Transforming the final solution to the supercentered cell.

[On the screen: Basic window]
Run EditM50, go to "Symmetry" and press button "RUN Stokes & Campbell SSG test"
This command connects the web page http://stokes.bye.edu/iso/findssg.html using the program "wget", which is distributed with Jana2006 under GNU licence.

With active "wget" the web tool immediately returns the result:

findssg

Input setting

Centering
none
Operators
(-x,y,-z+1/2,y-t); (-x,-y,-z,-t+1/2); (x,-y,z+1/2,-y+t+1/2); (x,y,z,t)

Standard settings

Superspace group: 13.1.3.2 P2/b(a,b,1/2)00 [Y:1.47]
Bravais class: 1.3 P2/m(a,b,1/2) [JJdW:1.3]
Transformation to supercentered setting: A1=a1, A2=a2, A3=2a3+a4, A4=a4

BASIC SPACE GROUP SETTING
Modulation vectors: q1'=(a,b,1/2)
Centering: (0,0,0,0)
Non-lattice generators: (-x,-y+1/2,z,z-t); (x,y+1/2,-z,-z+t)
Non-lattice operators: (x,y,z,t); (-x,-y+1/2,z,z-t); (-x,-y,-z,-t); (x,y+1/2,-z,-z+t)

SUPERCENTERED SETTING
Modulation vectors: Q1'=(A,B,0), where A=a, B=b
Centering: (0,0,0,0); (0,0,1/2,1/2)
Non-lattice generators: (-X,-Y+1/2,Z,-T); (X,Y+1/2,-Z,T)
Non-lattice operators: (X,Y,Z,T); (-X,-Y+1/2,Z,-T); (-X,-Y,-Z,-T); (X,Y+1/2,-Z,T)
Reflection conditions: HKLM:L+M=2n; HK0M:K=2n

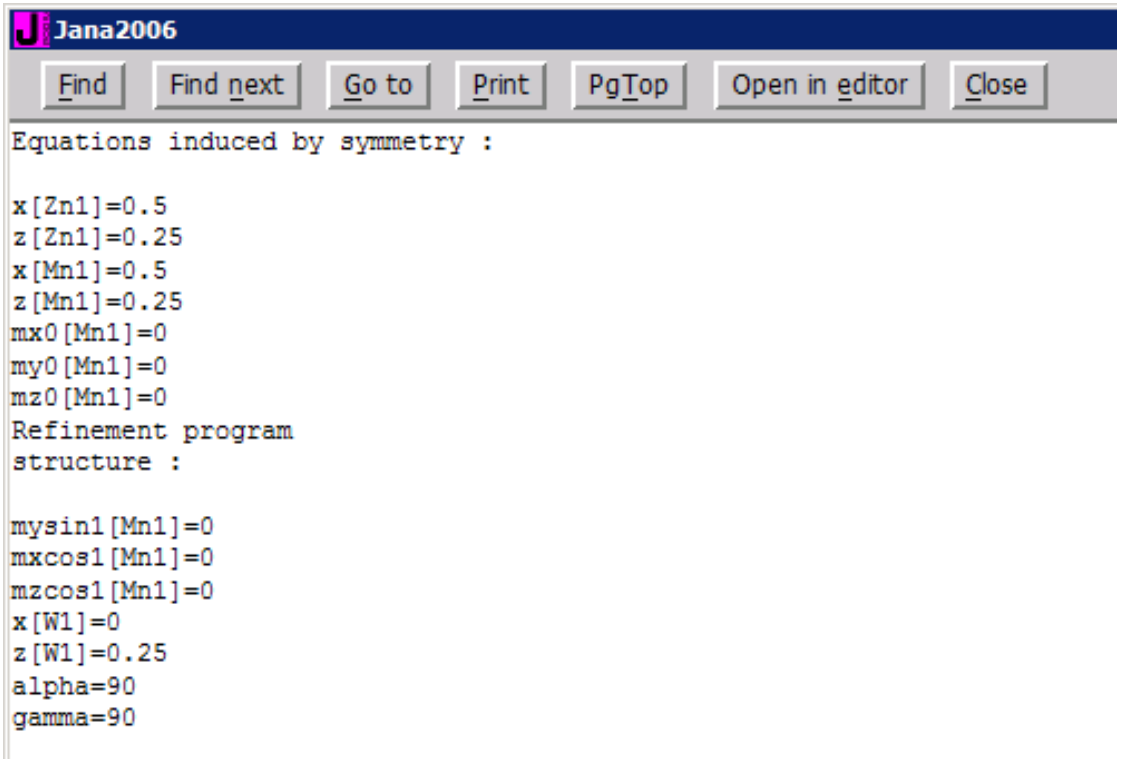
Note: this tool uses "c" as the monoclinic axis.
You can see that there are two different settings: the "basic space group setting" in the original unit cell and the "supercentered setting", which uses doubled monoclinic axis and additional centering vector. The latter setting leads to a complete separation of internal and external space.

Close your internet browser and editor; ESC in Editm50 window;
"Tools→Transformation→Transform to the X-centered cell";
Create new structure e.g. "MnWO4_02_X";
YES to the question "Do you want to continue with the new structure?";

11. Refinement in the supercentered setting

[On the screen: Basic window]
Run EditM50, go to "Symmetry"; Check the new SSG symbol; "X" stands for the supercentered setting. For more details about centering vectors expand the list of centering symbols at "cell centering" and re-select the symbol X
The generated supercell centering (0,1/2,0,1/2) is in accordance with the values from "findSSG" (except that the monoclinic axis is now "b")
ESC; ESC;
Right click on the icon "Refine"; Select the "Basic" and change the "Number of cycles" to 100 and the "Damping factor" to 0.1;
Select the page "Various"; Use the button "Restrictions" and restrict all atoms to have identical ADP parameters and not restricted occupancies; Add;
Restrict atoms Zn1 and Mn1, occupying the same position in the nuclear structure: identical

Coordinates + Modulation + ADP parameters and “keep overall sum” for occupancies;
Add; OK
Use button “Fixed commands” and fix coordinates for all atoms, i.e. *.
OK; OK; “Yes+start”;
The refinement should finish with Rp~5.0% and Bragg R factors R(obs)=4.3%, 3.0% and 6.4% for all, main and satellite reflections, respectively.
YES to the question “Open the listing”; Check the symmetry restrictions:



Now the y component of the sine wave and x and z components of the cos wave are fixed to zero values. This setting makes a full separation of internal and external space (before the transformation some wave components depended on the y coordinate).

Example 12.6: ISODISTORT

Practical introduction to Jana2006-ISODISTORTcommunication

Revised: 31 July 2014

Input files: Ba5Co5.m40, Ba5Co5.m41, Ba5Co5.m50, Ba5Co5.m90

12. Creating CIF file from the parent structure

[On the screen: Basic window]

Start Jana2006

Start "File → Structure → Open" and open the structure Ba5Co5;

"File → CIF utilities → Make CIF file";

[On the screen: Select Hermann-Mauguin symbol to be used:]

Select the short symbol P63/mmc;

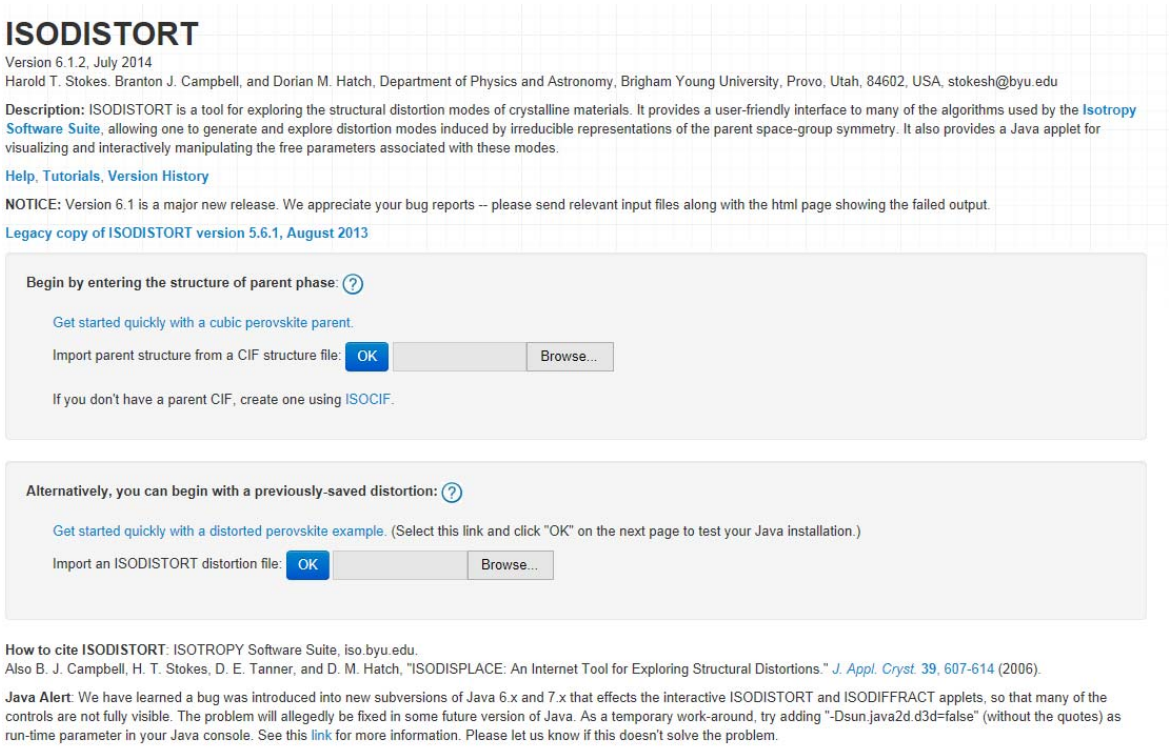
13. Start ISODISTORT home page

[On the screen: Basic window]

"Tools → Connect through internet to → Stokes, Campbell & Hatch ISODISTORT";

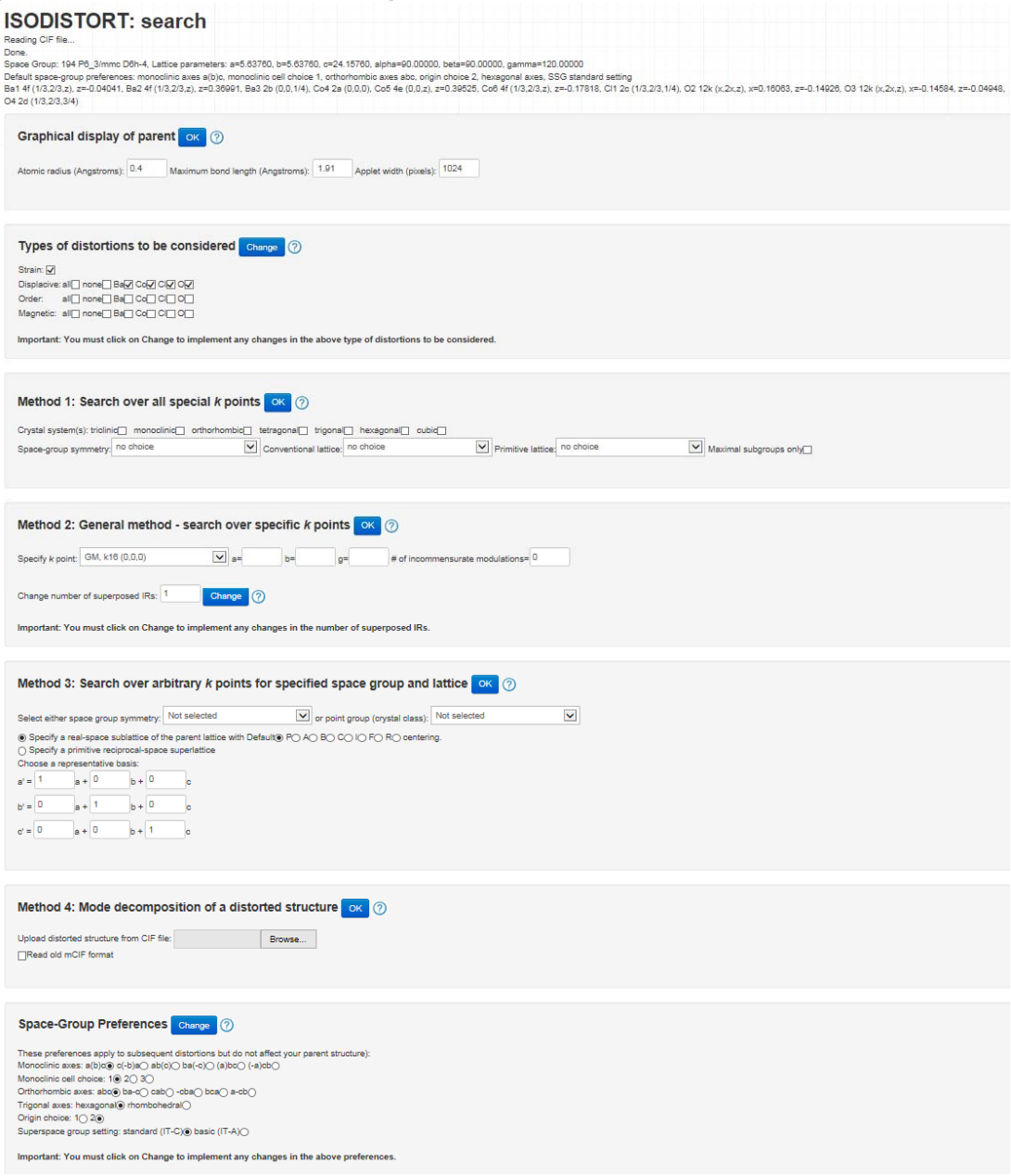
Your Internet default browser should open the page:

http://stokes.byu.edu/iso/isodistort.php

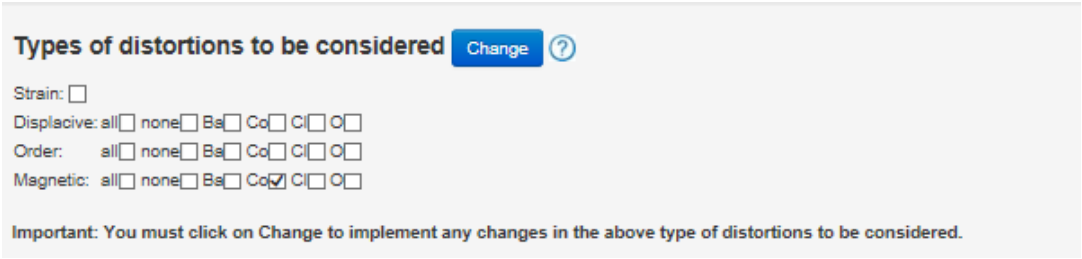


Browse for the CIF file created in the previous step; OK

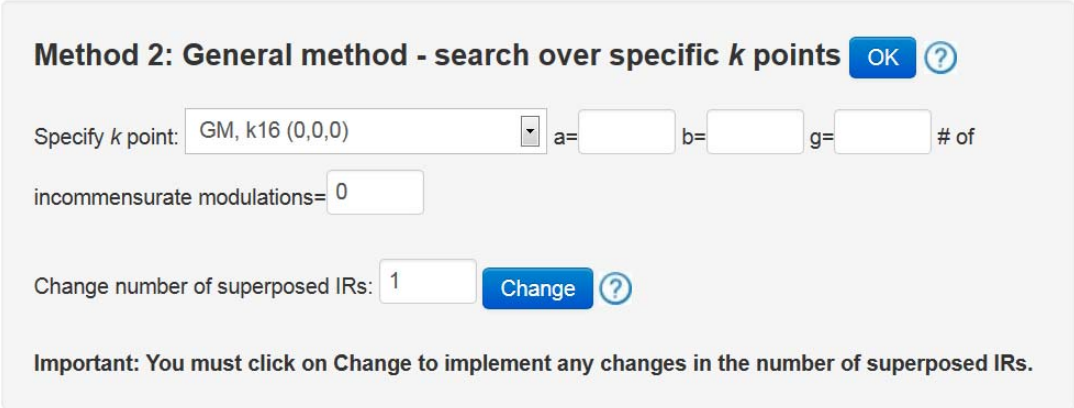
[On the screen: ISODISTORT search]



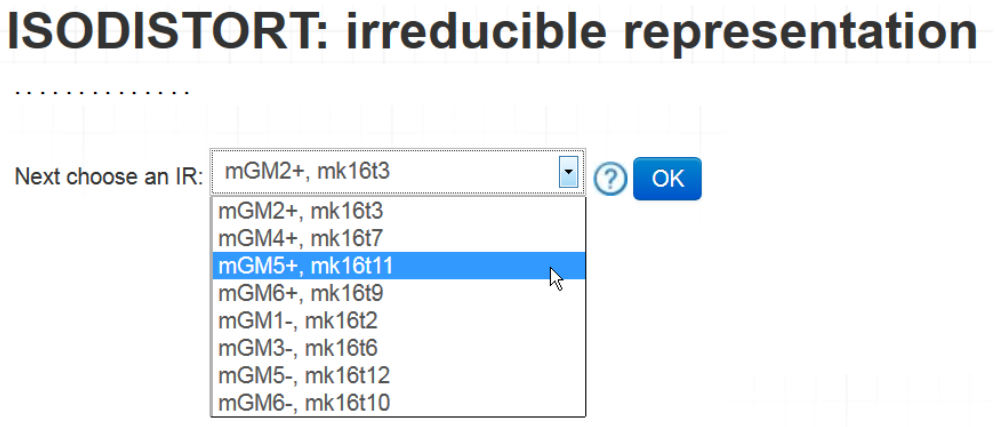
Define distortions to be considered:
In our case we are interested just in magnetic ordering;



Press Change;
In the box for "Method 2" select GM k16(0,0,0), which means k=(0,0,0):



Press OK;
Select irreducible representation
[On the screen: ISODISTORT irreducible representation]



Comparing with "Representation analysis" list of Jana2006, only irreducible representations allowing non-zero magnetic moments are listed. In the next step we will demonstrate two dimensional irreducible representation mGM5+.
Select the representation mGM5+ and press OK;

Represantation analysis of Jana2006 (for comparison):

Representation analysis:

Representation	Dimension	Shubnikov space group	Axes	Origin shift	
mGM1+	1	P63/mmc	(1,0,0|0,1,0|0,0,1)	(0,0,0)	Deta
mGM1-	1	P63/m'm'c'	(1,0,0|0,1,0|0,0,1)	(0,0,0)	Deta
mGM2+	1	P63/mm'c'	(1,0,0|0,1,0|0,0,1)	(0,0,0)	Deta
mGM2-	1	P63/m'mc	(1,0,0|0,1,0|0,0,1)	(0,0,0)	Deta
mGM3+	1	P63'/m'mc'	(1,0,0|0,1,0|0,0,1)	(0,0,0)	Deta
mGM3-	1	P63'/mm'c	(1,0,0|0,1,0|0,0,1)	(0,0,0)	Deta
mGM4+	1	P63'/m'm'c	(1,0,0|0,1,0|0,0,1)	(0,0,0)	Deta
mGM4-	1	P63'/mmc'	(1,0,0|0,1,0|0,0,1)	(0,0,0)	Deta
mGM5+	2	P21/m	(0,1,0|0,0,1|1,0,0)	(0,0,0)	Deta
mGM5-	2	P21/m'	(0,1,0|0,0,1|1,0,0)	(0,0,0)	Deta
mGM6+	2	P21'/m	(0,1,0|0,0,1|1,0,0)	(0,0,0)	Deta
mGM6-	2	P21'/m'	(0,1,0|0,0,1|1,0,0)	(0,0,0)	Deta

Display representations

[On the screen: ISODISTORT: order parameter direction]

ISODISTORT: order parameter direction

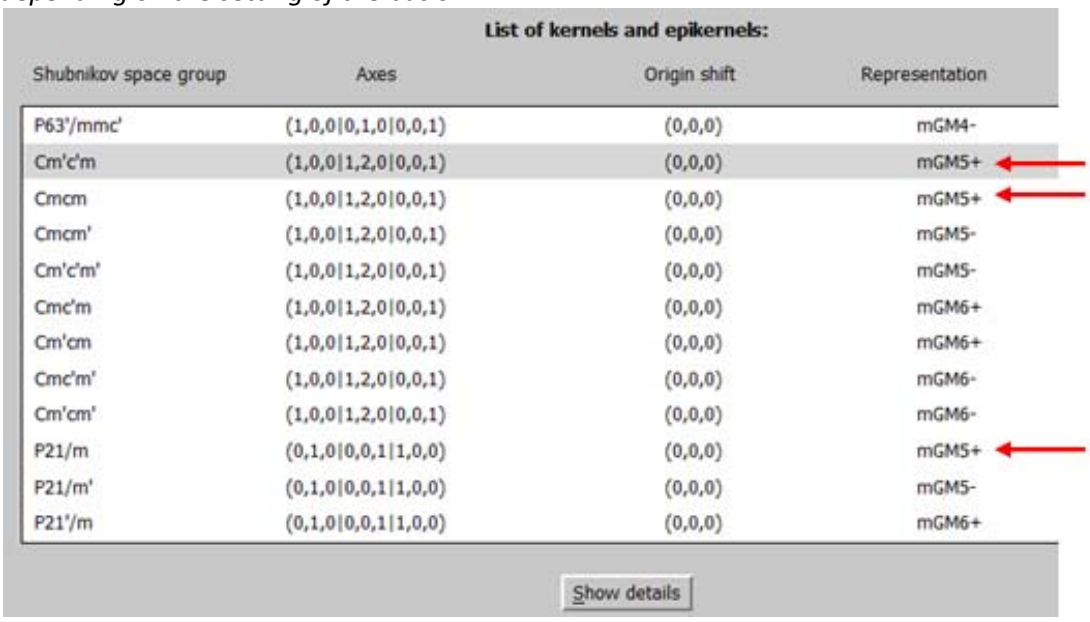
Space Group: 194 P6_3/mmc D6h-4, Lattice parameters: a=5.63780, b=5.63780, c=24.15830, alpha=90.00000, beta=90.00000, gamma=120.00000
Default space-group preferences: monoclinic axes a(b)c, monoclinic cell choice 1, orthorhombic axes abc, origin choice 2, hexagonal axes, SSG standard setting
Ba1 4f (1/3,2/3,z), z=-0.04041, Ba2 4f (1/3,2/3,z), z=0.36991, Ba3 2b (0,0,1/4), Co4 2a (0,0,0), Co5 4e (0,0,z), z=0.39525, Co6 4f (1/3,2/3,z), z=-0.17818, Cl1 2c (1/3,2/3,1/4), O2 12k (x,2x,z), x=0.16063 , z=-0.14926, O3 12k (x,2x,z), x=-0.14584 , z=-0.04948, O4 2d (1/3,2/3,3/4)
Irrep matrices: 2011 version for all k points
Include magnetic Co4 Co5 Co6 distortions
k point: GM, k16 (0,0,0)
IR: mGM5+, mk16t11

Finish selecting the distortion mode by choosing an order parameter direction
- P1 (a,-1.732a) 63.457 Cmcm, basis={(0,1,0),(-2,-1,0),(0,0,1)}, origin=(0,0,0), s=1, i=6, k-active= (0,0,0)
- P2 (a,0.577a) 63.462 Cm'c'm, basis={(0,1,0),(-2,-1,0),(0,0,1)}, origin=(0,0,0), s=1, i=6, k-active= (0,0,0)
- C1 (a,b) 11.50 P2_1/m, basis={(-1,0,0),(0,0,-1),(0,-1,0)}, origin=(0,0,0), s=1, i=12, k-active= (0,0,0)

OK

Here we can see the kernel and the set of epikernels (isotopic subgroups) associated with mGM5+.

List of kernels and epikernels in Jana2006 (for comparison). The order parameters may differ depending on the setting of the basis.

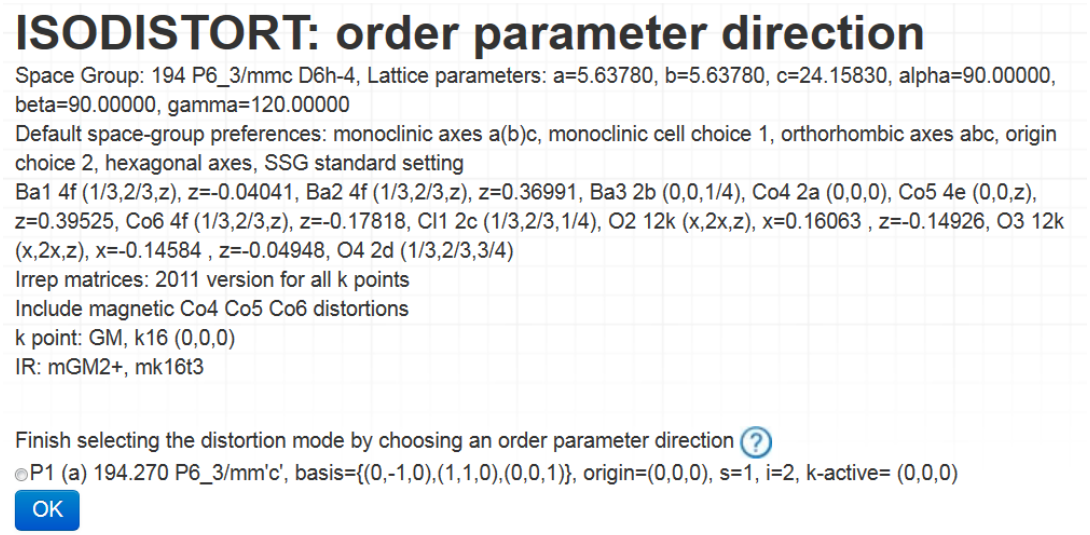


List of kernels and epikernels:

Shubnikov space group	Axes	Origin shift	Representation
P63'/mmc'	(1,0,0|0,1,0|0,0,1)	(0,0,0)	mGM4-
Cm'c'm	(1,0,0|1,2,0|0,0,1)	(0,0,0)	mGM5+
Cmcm	(1,0,0|1,2,0|0,0,1)	(0,0,0)	mGM5+
Cmcm'	(1,0,0|1,2,0|0,0,1)	(0,0,0)	mGM5-
Cm'c'm'	(1,0,0|1,2,0|0,0,1)	(0,0,0)	mGM5-
Cmc'm	(1,0,0|1,2,0|0,0,1)	(0,0,0)	mGM6+
Cm'cm	(1,0,0|1,2,0|0,0,1)	(0,0,0)	mGM6+
Cmc'm'	(1,0,0|1,2,0|0,0,1)	(0,0,0)	mGM6-
Cm'cm'	(1,0,0|1,2,0|0,0,1)	(0,0,0)	mGM6-
P21/m	(0,1,0|0,0,1|1,0,0)	(0,0,0)	mGM5+
P21/m'	(0,1,0|0,0,1|1,0,0)	(0,0,0)	mGM5-
P21'/m	(0,1,0|0,0,1|1,0,0)	(0,0,0)	mGM6+

Show details

Return one step back in your Internet browser and select the representation mGM2+; OK;
[On the screen: ISODISTORT: order parameter direction]



ISODISTORT: order parameter direction

Space Group: 194 P6_3/mmc D6h-4, Lattice parameters: a=5.63780, b=5.63780, c=24.15830, alpha=90.00000, beta=90.00000, gamma=120.00000
Default space-group preferences: monoclinic axes a(b)c, monoclinic cell choice 1, orthorhombic axes abc, origin choice 2, hexagonal axes, SSG standard setting
Ba1 4f (1/3,2/3,z), z=-0.04041, Ba2 4f (1/3,2/3,z), z=0.36991, Ba3 2b (0,0,1/4), Co4 2a (0,0,0), Co5 4e (0,0,z), z=0.39525, Co6 4f (1/3,2/3,z), z=-0.17818, Cl1 2c (1/3,2/3,1/4), O2 12k (x,2x,z), x=0.16063 , z=-0.14926, O3 12k (x,2x,z), x=-0.14584 , z=-0.04948, O4 2d (1/3,2/3,3/4)
Irrep matrices: 2011 version for all k points
Include magnetic Co4 Co5 Co6 distortions
k point: GM, k16 (0,0,0)
IR: mGM2+, mk16t3

Finish selecting the distortion mode by choosing an order parameter direction
P1 (a) 194.270 P6_3/mm'c', basis={(0,-1,0),(1,1,0),(0,0,1)}, origin=(0,0,0), s=1, i=2, k-active= (0,0,0)
OK

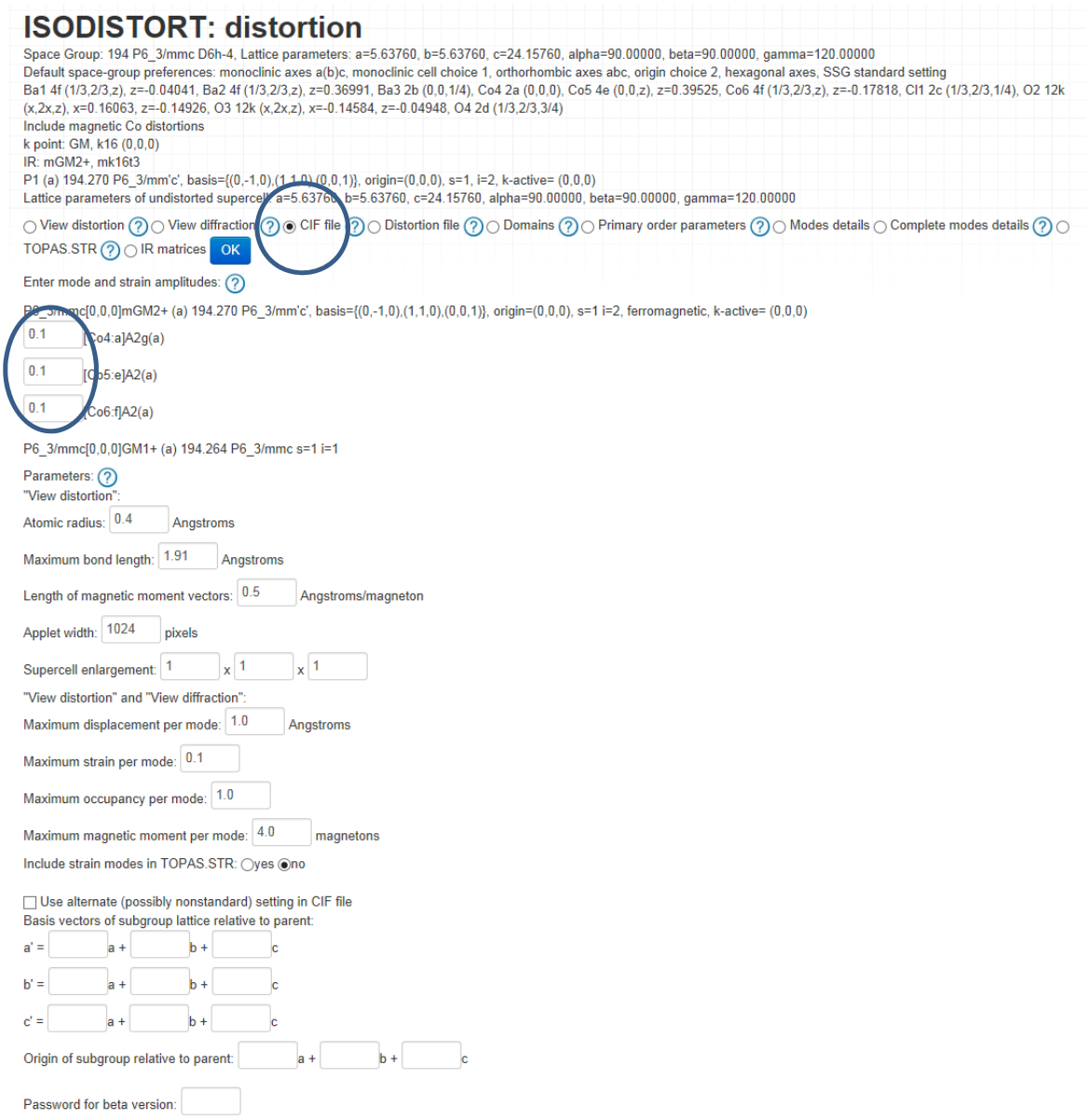
Select the listed ODP and press OK

[On the screen: ISODISTORT: distorsion]

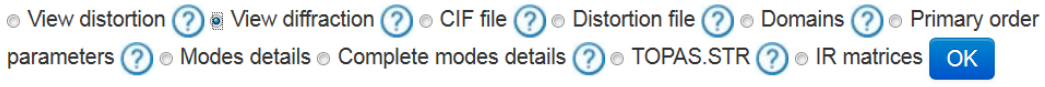
Select a small starting values (0.1) for magnetic moments of Co4, Co4 and Co6;

Select “CIF file” and press OK;

Save it as a Text Document with name GM2+ into the working directory.



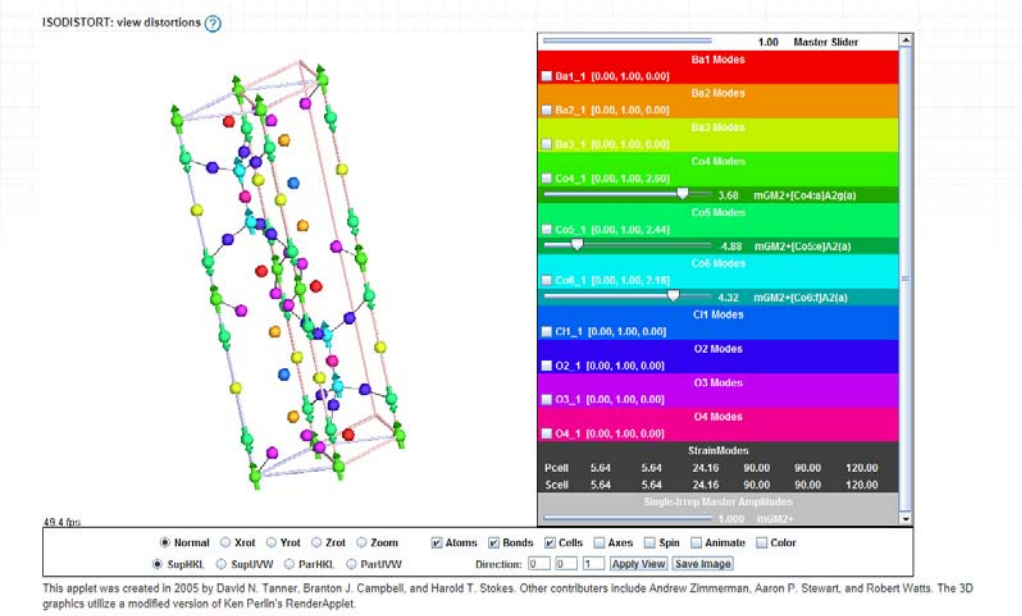
Select “View distortion” and press OK;



Make the steps related with usage of Java.

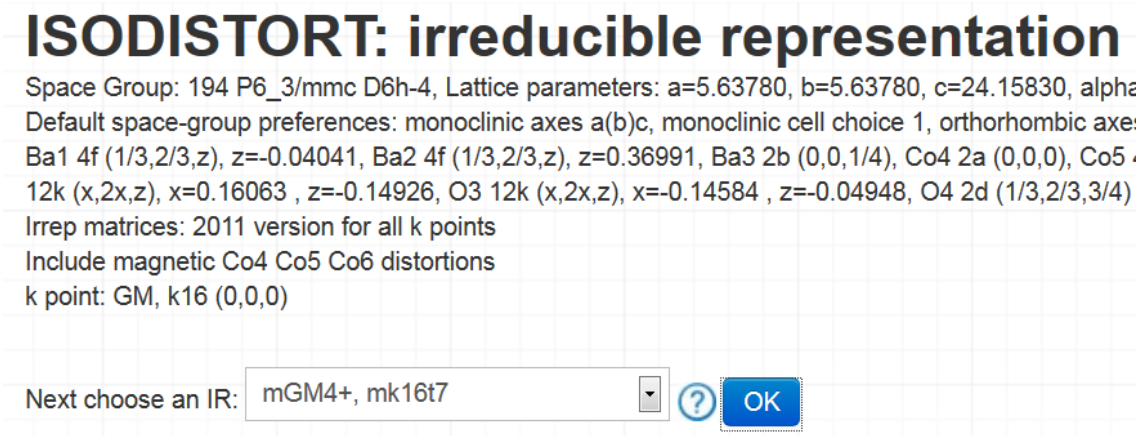
If Java offers an update please do it, otherwise the applet may be blocked. In some cases the Java security level needs to be adjusted in Java Control Panel (and the browser restarted and

the work repeated ...).



Here you can change magnetic moments of Co atoms to see mutual arrangement of magnetic moments of symmetry related atoms.

Return back to the page ISODISTORT irreducible representation and select now mGM4+



Repeat the work as for GM2+ and save the CIF file as a Text Document with name GM4+ into the working directory.
Return to Jana2006

14. *Refining the model mGM2+*

[On the screen: Basic Jana2006 window]
"File → Structure → New"; opens a file manager
On the right pane: Select (by the arrow button) structure name GM2+; OK
[On the screen: Specify type of the file to be imported]
Select "Structure: from CIF"

This opens a file manager
Caution: we cannot use "Magnetic: nuclear model from CIF" because it would create the parent phase. In our case we already have the parent phase Ba5Co5 and the structure generated by ISODISTORT is one of the test magnetic structures
On the right pane:
Uncheck "Use filter" and select the file GM2+.txt; OK;
The imported CIF file has line separators as for UNIX and this is the reason why the file must be converted to the Windows system.
YES to the question "Do you want to continue?".
NO to the question "Do you want to import data from file?";

[On the screen: Basic window]
"File → Structure → Copy in";
On the right pane: Select structure name Ba5Co5; OK
Copy in just m41 and m90 files; OK
Right click on the icon "Refine"; Select the "Basic" and change the "Number of cycles" to 100 and the "Damping factor" to 0.1;
Select the page "Various" and use the button "Restrictions";
Restrict all atoms (i.e. "*") to have identical ADP parameters and but not coordinates and occupancies; OK
On the page "Various" select "Fixed commands";
Fix coordinates for all atoms (wild character *);
OK; OK; "Yes+start";
Refinement converges with Rp~3.3%, R(obs)~8.4%.

15. Refining the model mGM4+

[On the screen: Basic Jana2006 window]
"File → Structure → New"; opens a file manager
On the right pane: Select structure name GM4+; OK
[On the screen: Specify type of the file to be imported]
Select from CIF; *opens a file manager*
On the right pane:
Uncheck "Use filter" and select the file GM4+.txt; OK;
The imported CIF file has line separators as for UNIX and this is the reason why the file must be converted to the Windows system.
YES to the question "Do you want to continue?".
NO to the question "Do you want to import data from file?";
[On the screen: Basic window]
"File → Structure → Copy in";
On the right pane: Select structure name Ba5Co5; OK
Copy in just m41 and m90 files; OK
Right click on the icon "Refine"; Select the "Basic" and change the "Number of cycles" to 100 and the "Damping factor" to 0.1;
Select the page "Various" and use the button "Restrictions";
Restrict all atoms to have identical ADP parameters and but not occupancies; OK
On the page "Various" select "Fixed commands";
Fix coordinates for all atoms (wild character *);

OK; OK; “Yes+start”;
Refinement converges with Rp~3.0%, R(obs)~6.0%.

The results are identical with those from example 12.1.