Example 12.09: $NiTe_2O_5$

Last update 21.11.2024

Data	Topic	Level
Neutron single crystal	Simple AFM structure (k = 0)	Intermediate

Input data

Data

Single crystal data collected on DEMAND at HFIR, ORNL neutron source at 4 K

Input files

NiTe2O5_nuc4K.int (single crystal data from FullProf)
NiTe2O5_nuclear.cif(nuclear structure)

Additional information

Chemical formula: $NiTe_2O_5$
Antiferromagnetic: k=(0,0,0)

References

J. H. Lee, M. Kratochvílová, H. Cao, Z. Yamani, J. S. Kim, J-G. Park, G. R. Stewart, Y. S. Oh (2019), Phys. Rev. B 100, 144441

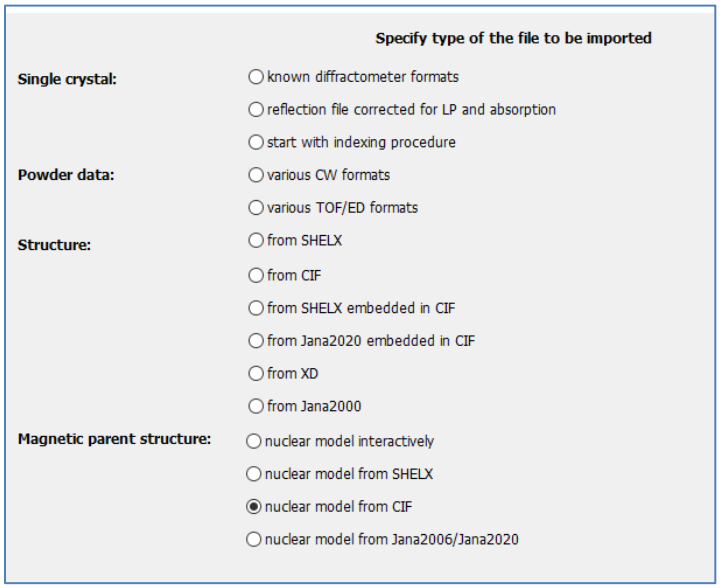
Highlights

In this example, a single crystal data file from FullProf is imported. Corrections for $\lambda/2$ and different extinction models are performed.

Instructions

1. *Data import*

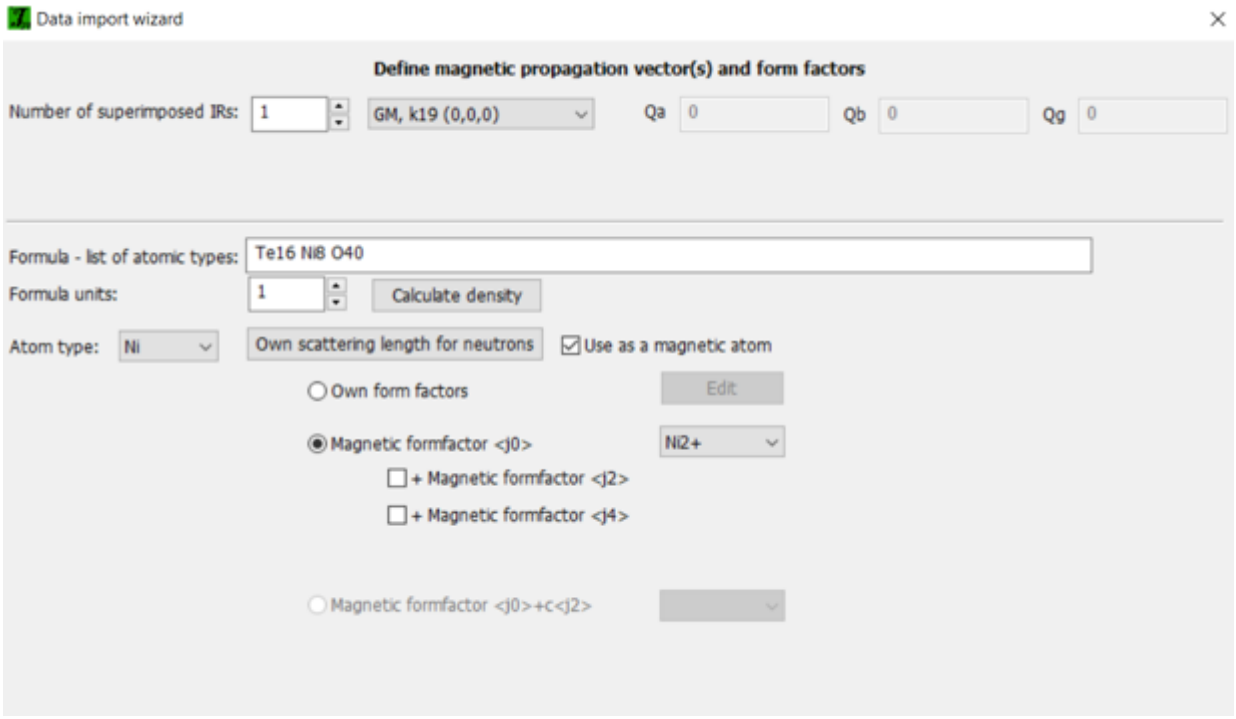
Start Jana2020
In the Main menu bar, use "Structure → New" and open new structure NiTe2O5 in the directory of Example 12.09
[On the screen: Specify type of the file to be imported]
Select "Magnetic parent structure: nuclear model from CIF"; NEXT



In Windows file explorer: select the input file NiTe2O5_nuclear.cif; Open

2. *Define the magnetic propagation vector and form factors*

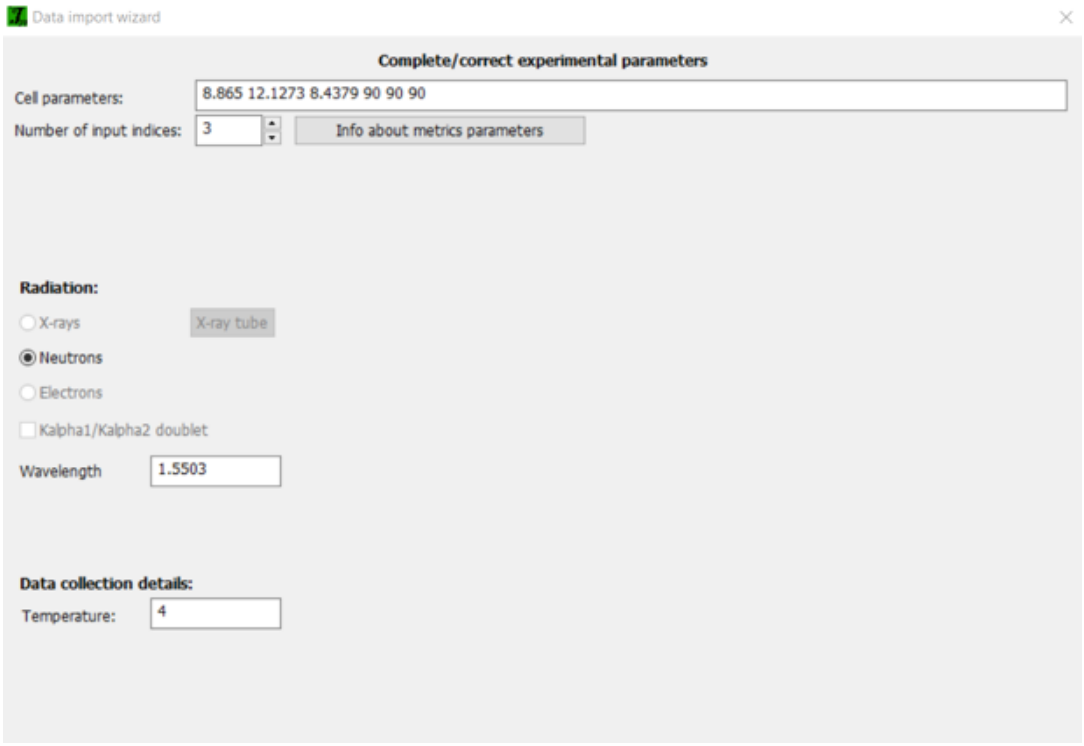
[On the screen: Define magnetic propagation vector(s) and form factors]
Set the "Number of superimposed IRs:" to '1' and select the propagation magnetic vector to be "GM, k19 (0,0,0)" (if not there by default)
Select "Atom type" Ni; check "Use as a magnetic atom" and for "Magnetic formfactor <j0>" select "Ni2+"



NEXT;
[On the screen: Information]
The parent structure is now created.
NEXT;

3. *Import of the reflection file*

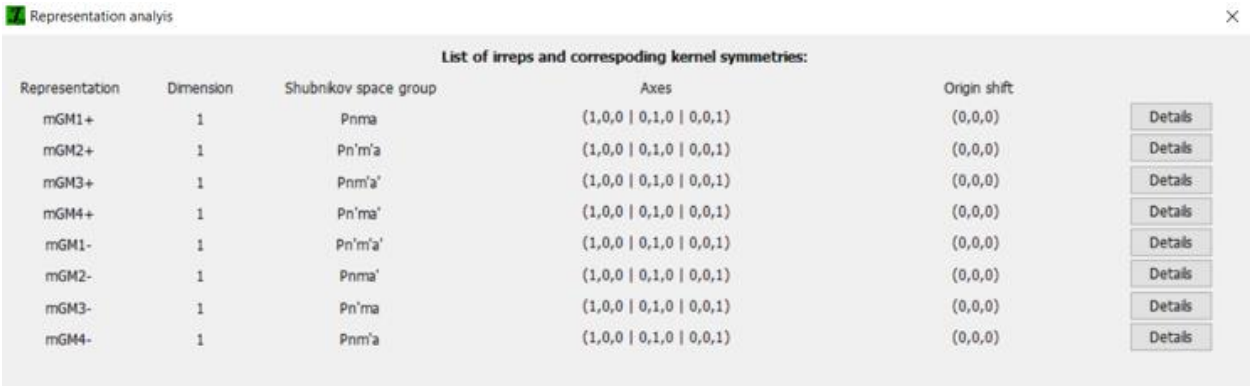
[On the screen: Specify type of the file to be imported]
Select “Single crystal: Reflection file corrected for LP and absorption”; NEXT
Select the format “From FullProf file”; Check that the name of the input file is “NiTe2O5_nuc4K.int”; OPEN; NEXT
[On the screen: Complete/correct experimental parameters]
Check that wavelength is 1.5503; Temperature is 4 K



NEXT; NEXT; OK
[On the screen: Information]
FINISH
[On the screen: Data repository]
OK

4. Testing different irreps to get the best model

[On the screen: Information]
OK
[On the screen: List of irreps and corresponding kernel symmetries]



Notes

This window has an informative 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

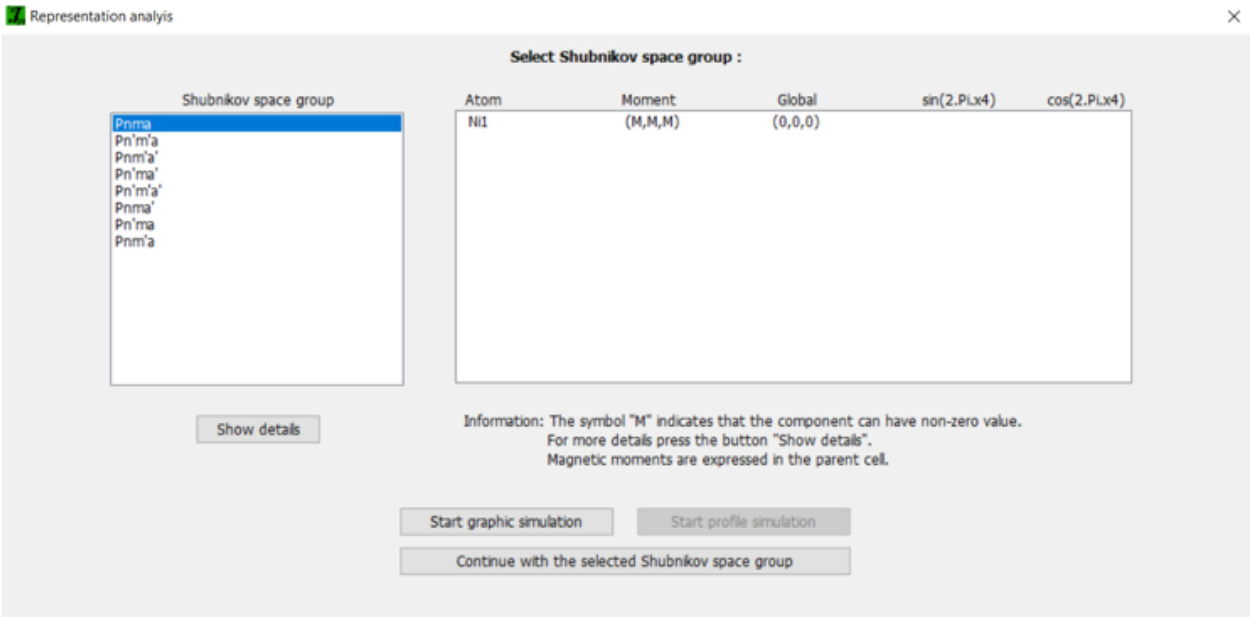
[On screen: List of kernels and epikernels]
The eight orthorhombic Shubnikov space groups are selected by default (if not, just click over one of them to select the block); NEXT

Notes
This will pass all the magnetic space groups to further testing.

[On the screen: Select Shubnikov space group]

Notes
For each space group, the components allowed for the magnetic moment of Ni are indicated in the column 'Moment'. Five of those groups have global moment zero corresponding to antiferromagnetic configurations.

Select the first Shubnikov group *Pnma*



5. Refinement of the magnetic structure in Pnma (mGM1+) and testing of the different extinction models

Press 'Continue with the selected Shubnikov space group'
[On the screen: Select structure name]
Use the default name i.e. NiTe2O5_01; Save

Notes
The test runs under the new job name in a new window to keep the parent structure unchanged. The Jana window with the parent structure remains open. *Jana2020 makes averaging of the data automatically, contrary to FullProf that uses non-averaged data. To make a comparison, we can remake the refinement file.*

[On the screen: Basic window of Jana2020]
In the Command tree, expand "Reflection file" and open "Create refinement reflection file"
[On the screen: Reflection file wizard]
NEXT
[On the screen: Import/Statistics-obs/all]
OK

[On the screen: Reflection file wizard: Process refinement file]
Choose "Use **non-averaged** data", NEXT; FINISH
[On the screen: Basic window of Jana2020]
In the Command tree, expand "Refinement" and open "Refinement commands"
[On the screen: Refinement commands]
On the page "Restrains/Constraints", select "Restrictions" and ensure that all atoms are restricted to have identical ADP parameters and not restricted occupancies ("restric *12"); OK
On the page "Restrains/Constraints", select "Fixed commands" and ensure that all atomic positions are fixed in their current values ("fixed xyz *")
OK

Notes

For this specific case we will see that the positions of atoms are not correct and that should be refined later.

On the page "Basic", check that the "Number of cycles" is set to 100 and the "Damping factor" to 0.1; ensure that "refinements on F(obs)**2" is **not** selected;
OK; YES+START to start the refinement;

Notes

R factors : [582=564+18/5]
GOF(obs)~12.0 GOF(all)~11.8
R(obs)~23.1 wR(obs)~25.2 R(all)~23.2 wR(all)~25.3
R factor from nuclear contribution
R(obs)~21.0 wR(obs)~23.5 R(all)~21.0 wR(all)~23.5
R factor from magnetic contribution
R(obs)~26.0 wR(obs)~28.0 R(all)~26.4 wR(all)~28.1

In the RF factors overview window, we can see that there is a line containing 'R factors from magnetic contribution'. In this case, the propagation vector is zero, but because we have Pnma symmetry, we can create new reflections that were originally systematically absent for the nuclear structure. These reflections are very weak, leading to very high R values. To see better how this refinement works we can use manual culling.

[There is a 'Serious warning' message concerning negative displacement parameters. We shall fix it later if needed.]

Notes

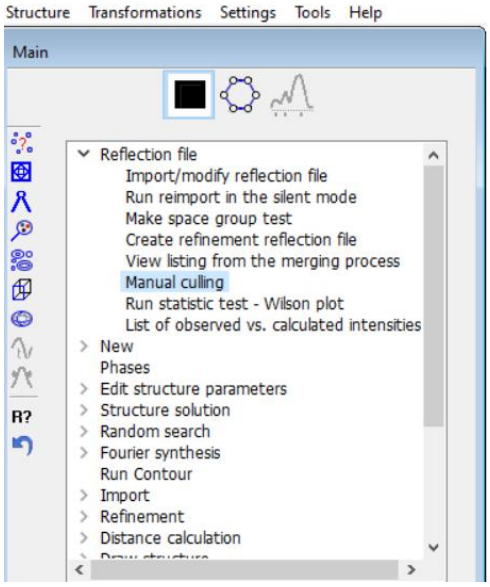
R factors are very large. We'll now try to refine the positions of atoms.

In the Command tree, expand "Refinement" and open "Refinement commands"
[On the screen: Refinement commands]
On the page "Restrains/Constraints", select "Fixed commands". Click over the line "fixed xyz *", and press "Disable"; OK
OK; YES+START

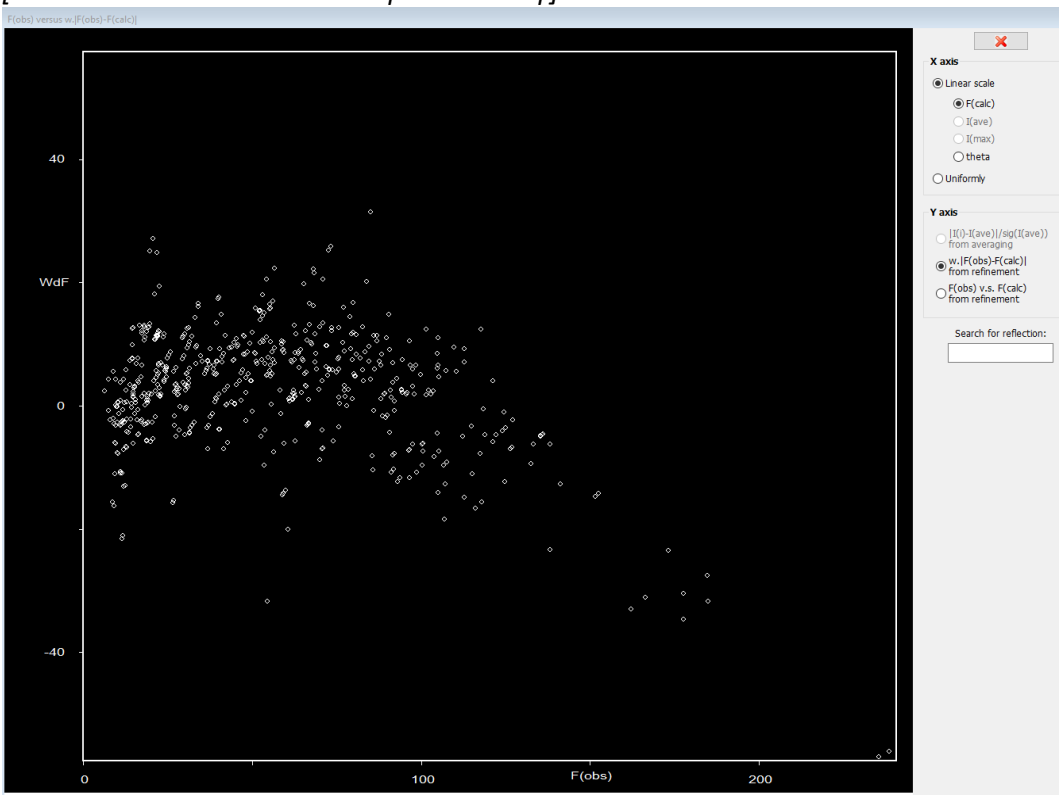
Notes
R factors : [582=564+18/31] *GOF(obs)~10.5 GOF(all)~10.4* *R(obs)~19.0 wR(obs)~21.5 R(all)~19.1 wR(all)~21.5* *R factor from nuclear contribution* *R(obs)~16.8 wR(obs)~19.5 R(all)~16.8 wR(all)~19.5* *R factor from magnetic contribution* *R(obs)~23.8 wR(obs)~25.9 R(all)~24.1 wR(all)~26.0*

[On the screen: Basic window of Jana2020]

In the Command tree, expand “Reflection file” and open “Manual culling”

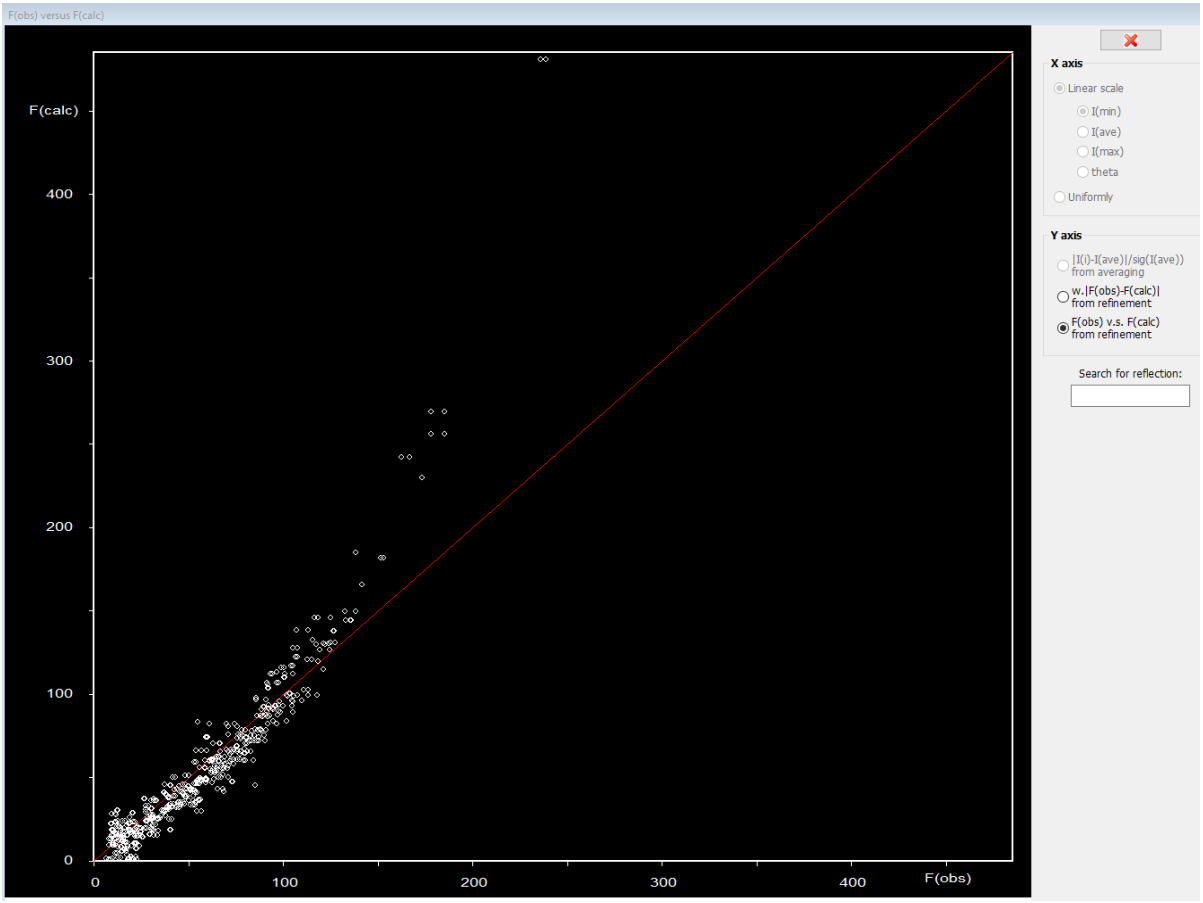


[On the screen: Fobs versus w|Fobs-Fcalc|]



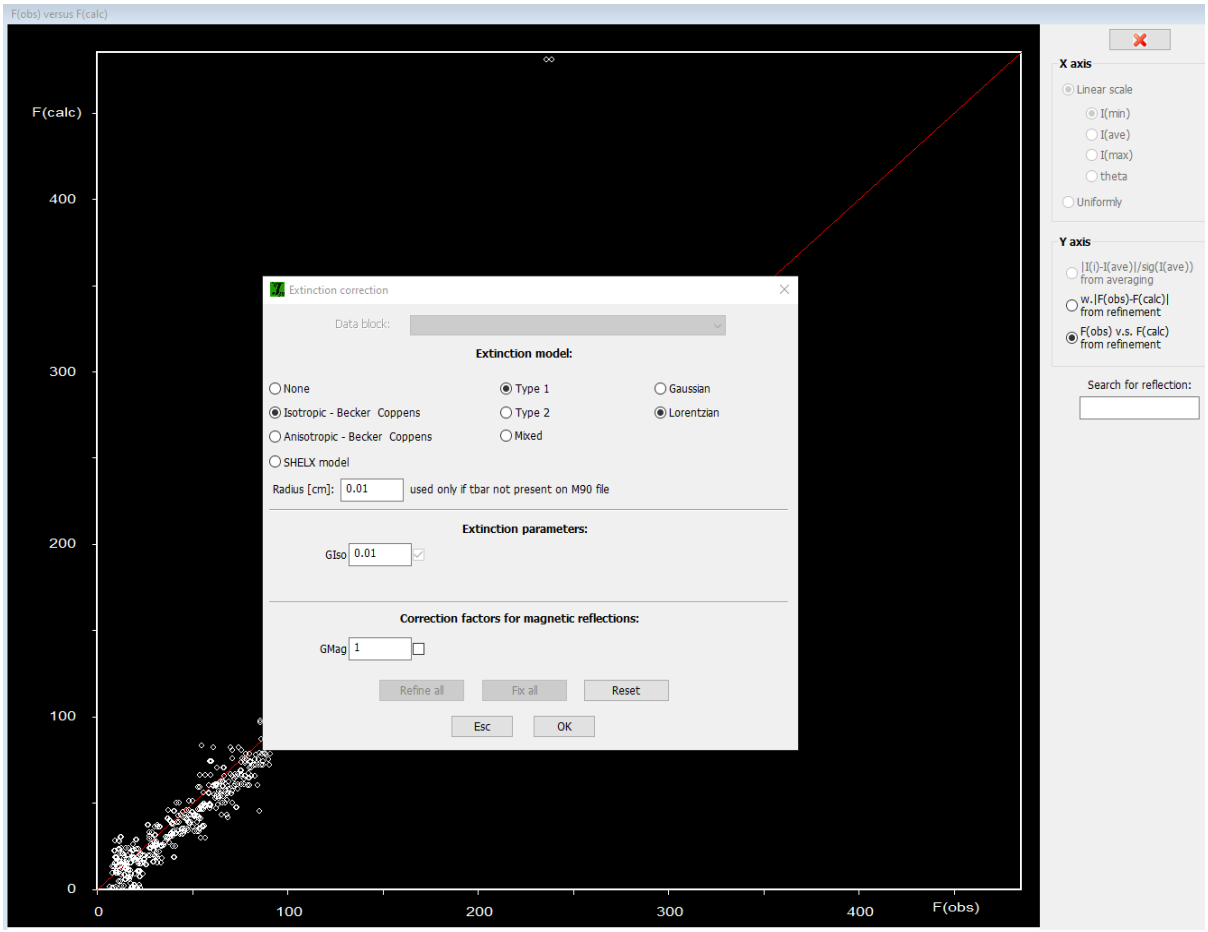
Notes
In this window one can see the Fobs-Fcalc plot and follow its changes during the refinement.

On the right pane choose “Fobs v.s. Fcalc from refinement ”



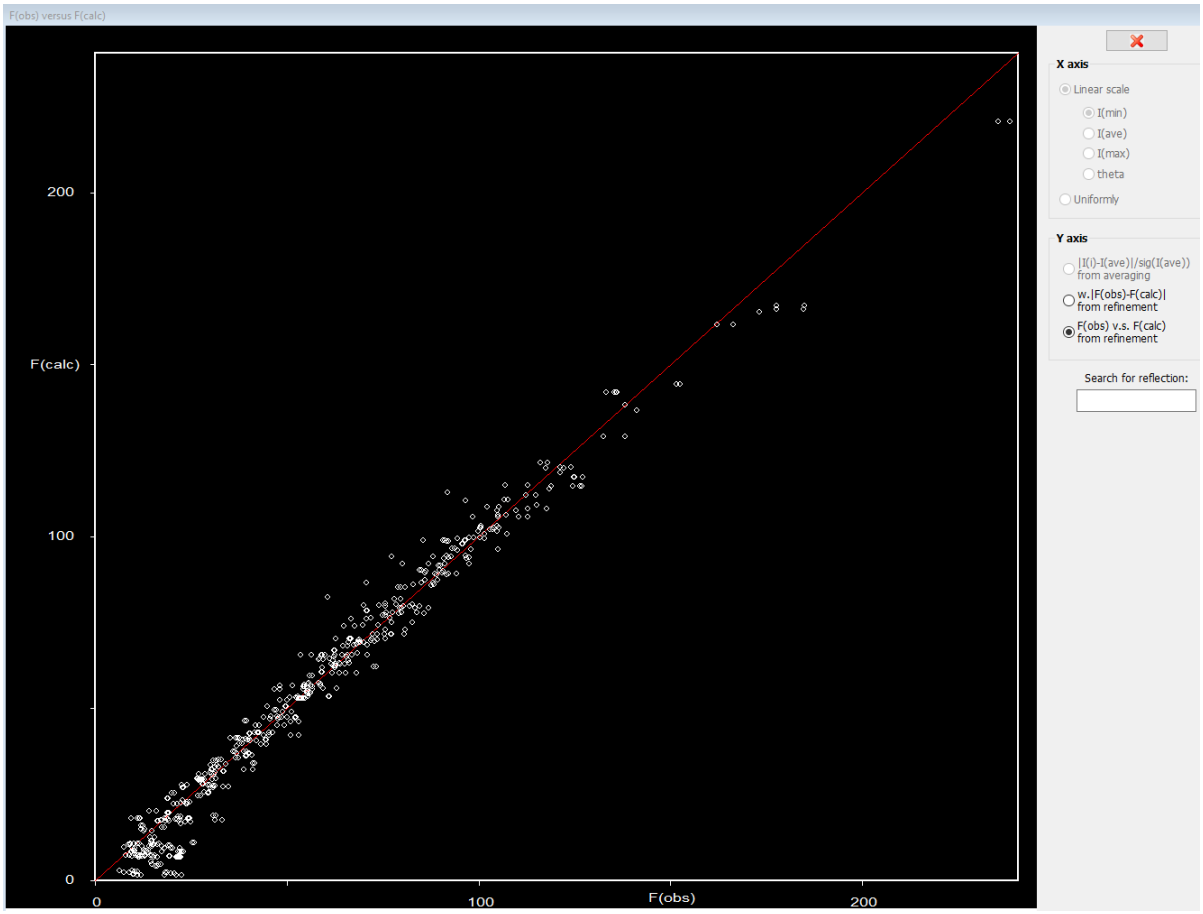
Notes
Here, it is clear that the observed intensity of strong reflections is much weaker than expected. An extinction correction is needed.

In the Command tree, expand “Edit structure parameters” and open “Edit extinction parameters”;
[On the screen: Extinction correction
Select “Isotropic”; “Type 1”; “Lorentzian”;



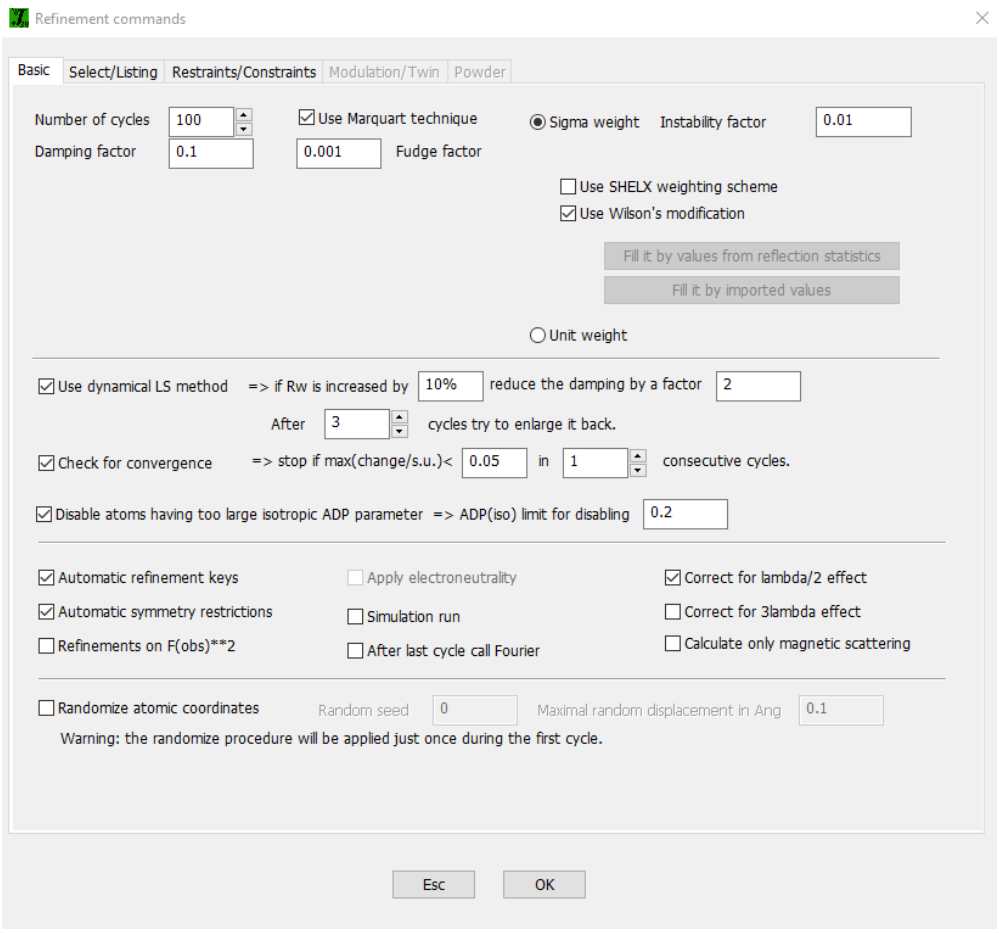
OK; YES; Run refinement or press 

Notes
R factors : [582=564+18/32]
GOF(obs)~5.5 GOF(all)~5.5
R(obs)~7.9 wR(obs)~11.2 R(all)~8.1 wR(all)~11.2
R factor from nuclear contribution
R(obs)~5.3 wR(obs)~6.9 R(all)~5.3 wR(all)~6.9
R factor from magnetic contribution
R(obs)~17.9 wR(obs)~22.3 R(all)~18.4 wR(all)~22.4



Notes
After refinement there is a great improvement in the model, especially for the nuclear contribution. Another important point for this data file is that it was measured on single crystal neutron diffraction for which there is a strong effect of $\lambda/2$ which needs to be accounted for.

In the Command tree, expand “Refinement” and open “Refinement commands”.
[On the screen: Refinement commands]
In the “Basic” tab, tick the option “Correct for lambda/2 effect”;



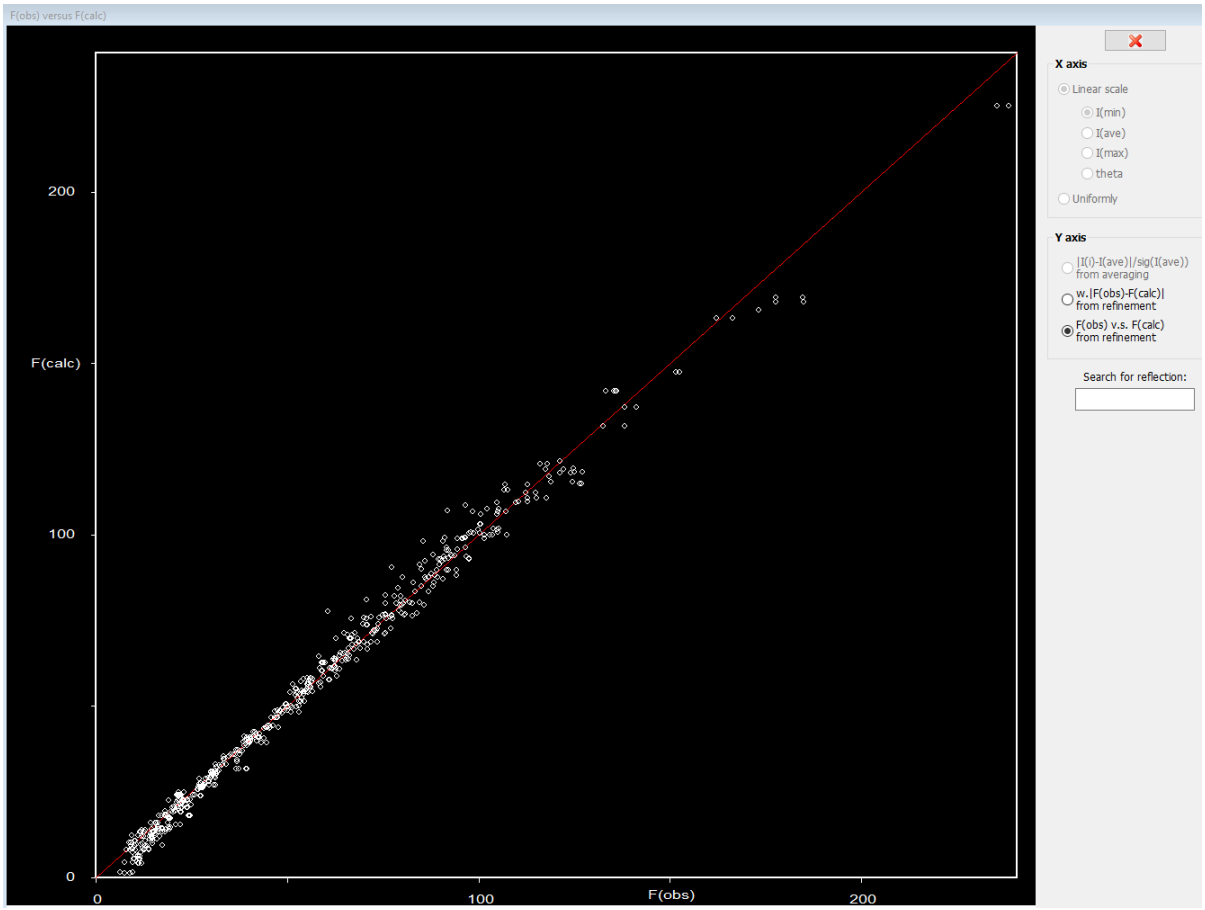
OK; YES

In the Command tree, expand “Edit Structure parameters” and open “Edit scale factors”.

[On the screen: Edit scale parameters]

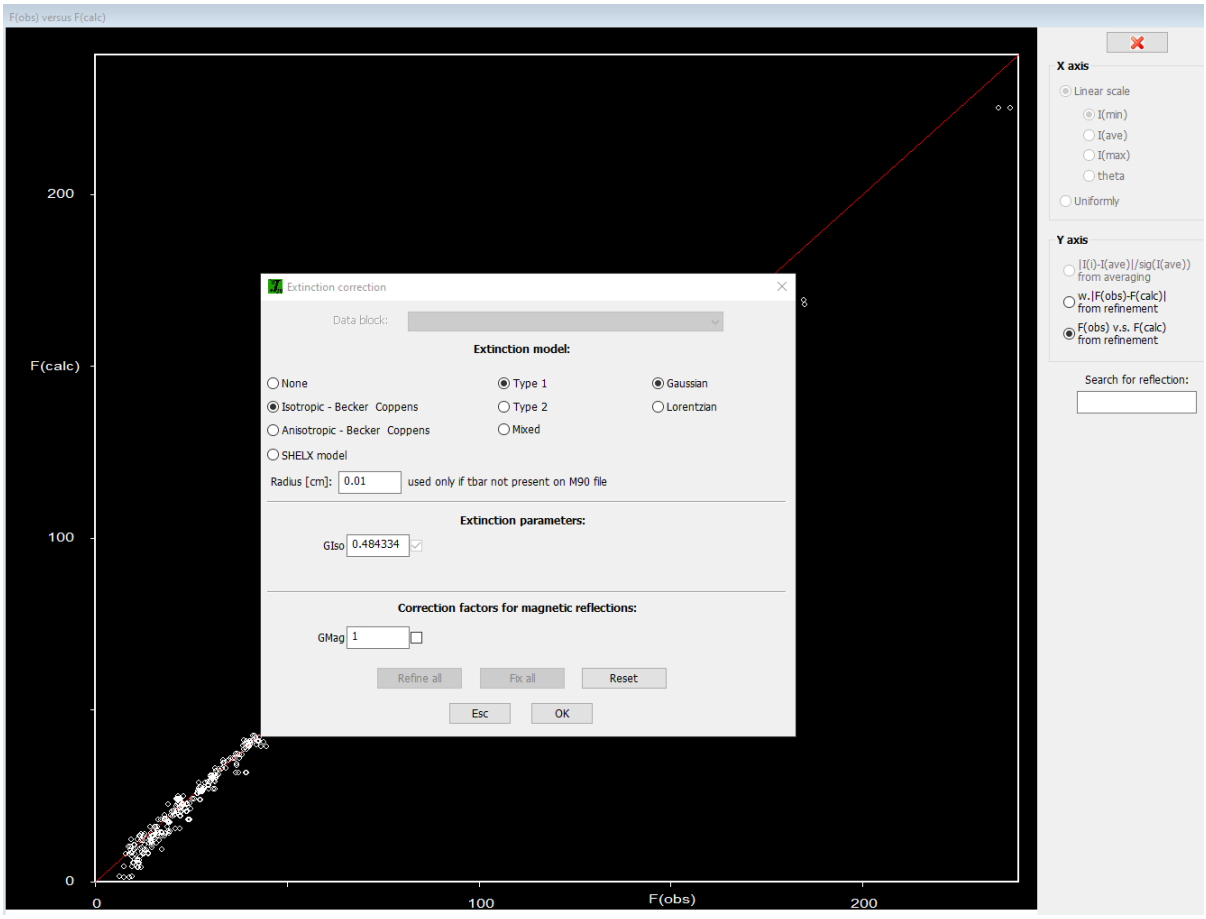
Tick the parameter “sclam/2”. OK; YES; Start refinement

Notes
R factors : [582=564+18/33]
GOF(obs)~2.7 GOF(all)~2.6
R(obs)~4.4 wR(obs)~5.3 R(all)~4.6 wR(all)~5.4
R factor from nuclear contribution
R(obs)~4.1 wR(obs)~5.0 R(all)~4.2 wR(all)~5.1
R factor from magnetic contribution
R(obs)~7.4 wR(obs)~8.2 R(all)~7.7 wR(all)~8.3



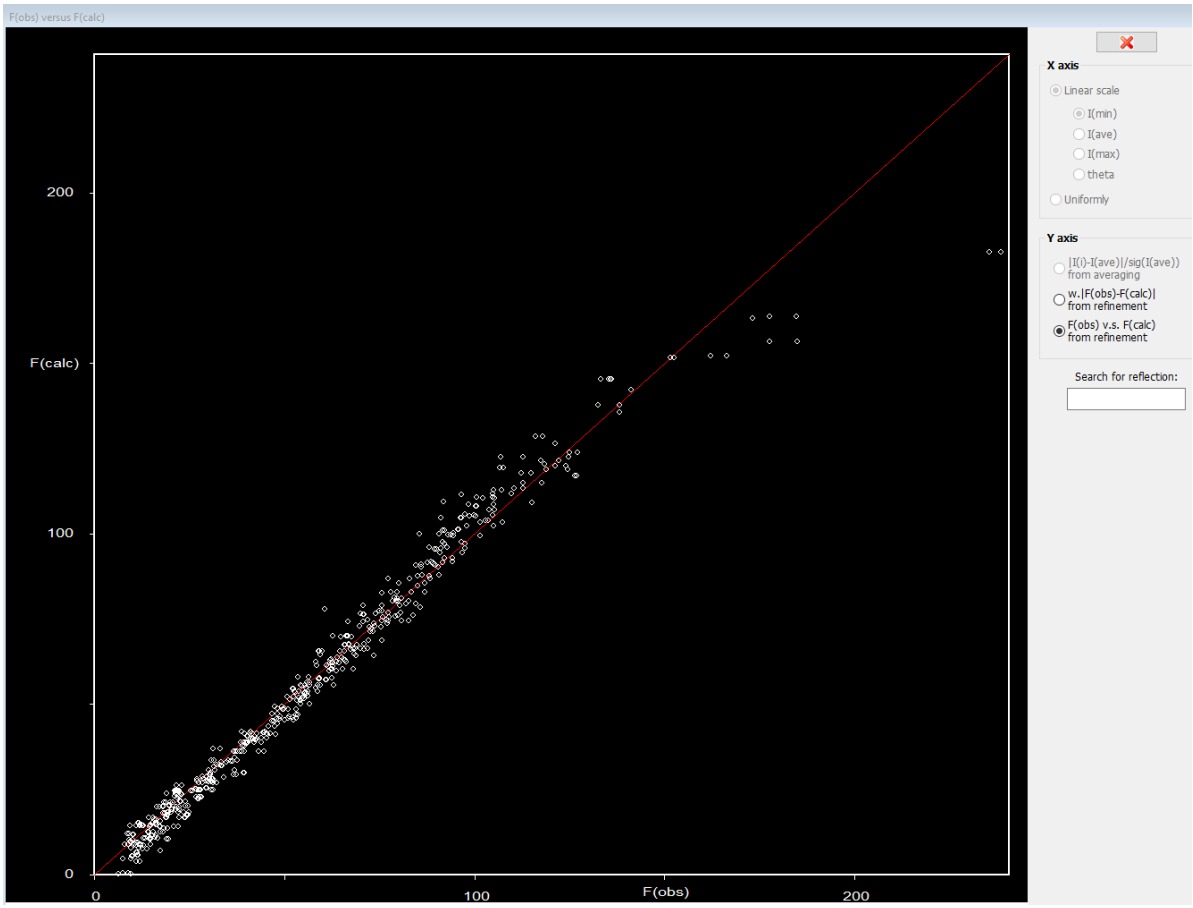
Notes
This result shows the importance of the extinction correction to reach good models. We shall now try another extinction model.

In the Command tree, expand “Edit structure parameters” and open “Edit extinction parameters”;
[On the screen: Extinction correction]
Select “Isotropic”; “Gaussian”; “Type 1”;



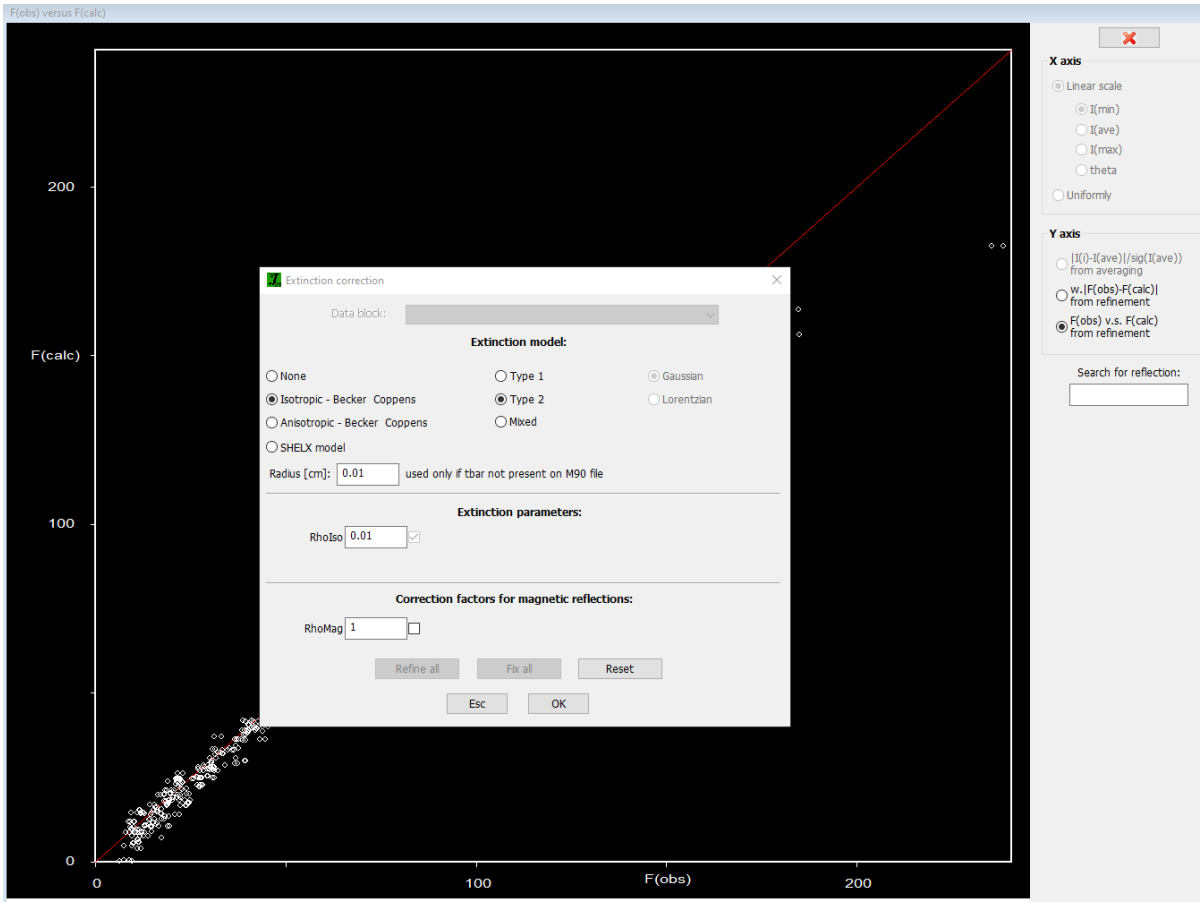
OK; YES; Start refinement

Notes
R factors : [582=564+18/33]
GOF(obs)~3.8 GOF(all)~3.8
R(obs)~6.5 wR(obs)~7.7 R(all)~6.7 wR(all)~7.8
R factor from nuclear contribution
R(obs)~6.1 wR(obs)~7.4 R(all)~6.2 wR(all)~7.4
R factor from magnetic contribution
R(obs)~9.7 wR(obs)~10.2 R(all)~10.2 wR(all)~10.4



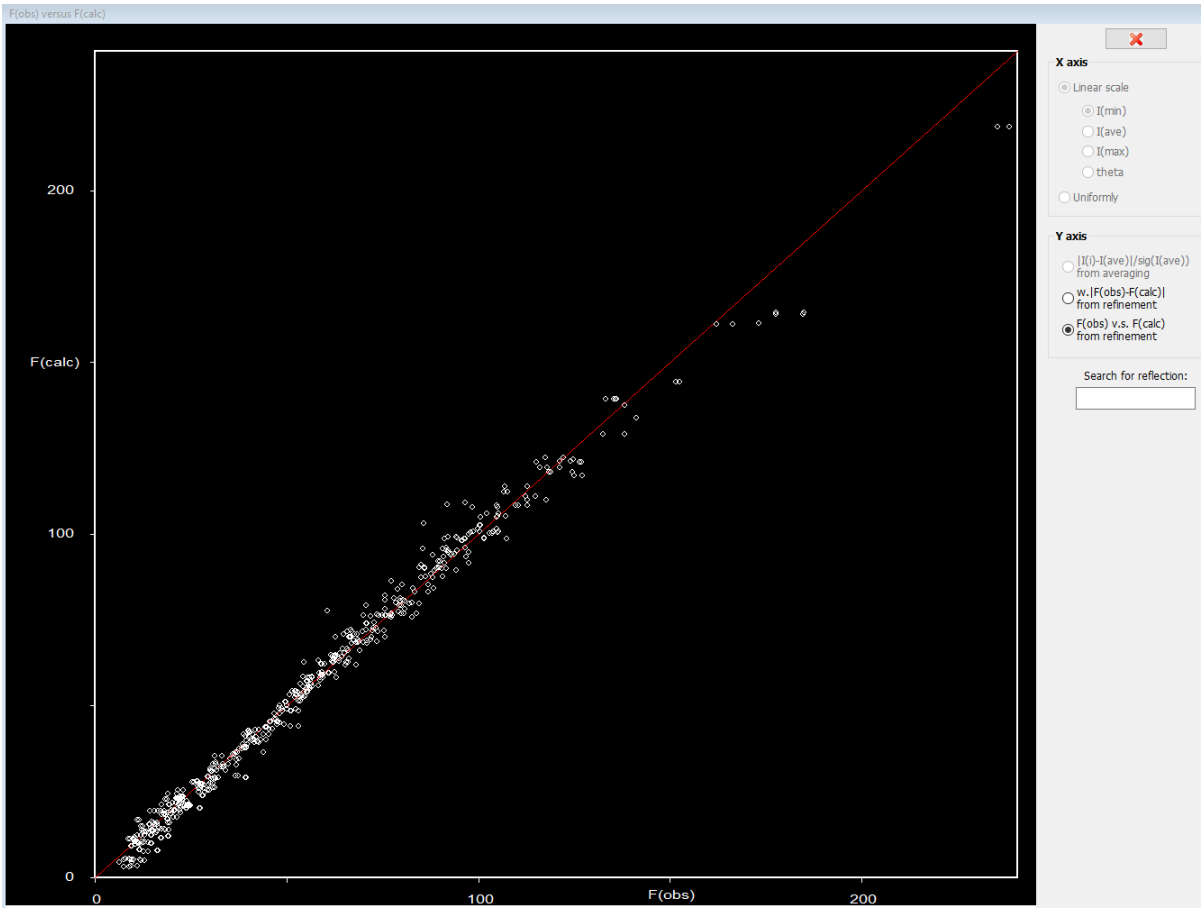
Notes
For the strong reflections, this extinction model gives much worse R factors than the previous one. We can try now also the extinction model type 2, which accounts for the dominating contribution of the crystal size to extinction.

In the Command tree, expand “Edit structure parameters” and open “Edit extinction parameters”;
[On the screen: Extinction correction]
Select “Isotropic”; “Type 2”;



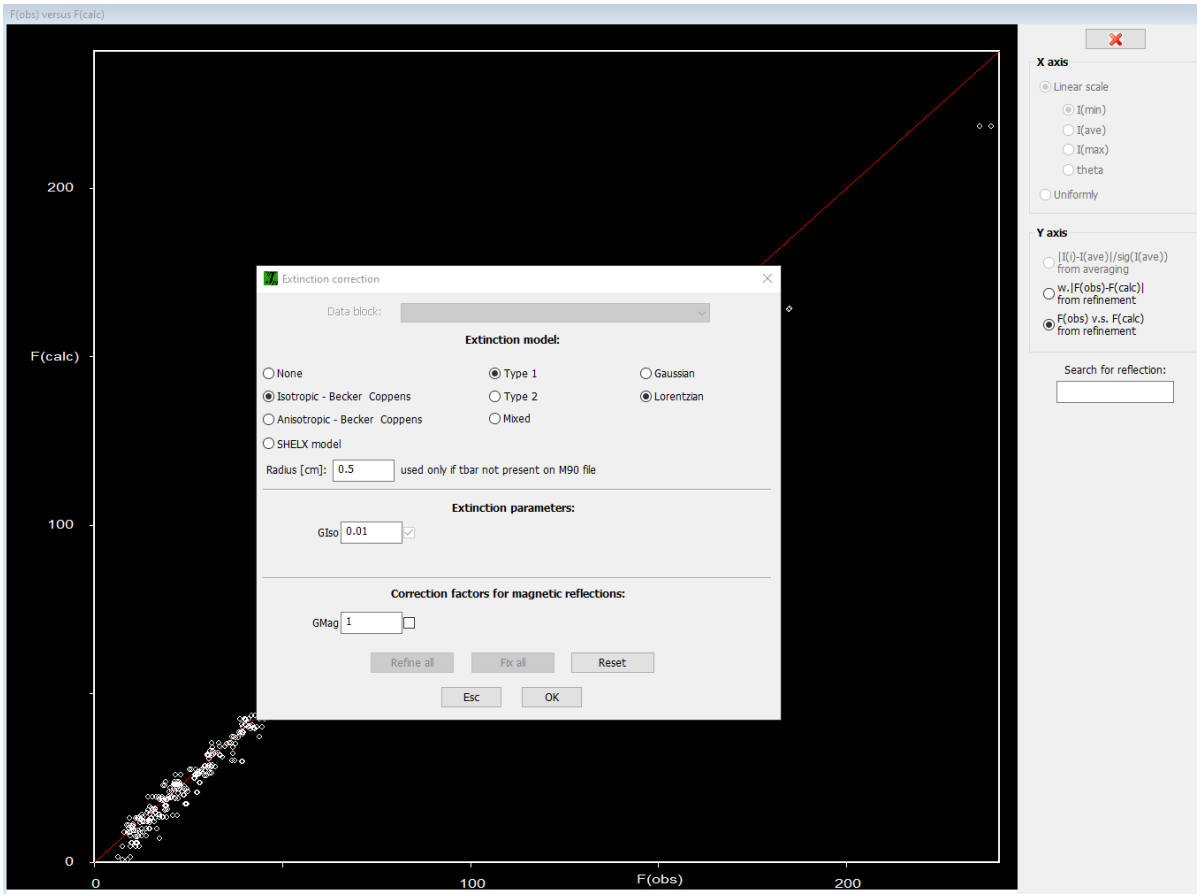
OK; YES; Start refinement

Notes
R factors : [582=564+18/33]
GOF(obs)~3.0 GOF(all)~3.0
R(obs)~4.9 wR(obs)~6.1 R(all)~5.1 wR(all)~6.1
R factor from nuclear contribution
R(obs)~4.5 wR(obs)~5.6 R(all)~4.7 wR(all)~5.6
R factor from magnetic contribution
R(obs)~8.4 wR(obs)~9.6 R(all)~8.6 wR(all)~9.7



Notes
The best model for extinction seems to be the Type 1 Lorentzian. We shall also change the sample radius to a larger value, as the samples for neutron diffraction are generally larger than for x-ray diffraction (the default value of 0.01).

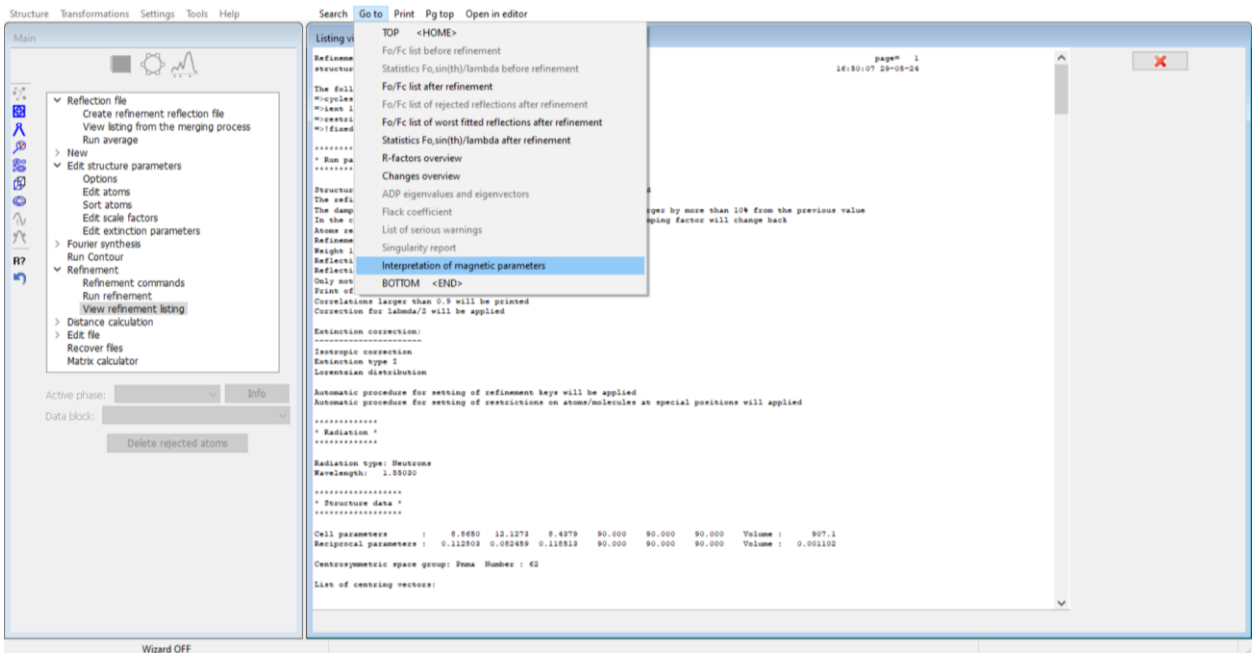
In the Command tree, expand “Edit structure parameters” and open “Edit extinction parameters”;
[On the screen: Extinction correction]
Select “Isotropic”; “Lorentzian”; “Type 1”;
Change the sample radius to 0.5.



OK; YES; Start refinement

Notes
R factors : [582=564+18/33] *GOF(obs)~2.7 GOF(all)~2.6* *R(obs)~4.4 wR(obs)~5.3 R(all)~4.6 wR(all)~5.4* *R factor from nuclear contribution* *R(obs)~4.1 wR(obs)~5.0 R(all)~4.2 wR(all)~5.1* *R factor from magnetic contribution* *R(obs)~7.4 wR(obs)~8.2 R(all)~7.7 wR(all)~8.3* *We can now check the magnetic moment of Ni after the best extinction correction.*

In the Command tree, expand “Refinement” and open “View refinement listing”;
[On the screen: Listing viewer from refinement]
In the menu, select “Go to → Interpretation of magnetic parameters” and check the refined magnetic moments Mx0, My0, and Mz0 for the Ni atom.




```
*****************************************
* Interpretation of magnetic parameters *
*****************************************
```

	Magnetic moments			
Atom	along a	along b	along c	length
Ni1	0.30(5)	1.97(5)	0.31(4)	2.02(8)

Sum of magnetic moments over the whole cell

Atom	along a	along b	along c
Ni1	0.00(15)	0.00(13)	0.00(10)
Summa	0.00(15)	0.00(13)	0.00(10)

```
Program started at 15:33:41 ended   at 15:33:58 cpu time :     14.70
```

Notes
The refined components for the magnetic moment of Ni are similar to those refined in FullProf.

6. *Visualization of the refined model with JanaDraw*

Close the refinement list and the plot panel by 

[On the screen: Basic window]

Change focus to “Janadraw”

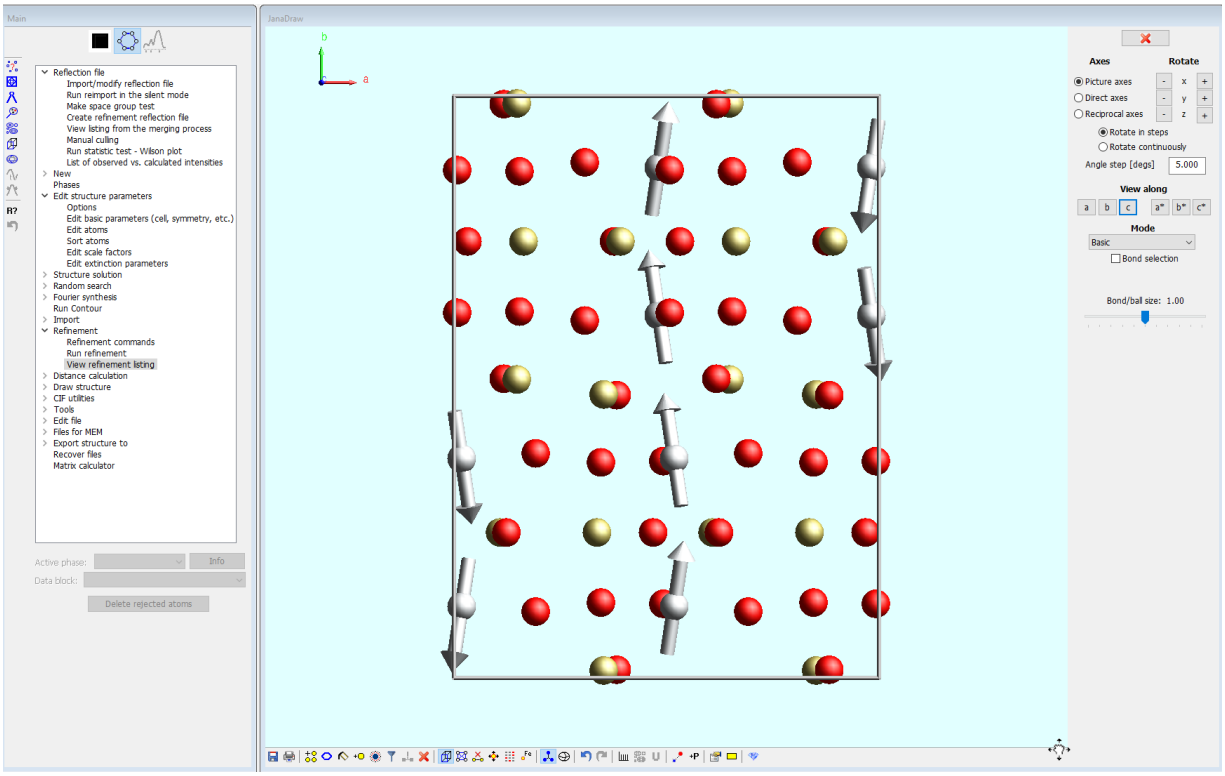


[On the screen: JanaDraw]

Fill the unit cell by the button 

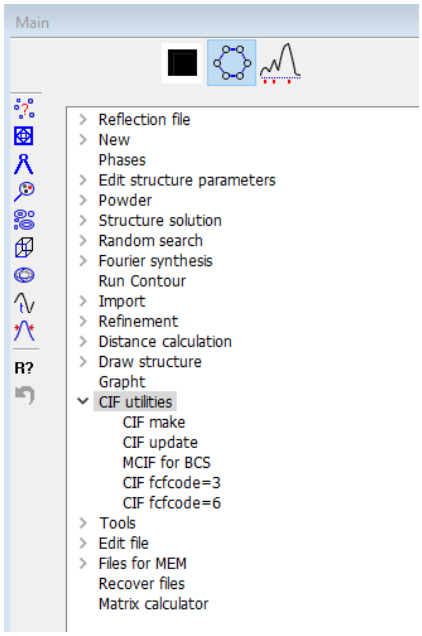
In the right pane, select “View along” c;

Adjust the figure by the button  and remove all bonds by the button 



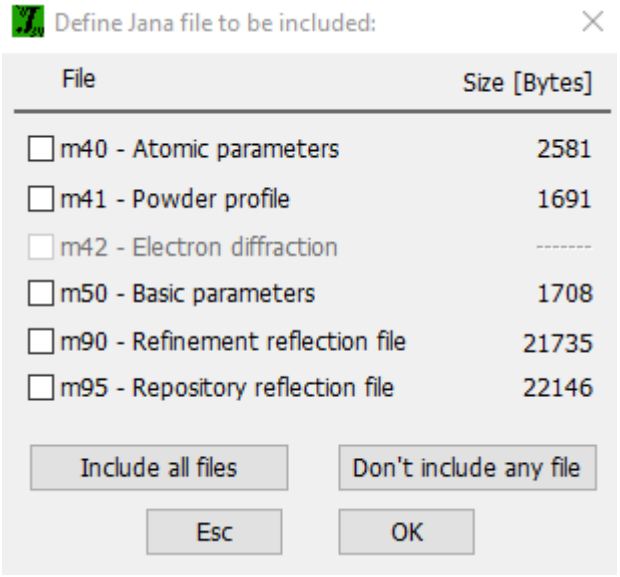
7. *Producing the CIF file*

Close JanaDraw; Or go to the Command tree; expand “CIF utilities” and double-click “CIF make”



Save the CIF file with the default name;

[On screen: Define Jana file to be included]



Select ‘Include all files’; OK
No to the question ‘ Do you want to run the CheckCIF program right now?’

Notes
The file .cif is now stored in the working directory. It can be used for visualization and description of the structure, communication with other programs, and reporting and publication of the structure. *The option ‘MCIF for BCS’ produces a shorter version of the CIF file specifically for communicating with the tools of the Bilbao Crystallographic Server.*