# Example 12.10: NiTe<sub>2</sub>O<sub>5</sub>

Last update 19.6.2024

Data Neutron single crystal Topic Simple antiferromagnetic structure (k = 0) Level 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\_powder X-ray.cif (nuclear structure)

### Additional information

Chemical formula: NiTe<sub>2</sub>O<sub>5</sub> 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  $\rightarrow$  New" and open new structure NiTe2O5 in the directory of Example 12.9

[On the screen: Specify type of the file to be imported] Select "Magnetic parent structure: nuclear model from CIF"; NEXT

	Specify type of the file to be imported
Single crystal:	O known diffractometer formats
	$\bigcirc$ reflection file corrected for LP and absorption
	⊖ start with indexing procedure
Powder data:	🔿 various CW formats
	○ various TOF/ED formats
Structure:	⊖ from SHELX
	◯ from CIF
	○ from SHELX embedded in CIF
	○ from Jana2020 embedded in CIF
	⊖ from XD
	◯ from Jana2000
Magnetic parent structure:	O nuclear model interactively
	O nuclear model from SHELX
	<ul> <li>nuclear model from CIF</li> </ul>
	O nuclear model from Jana2006/Jana2020

In Windows file explorer: select the input file NiTe2O5\_powder X-ray.cif; Open Choose the CIF part to be imported: NiTe2O5 Neutron; OK

	Specify type of the file to be imported	
Single crystal:	O known diffractometer formats	
	O reflection file corrected for LP and absorption	
	O start with indexing procedure	
Powder data:	○ various CW for Select CIF part to be imported ×	
	O various TOF/EI 26533	
Structure:	O from SHELX Normal XRD	
	O from CIF	
	O from SHELX er	
	O from Jana2020	
	O from XD	
	O from Jana2000	
lagnetic parent structure:	O nuclear model i	
	O nuclear model 1	
	nuclear model 1	
	O nuclear model	

2. Define the magnetic propagation vector and form factors

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

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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+"

📕 Data import wizard					×		
Define magnetic propagation vector(s) and form factors							
Number of superimposed IRs:	1 GM, k19 (0,0,0) ~	Qa 0	Qb 0	Qg 0			
Formula - list of atomic types:	Te16 NI8 O40						
Formula units:	1 Calculate density						
Atom type: Ni V	Own scattering length for neutrons	a magnetic atom					
	○ Own form factors	Edit					
	Magnetic formfactor <j0></j0>	Ni2+ ~					
	+ Magnetic formfactor <j2></j2>						
	+ Magnetic formfactor <j4></j4>						
	○ Magnetic formfactor <j0>+c<j2></j2></j0>	~					

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"; NEXT [On the screen: Complete/correct experimental parameters] Check that wavelength is 1.5503; Temperature is 4 K

Data import wizard		×					
	Complete/correct experimental parameters						
Cell parameters:	Il parameters: 8.865 12.1273 8.4379 90 90 90						
Number of input indices:	3 Info about metrics parameters						
Radiation:							
○ X-rays	X-ray tube						
Neutrons							
<ul> <li>Electrons</li> </ul>							
Kalpha1/Kalpha2 doub	et						
Wavelength 1.55	03						
Data collection details							
Temperature: 4							

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]

ОК

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

		List of im	eps and correspoding kernel symmetries:		
lepresentation	Dimension	Shubnikov space group	Axes	Origin shift	
mGM1+	1	Pnma	(1,0,0   0,1,0   0,0,1)	(0,0,0)	Details
mGM2+	1	Pn'm'a	(1,0,0   0,1,0   0,0,1)	(0,0,0)	Details
mGM3+	1	Pnm'a'	(1,0,0   0,1,0   0,0,1)	(0,0,0)	Details
mGM4+	1	Pn'ma'	(1,0,0   0,1,0   0,0,1)	(0,0,0)	Details
mGM1-	1	Pn'm'a'	(1,0,0   0,1,0   0,0,1)	(0,0,0)	Details
mGM2-	1	Pnma'	(1,0,0   0,1,0   0,0,1)	(0,0,0)	Details
mGM3-	1	Pn'ma	(1,0,0   0,1,0   0,0,1)	(0,0,0)	Detais
mGM4-	1	Pnm'a	(1,0,0   0,1,0   0,0,1)	(0,0,0)	Details

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

7 Representation analyis							
Select Shubnikov space group :							
,	Shubnikov space group	Atom	Moment	Global	sin(2.Pi.x4)	cos(2.Pi.x4)	
Prima Prima Prima Prima Prima Prima Prima		NI	(M,M,M)	(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 pro	file simulation			
		Continue with the	selected Shubnikov sp	ace group			

## 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 nonaveraged 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

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[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

OK; YES+START to start the refinement;

Notes

R factors : [582=564+18/5] GOF(obs)~16.0 GOF(all)~15.8 R(obs)~36.6 wR2(obs)~62.9 R(all)~36.8 wR2(all)~63.2 R factors for nuclear reflections : [472=466+6] R(obs)~35.5 wR2(obs)~62.4 R(all)~35.7 wR2(all)~62.7 R factors for magnetic reflections : [110=98+12] R(obs)~52.8 wR2(obs)~77.8 R(all)~52.8 wR2(all)~78.3

In the RF factors overview window, we can see that there is a line containing 'R factors for magnetic reflections'. 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.]

[On the screen: Basic window of Jana2020] In the Command tree, expand "Reflection file" and open "Manual culling" Protective Tendematics Setting Teols Help New Control of Protection file Reflection file Ref

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

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*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"; "Lorentzian"; "Type 1";

F(obs) versus F(ca	lc)				
					×
400			00		X axis
					Linear scale
					● I(min)
					(ave)
					(max)
r (caic)					
					C dimonity
					Yaxis
					I(i)-I(ave) /sig(I(ave))
300	The Extinction correction		×		w.IF(obs)-F(calc)
	Data block:		$\sim$		from refinement
		Extinction model:			F(ODS) V.S. F(Calc) from refinement
		Extinction model.			
,	○ None	Type 1	<ul> <li>Gaussian</li> </ul>		Search for reflection:
	Isotropic - Becker Coppens	O Type 2	<ul> <li>Lorentzian</li> </ul>		
	Anisotropic - Becker Coppen	s O Mixed			
200	O SHELX model				
200	Radius [cm]: 0.01 use	d only if tbar not present on M90 file			
		Extinction parameters:			
	GIso 0.01				
	Corre	ction factors for magnetic reflec	tions:		
	cu[1	,			
	GMay				
100	<ul> <li>Refine</li> </ul>	e all Fix all	Reset		
		Fre OV			
	° ° ° ′	ESC OK			
0				F(obs)	100
		200	300		400
Rint(obs)/Rint(al R(obs)/R(all): 3	i):/ N(obs)/N(all): 564/582 Redundanc i.19/36.36 Rw(obs)/Rw(all): 62.67/62.99 GOF: 15.63	y:1 Culled(fit)/ 57	cuiled(man)/Culled(auto): 0/0/0		

OK; YES; Run refinement or press 🖾

#### Notes

R factors : [582=564+18/6] GOF(obs)~13.7 GOF(all)~13.8 R(obs)~28.0 wR2(obs)~53.8 R(all)~28.4 wR2(all)~54.7 R factors for nuclear reflections : [472=466+6] R(obs)~26.8 wR2(obs)~53.6 R(all)~27.1 wR2(all)~54.5 R factors for magnetic reflections : [110=98+12] R(obs)~45.4 wR2(obs)~60.7 R(all)~45.8 wR2(all)~61.0



From the plot and R factors, it is obvious that the refinement looks better. Nevertheless, the R factors are yet 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+18/32] GOF(obs)~4.0 GOF(all)~4.0 R(obs)~7.6 wR2(obs)~16.2 R(all)~7.9 wR2(all)~16.3 R factors for nuclear reflections : [472=466+6] R(obs)~5.2 wR2(obs)~12.5 R(all)~5.2 wR2(all)~12.5 R factors for magnetic reflections : [110=98+12] R(obs)~43.3 wR2(obs)~56.3 R(all)~43.7 wR2(all)~56.5



After refinement there is a great improvement in the model, especially for the nuclear reflections. 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";

Refinement commands	×
Basic Select/Listing Restraints/Constraints Modulation/Twin Powder	
Number of cycles 100 💌 🗹 Use Marquart technique Damping factor 0.1 0.001 Fudge factor	Sigma weight Instability factor     0.01
	Use SHELX weighting scheme
	Use Wilson's modification
	Fill it by values from relection statistics
	Fill it by imported values
	○ Unit weight
Use dynamical LS method => if Rw is increased by 10% red After 3 cycles try to	uce the damping by a factor 2 enlarge it back.
Check for convergence => stop if max(change/s.u.)< 0.05	in 1 consecutive cycles.
☑ Disable atoms having too large isotropic ADP parameter => ADP(iso	) limit for disabling 0.2
Automatic refinement keys	y Correct for lambda/2 effect
Automatic symmetry restrictions	Correct for 3lambda effect
☑ Refinements on F(obs)**2	urier Calculate only magnetic scattering
Randomize atomic coordinates Random seed 0 Warning: the randomize procedure will be applied just once during t	Maximal random displacement in Ang 0.1 the first cycle.
Esc	ОК

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)~2.7 R(obs)~4.7 wR2(obs)~10.8 R(all)~4.9 wR2(all)~10.8 R factors for nuclear reflections : [472=466+6] R(obs)~4.0 wR2(obs)~9.8 R(all)~4.1 wR2(all)~9.8 R factors for magnetic reflections : [110=98+12] R(obs)~14.3 wR2(obs)~24.9 R(all)~15.5 wR2(all)~25.4



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";

F(obs) versus F(calc)			G		
					×
200					x axis ○ Linear scale ○ I(min) □ I(ave) □ I(max) ○ theta
200					O Uniformly
					Yaxis
	J. Extinction corr	ection		×	II(I)-I(ave) /sig(I(ave)) from averaging w.IF(obs)-F(calc)
	Data	block:		~	F(obs) v.s. E(calc)
		Extincti	on model:		• from refinement
F(calc) -	○ None	•	Type 1	ian	Search for reflection:
	Isotropic - Beck	er Coppens	Type 2 O Loren	tzian	
	🔿 Anisotropic - Be	cker Coppens	fixed		
	⊖ SHELX model				
	Radius [cm]: 0.0	used only if thar not p	resent on M90 file		
100 -	GIso	Extinction	parameters:		
		Correction factors for	magnetic reflections:		
	GMag 1				
			Death		
		Ketine all	x dii Keset		
-		Esc	ОК		
	A COLORIDA				
	<i>ڰ</i>				
	<u></u>				
	1000 °				
0 -4	· 60				
Rint(obs)/Rint(all)	/ N(obs)/N(all): 564/582	100 Redundancy : 1	Culled(fit)/Culled(man)/Culled	(auto): 0/0/0	<u>+</u> ق+
R(obs)/R(all): 7.53	7.78 Rw(obs)/Rw(all): 16.15/16.21	GOF : 3.919	same and a second se	·······	Ŷ

## OK; YES; Start refinement

Notes *R* factors : [582=564+18/33] *GOF*(obs)~3.8 *GOF*(all)~3.8 *R*(obs)~6.9 wR2(obs)~15.4 *R*(all)~7.0 wR2(all)~15.4 *R* factors for nuclear reflections : [472=466+6] *R*(obs)~6.2 wR2(obs)~14.6 *R*(all)~6.2 wR2(all)~14.6 *R* factors for magnetic reflections : [110=98+12] *R*(obs)~16.6 wR2(obs)~29.8 *R*(all)~17.7 wR2(all)~30.2



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";

Structure Transformations Settings Tools Help	File Tools	
Main	F(obs) versus F(calc)	
Reflection file		X axis
Create refinement reflection file View listing from the merging process		(i) Linear scale
> New	200 - Kinction correction X	O I(ave)
Control Contro	Data block:	O I(max)
Edit atoms     Sort atoms     Edit scale factors	Extinction model:	Uniformly
The Edit extinction parameters	O None O Type 1      Gaussian	Yaxis
R? Run Contour > Refinement	(@ Isotropic - Becker Coppens     (@ Isotropic - Becker Coppens     () Anisotropic - Becker Coppens     () Mixed	<pre>[I(i)-I(ave)]/sig(I(ave)) from averaging</pre>
> Distance calculation > Edit file Recover files	O SHELX model	O w. (F(obs)-F(calc)) from refinement
Matrix calculator	Radius [cm]: 0.01 used only if thar not present on M90 file	F(obs) v.s. F(calc) from refinement
Active phase:	Extinction parameters:	Search for reflection:
Delete rejected atoms	Correction factors for magnetic reflections:	
	RhoMag 1	
	Refine all Fix all Reset	
	Rimi(obs)/Rimi(all):/         N(obs)/R(all): 564/582         Redundancy: 1         Cuiled(fit)/Cuiled(man)/Cuiled(auto): 0/0/0           R(obs)/R(all): 6.63/6.70         Rw(obs)/R(all): 15.08/15.12         GOF         Cuiled(fit)/Cuiled(man)/Cuiled(auto): 0/0/0	

OK; YES; Start refinement

#### Notes

R factors : [582=564+18/33] GOF(obs)~3.0 GOF(all)~3.0 R(obs)~5.2 wR2(obs)~12.1 R(all)~5.3 wR2(all)~12.1 R factors for nuclear reflections : [472=466+6] R(obs)~4.3 wR2(obs)~10.6 R(all)~4.4 wR2(all)~10.6 R factors for magnetic reflections : [110=98+12] R(obs)~17.1 wR2(obs)~32.1 R(all)~18.0 wR2(all)~32.4



The best model for the 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.

Main -	F(obs) versus F(calc)	
Wan         Image: Second Sec	200       Image: Extinction correction       X         Data block:       Image: Extinction model:       Image: Extinction model:         Image: Extinction extinction       Image: Extinction model:       Image: Extinction model:         Image: Extinction extinction       Image: Extinction model:       Image: Extinction model:         Image: Extinction extinction       Image: Extinction model:       Image: Extinction model:         Image: Extinction extinction       Image: Extinction extinction extinction extinction extinction extinction parameters:       Image: Extinction	X axis Unear scale ((rm)) ((ave)) ((max)) (
Active phase: v Infe Data block: v Delete rejected atoms	Glio       0.457341         Glio       0.457341         Correction factors for magnetic reflections:         GHap       1         Refne all       Fix all         Reset       0         Bio       100         Fix all       Reset         Correction factors for magnetic reflections:       0         Glio       100         Fix all       Reset         Esc       0K         100       F(Obs)         200       Redmdancy: 1         Called(R)/Called(ma)/Called(auto): 0/0/0         GoF: 2.881       0	

### OK; YES; Start refinement

Notes

R factors : [582=564+18/33] GOF(obs)~2.7 GOF(all)~2.7 R(obs)~4.7 wR2(obs)~10.8 R(all)~4.9 wR2(all)~10.8 R factors for nuclear reflections : [472=466+6] R(obs)~4.0 wR2(obs)~9.8 R(all)~4.1 wR2(all)~9.8 R factors for magnetic reflections : [110=98+12] R(obs)~14.3 wR2(obs)~25.0 R(all)~15.5 wR2(all)~25.4

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  $\rightarrow$  Interpretation of magnetic parameters" and check the refined magnetic moments Mx0, My0, and Mz0 for the Ni atom.

Structure Transformations Settings Tools Help	Search Go to Print Pg top Open in editor		
Main	Listing vi TOP <home></home>		
- P9 A	Fo/Fc list before refinement		
	Statistics Fo,sin(th)/lambda before refinement	16:50:07 29-05-24	~ ×
14	The foll Fo/Fc list after refinement		
✓ Reflection file	Fo/Fc list of rejected reflections after refinement		
Create refinement reflection file	=>restri Fo/Fc list of worst fitted reflections after refinement		-
Run average	=>!fixed Statistics Fo.sin(th)/lambda after refinement		
<sup>30</sup> > New	Refactors overview		
Continue parameters	Changes overview		
Edit atoms	Structur ADD eigenvalues and eigenvectors		
Sort atoms	The refi Flock coefficient	over hy more than 10% from the previous value	
Edit scale factors	In the c	ping factor will change back	
Edit extinction parameters     Equiper synthesis	Atoms re List of serious warnings Refineme		
R2 Run Contour	Weighe 1 Singularity report		
♥ Refinement	Reflecti Interpretation of magnetic parameters		
- ) Refinement commands	Coly not BOTTOM <end></end>		
View refinement listing	Correlations larger than 0.9 will be printed Correction for labmda/2 will be applied		
> Distance calculation			
> Edit file	Extinction correction:		
Matrix calculator	Isotropic correction Extinction type I		
	Lorentsian distribution		
Active phase: V Info	Automatic procedure for setting of refinement keys will be applied		
Data Mork:	Automatic procedure for setting of restrictions on atoms/molecules at	t special positions will applied	
	* Radiation *		
Delete rejected atoms			
	Radiation type: Neutrons		
	Wavelength: 1.55030		
	A Remember data a		
	Cell parameters : 0.8650 12.1273 0.4279 90.000 9	90.000 90.000 Volume : 907.1	
	Reciprocal parameters : 0.112803 0.082459 0.118513 90.000 9	90.000 90.000 Volume : 0.001102	
	Centrosymmetric space group: Pnma Number : 62		
	List of centring vectors:		
			~
Ward OFF			

Atom	Magnetic moments			
	along a	along b	along c	length
Nil	0.30(5)	1.97(5)	0.31(4)	2.02(8)
	Sum of magnetic r	moments over t	he whole cell	
Atom	along a	along b	along c	
Nil Summa	0.00(15) 0.00(15)	0.00(13) 0.00(13)	0.00(10) 0.00(10)	
Program started at 15:33:41	ended at 15:33:50	8 cpu time :	14.70	

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

## 6. Visualization of the refined model with JanaDraw



[On the screen: JanaDraw]

Fill the unit cell by the button  $^{igstyle h}$ 



In the right pane, select "View along" c; Adjust the figure by the button 💠 and remove all bonds by the button 🔀

