

Example of Jana refinement of magnetic structure on single-crystal data

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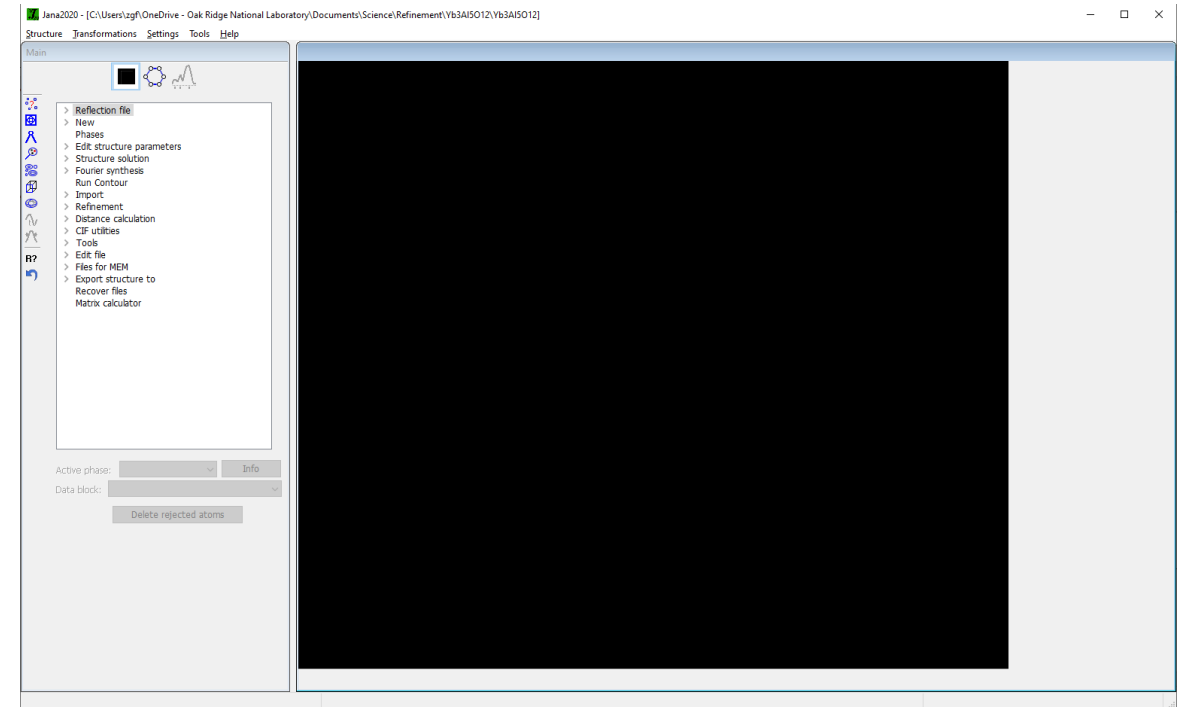
Oak Ridge National Laboratory

**Magnetic Structure Determination from Neutron
Diffraction Data, October 3-7, 2022**

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Yb₃Al₅O₁₂: Jana 2020 Single Crystal Refinement Tutorial

- Open Jana2020
- Structure > New
- Navigate to Yb₃Al₅O₁₂
- Choose Yb₃Al₅O₁₂_300K.hkl



Yb₃Al₅O₁₂: Jana 2020 Single Crystal Refinement Tutorial

- Choose Structure from CIF

Specify type of the file to be imported

Single crystal:	<input type="radio"/> known diffractometer formats
	<input type="radio"/> reflection file corrected for LP and absorption
	<input type="radio"/> start with indexing procedure
Powder data:	<input type="radio"/> various CW formats
	<input type="radio"/> various TOF/ED formats
Structure:	<input type="radio"/> from SHELX
	<input checked="" type="radio"/> from CIF
	<input type="radio"/> from SHELX embedded in CIF
	<input type="radio"/> from Jana2020 embedded in CIF
	<input type="radio"/> from XD
	<input type="radio"/> from Jana2000
Magnetic parent structure:	<input type="radio"/> nuclear model interactively
	<input type="radio"/> nuclear model from SHELX
	<input type="radio"/> nuclear model from CIF
	<input type="radio"/> nuclear model from Jana2006/Jana2020

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$\text{Yb}_3\text{Al}_5\text{O}_{12}$: Jana 2020 Single Crystal Refinement Tutorial

- Choose Single Crystal reflection file corrected for LP and absorption

Specify type of the file to be imported

Single crystal:

- ☐ known diffractometer formats
- ☒ reflection file corrected for LP and absorption
- ☐ start with indexing procedure

Powder data:

- ☐ various CW formats
- ☐ various TOF/ED formats

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$\text{Yb}_3\text{Al}_5\text{O}_{12}$: Jana 2020 Single Crystal Refinement Tutorial

- Input file name is Yb3Al5O12_300K.hkl
 - General file on I

Single crystal data from:

Input file name:

☐ SHELX on F ☐ From G_{rain}index file

☐ SHELX on I ☐ From Full_{prof} file

☐ SHELX HKLF5 ☐ From X_D file

☐ IPDS STOE ☐ Jana-M90

☐ CCD Bruker ☐ General file on F

☐ From CIF file ☒ General file on I

Input format:

$\text{Yb}_3\text{Al}_5\text{O}_{12}$: Jana 2020 Single Crystal Refinement Tutorial

- Choose Neutron
- Temperature is 300 K

Complete/correct experimental parameters

Cell parameters: 11.9182 11.9182 11.9182 90 90 90

Number of input indices: 3 [Info about metrics parameters](#)

1st modulation vector:

2nd modulation vector:

3rd modulation vector:

Radiation:

☐ X-rays [X-ray tube](#)

☒ Neutrons

☐ Electrons

☐ Kalpha1/Kalpha2 doublet

Wave length:

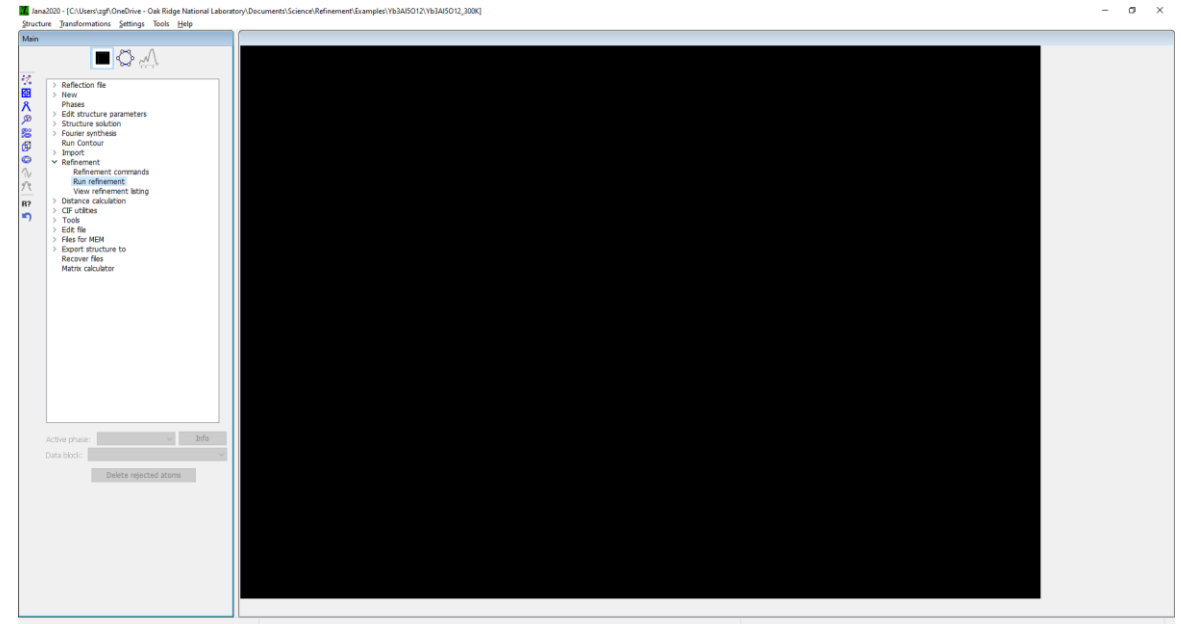
Data collection details:

Temperature:

[Back](#) [Next](#) [Cancel](#)

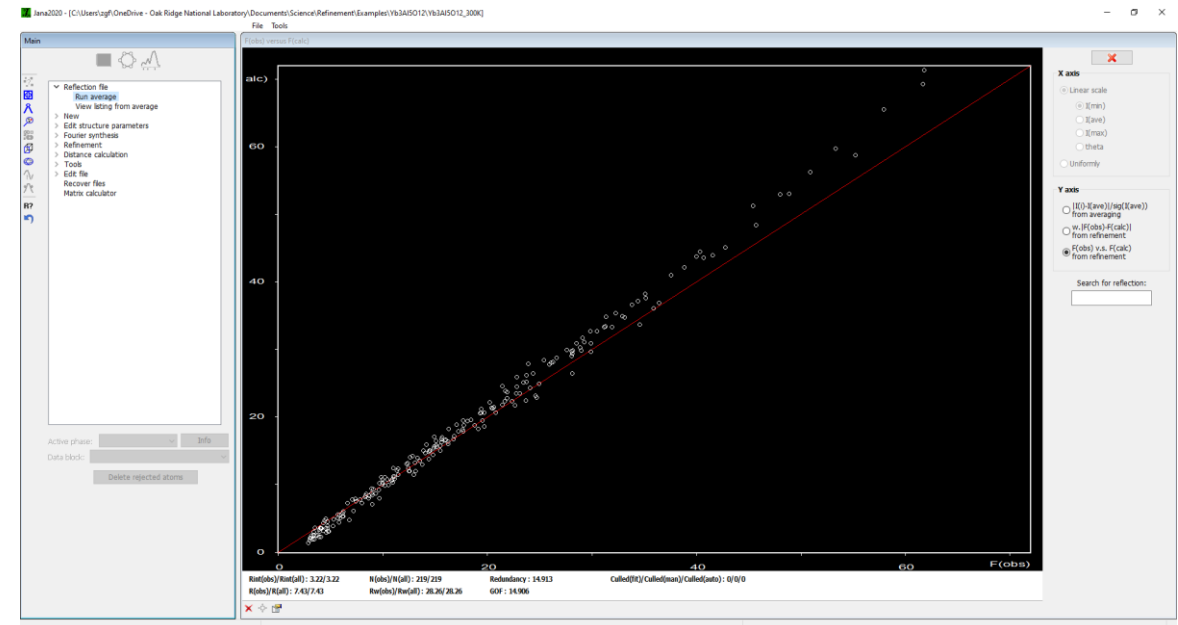
$\text{Yb}_3\text{Al}_5\text{O}_{12}$: Jana 2020 Single Crystal Refinement Tutorial

- Run Refinement
 - Square button



$\text{Yb}_3\text{Al}_5\text{O}_{12}$: Jana 2020 Single Crystal Refinement Tutorial

- Reflection file > Manual culling
- Yaxis F(obs) vs F(calc) from refinement



$\text{Yb}_3\text{Al}_5\text{O}_{12}$: Jana 2020 Single Crystal Refinement Tutorial

OPTIONAL:

- Right click on refinement
- Select/Listing tab
- Skip reflections 15
- Yes+Start

The screenshot shows the 'Refinement commands' dialog box in Jana 2020, with the 'Select/Listing' tab selected. The 'Indicate/Select reflections' section contains the following settings:

- Unobserved reflections: $I < 3 * \text{sig}(I)$ (checked)
- Not matching reflections: $|F(\text{obs}) - F(\text{calc})| > 3 * \text{sig}(F(\text{obs}))$ (checked)
- Skip reflections: $|F(\text{obs}) - F(\text{calc})| > 15 * \text{sig}(F(\text{obs}))$ (checked)
- Apply $\sin(\theta)/\lambda$ limits: $\sin(\theta)/\lambda$ from 0 to max. 10 (unchecked)

The 'How to handle weak reflections:' section has the following settings:

- Print of reflections suppressed: ☐ (unchecked)
- Print of reflections: ☒ before the first cycle and after the last cycle, ☐ after the last cycle, ☐ Not matching reflections, ☐ All reflections
- Print correlation larger than: 0.9
- Print twin/overlap details: ☐ (unchecked)
- Print statistics: ☒ (checked)

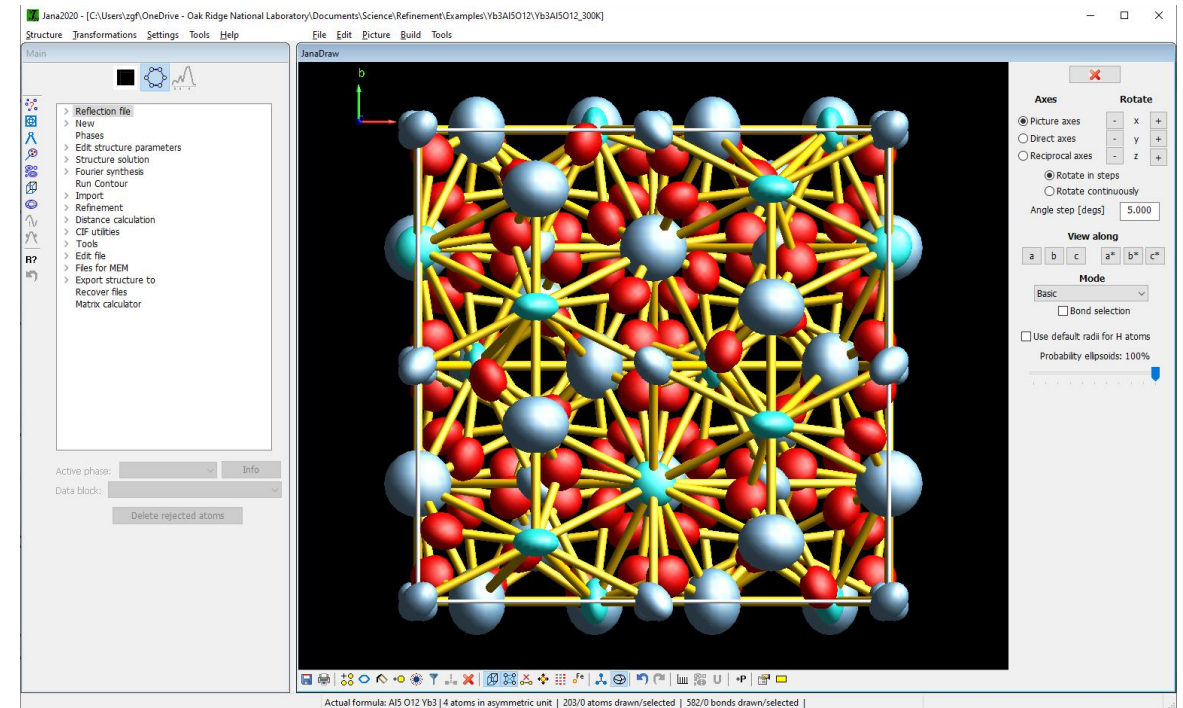
The 'Listing commands:' section is empty.

A dialog box titled 'Do you want to save new commands?' is overlaid on top of the main dialog. It contains four buttons: 'Yes', 'No', 'Yes+Start' (highlighted with a blue border), and 'No+Start'.

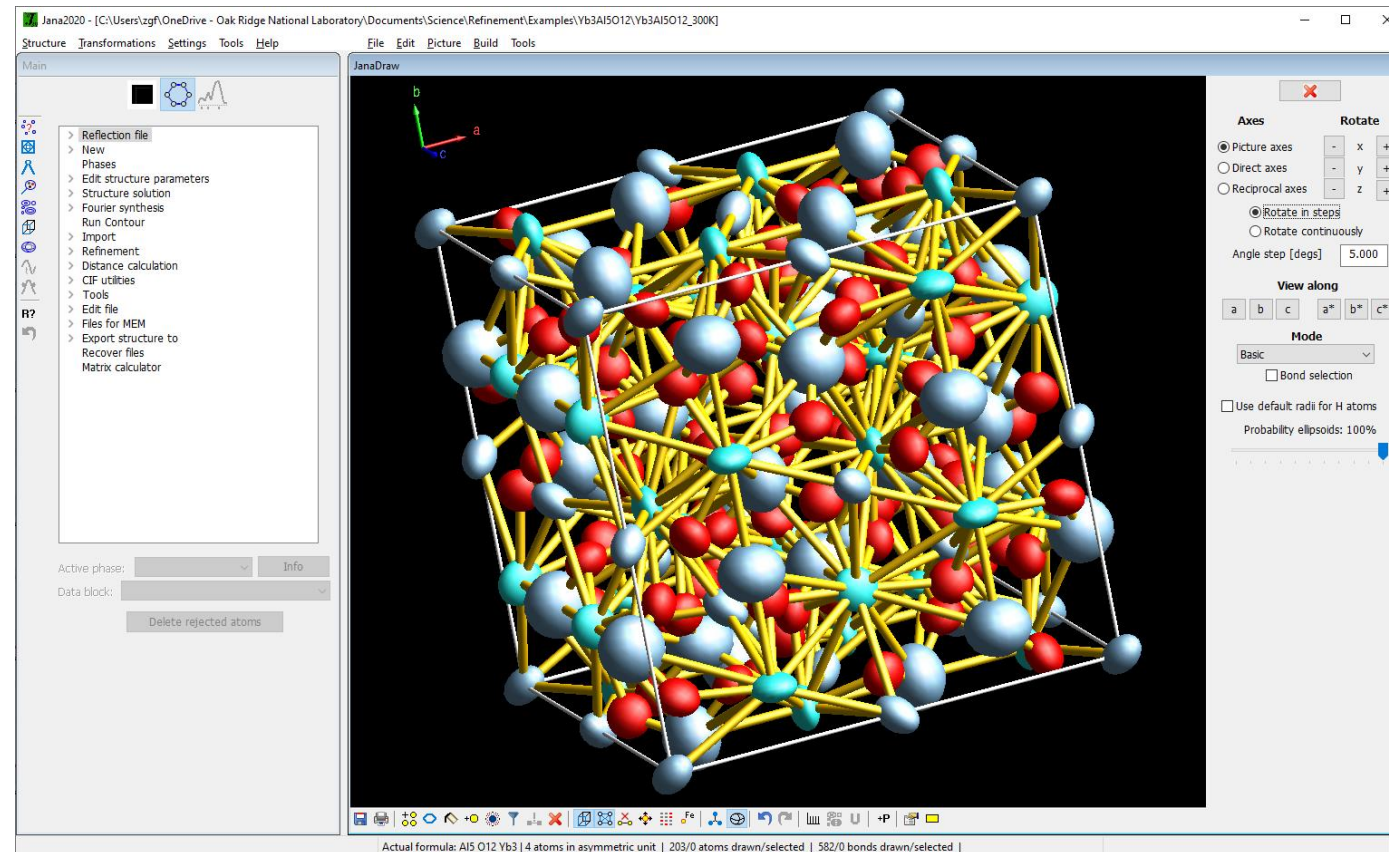
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OPTIONAL:

- Draw only independent atoms
- Fill unit cell
- Draw ellipsoids > slider 99% probability



Yb₃Al₅O₁₂: Jana 2020 Single Crystal Refinement Tutorial



Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- Structure > New
- Navigate to Tb2Ir3Ga9
- Choose Tb2Ir3Ga9_007K.hkl
- Magnetic parent structure nuclear model from CIF
- Choose Tb2Ir3Ga9.cif

Specify type of the file to be imported

Single crystal:	<input type="radio"/> known diffractometer formats
	<input type="radio"/> reflection file corrected for LP and absorption
	<input type="radio"/> start with indexing procedure
Powder data:	<input type="radio"/> various CW formats
	<input type="radio"/> various TOF/ED formats
Structure:	<input type="radio"/> from SHELX
	<input type="radio"/> from CIF
	<input type="radio"/> from SHELX embedded in CIF
	<input type="radio"/> from Jana2020 embedded in CIF
	<input type="radio"/> from XD
	<input type="radio"/> from Jana2000
Magnetic parent structure:	<input type="radio"/> nuclear model interactively
	<input type="radio"/> nuclear model from SHELX
	<input checked="" type="radio"/> nuclear model from CIF
	<input type="radio"/> nuclear model from Jana2006/Jana2020

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Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- Select Tb atom type
 - Use as magnetic atom
 - Magnetic form factor <j0>
- Number of superimposed IRs is 1
 - 0 0 0 propagation vector

Define magnetic propagation vector(s) and form factors

Number of superimposed IRs: 1 GM, k14 (0,0,0) Qa 0 Qb 0 Qc 0

Formula - list of atomic types: Ga Tb Ir

Formula units: 1 Calculate density

Atom type: Tb Own scattering length for neutrons ☒ Use as a magnetic atom

☐ Own form factors Edit

☒ Magnetic formfactor <j0> Tb3+ ☐ + Magnetic formfactor <j2>

☐ + Magnetic formfactor <j4>

☐ + Magnetic formfactor <j6>

☐ Magnetic formfactor <j0>+<j2>

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Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

Specify type of the file to be imported

Single crystal:

- ☐ known diffractometer formats
- ☒ reflection file corrected for LP and absorption
- ☐ start with indexing procedure

Powder data:

- ☐ various CW formats
- ☐ various TOF/ED formats

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Single crystal data from:

Input file name: Tb2Ir3Ga9_007K.hkl Browse

- ☐ SHELX on F
- ☐ SHELX on I
- ☐ SHELX HKLF5
- ☐ IPDS STOE
- ☐ CCD Bruker
- ☐ From CIF file
- ☐ From Graindex file
- ☐ From FullProf file
- ☐ From XD file
- ☐ Jana-M90
- ☐ General file on F
- ☒ General file on I

Input format: (34,2f8.2)

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- Temperature is 7K

Complete/correct experimental parameters

Cell parameters: 13.011 7.542 9.452 90 90 90

Number of input indices: 3 [Info about metrics parameters](#)

1st modulation vector:

2nd modulation vector:

3rd modulation vector:

Radiation:

☐ X-rays [X-ray tube](#)

☒ Neutrons

☐ Electrons

☐ Kalpha1/Kalpha2 doublet

Wave length:

Data collection details:

Temperature:

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Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

Define the reference cell

Cell parameters of the block#113.01107.54209.452090.00090.00090.000
Reference cell parameters derived from the block#113.01107.54209.452090.00090.00090.000
Reference cell parameters original:13.01107.54209.452090.00090.00090.000

Target dimension:

8

1st modulation vector:

2nd modulation vector:

3rd modulation vector:

4th modulation vector:

Max. satellite index:

Accuracy:

Matrix calculator

Apply the transformation

H=

1

*h+

0

*k+

0

*l
K=

0

*h+

1

*k+

0

*l
L=

0

*h+

0

*k+

1

*l

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☐ Twinning

Twining matrices

Number of domains:

1

Data related

Multiply input

☐ HKLF5 file

☐ Import g

Decide what to do:

S.U. of original cell parameters differs from those just imported.

☒ Continue with the old ones
☐ Use the new ones

OK

List of irreps and corresponding kernel symmetries:

Representation	Dimension	Shubnikov space group	Axes	Origin shift	
mGM1+	1	Cmcm	(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>
mGM2+	1	Cm'c'm	(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>
mGM3+	1	Cmc'm'	(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>
			(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>
			(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>
			(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>
			(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>
			(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>
			(1,0,0 0,1,0 0,0,1)	(0,0,0)	<div>Details</div>

Display representations

Modify the set of active vectors

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Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- 8 Shubnikov groups selected
- Next

List of kernels and epikernels:

Shubnikov space group	Axes	Origin shift	Representation	ODP
Cmcm	(1,0,0 0,1,0 0,0,1)	(0,0,0)	mGM1+	(a)
Cm'c'm	(1,0,0 0,1,0 0,0,1)	(0,0,0)	mGM2+	(a)
Cm'c'm'	(1,0,0 0,1,0 0,0,1)	(0,0,0)	mGM3+	(a)
Cm'c'm'	(1,0,0 0,1,0 0,0,1)	(0,0,0)	mGM4+	(a)
Cm'c'm'	(1,0,0 0,1,0 0,0,1)	(0,0,0)	mGM1-	(a)
Cmcm'	(1,0,0 0,1,0 0,0,1)	(0,0,0)	mGM2-	(a)
Cm'cm	(1,0,0 0,1,0 0,0,1)	(0,0,0)	mGM3-	(a)
Cm'c'm	(1,0,0 0,1,0 0,0,1)	(0,0,0)	mGM4-	(a)

Number of epikernels: 8

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

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- Choose Cm'cm'
 - Moment is (M,M,0)
- Continue with the selected Shubnikov group
- Save with a different name than the parent space group
 - Tb2Ir3Ga9_007K_Cmpcmp.m50
- A new Jana2020 instance will be opened
- Run refinement

Select Shubnikov space group :

Shubnikov space group	Atom	Moment	Global	sin(2.Π.x4)	cos(2.Π.x4)
Cmcm Cm'cm Cm'cm' Cm'cm' Cm'c'm' Cmcm' Cm'cm Cmc'm	Tb3	(M,M,0)	(0,M,0)		

Show details

Information: The symbol "M" indicates that the component can have non-zero value.
For more details press the button "Show details".
Magnetic moments are expressed in the parent cell.

Start graphic simulation Start profile simulation

Continue with the selected Shubnikov space group

Back Finish Cancel

Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- Edit structure parameters > Edit basic parameters
- Check twinning (under cell tab)
 - Number of twin domains is 3
- Matrices

Edit basic parameters (cell, symmetry, etc.)

Cell Symmetry Composition Multipole parameters Magnetic parameters

Title of the structure:

Phase label:

Cell parameters:

s.u.'s of cell parameters:

Dimension: Number of composite parts:

☒ Twinning Number of twin domains:

☐ Commensurate case Supercell:

☐ Use simple supercell TZero:

Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- Twin domain #2
 - 60 deg proper rotation in direct base about 0 0 1
 - Up arrow
 - Round to closest rational
- Increment to next domain
- Yes

2nd twinning matrix

Twin domain# Applied to the phase:

$h' = \frac{1}{2}$ *h+ $\frac{3}{2}$ *k+ 0 *l
 $k' = -\frac{1}{2}$ *h+ $\frac{1}{2}$ *k+ 0 *l
 $l' = 0$ *h+ 0 *k+ 1 *l

Six-fold axis in direction (0,0,-1)

Select alternative representative of the coset

Round to closest integers Round to closest rationals

Matrix calculator

Original cell parameters
13.011 7.542 9.452 90.00 90.00 90.00

Twinned cell parameters
13.050 7.519 9.452 90.00 90.00 89.80

Rotation axis:

☒ in direct base ☒ proper rotation
☐ in reciprocal base ☐ improper rotation

Rotation angle:
☐ 0 deg ☐ 90 deg ☐ 180 deg ☐ 270 deg ☐ explicit=>
☒ 60 deg ☐ 120 deg ☐ 240 deg ☐ 300 deg

Esc OK

Question

The difference between the original and transformed cell parameters seems to be too large.

Do you want to accept the twin matrix anyhow?

Yes No

Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- Twin domain #3
 - 300 deg proper rotation in direct base about 0 0 1
 - Up arrow
 - Round to closest rational
- OK
- Yes
- Accept all defaults
- Re-run refinement

3rd twinning matrix

Twin domain# Applied to the phase:

$h' = \frac{1}{2}h + \frac{-3}{2}k + 0l$
 $k' = \frac{1}{2}h + \frac{1}{2}k + 0l$
 $l' = 0h + 0k + 1l$

Six-fold axis in direction (0,0,-1)

Select alternative representative of the coset

Original cell parameters

3.011	7.542	9.452	90.00	90.00	90.00
-------	-------	-------	-------	-------	-------

Twinned cell parameters

3.050	7.519	9.452	90.00	90.00	90.20
-------	-------	-------	-------	-------	-------

Rotation axis:

☒ in direct base ☒ proper rotation
☐ in reciprocal base ☐ improper rotation

Rotation angle:

☐ 0 deg ☐ 90 deg ☐ 180 deg ☐ 270 deg ☐ explicit=>
☐ 60 deg ☐ 120 deg ☐ 240 deg ☒ 300 deg

Question

The program has detected a change of the symmetry/twinning which call for re-creation of the reflection file.

Do you want to re-create refinement reflection file just now?

Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- Edit structure parameters > Edit twin phase/volume fractions
- Refine all

Edit twin fractions

Data block:

twvol2 ☒ twvol3 ☒

Six-fold axis in direction (0,0,-1)

h'		1/2	3/2	0		h
k'	=	-1/2	1/2	0	*	k
l'		0	0	1		l

Refine all Fix all Reset

Esc OK

Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- Edit structure parameters > Edit scale parameters
- Fix all
- Re-run refinement

Edit scale parameters

Data block:

TOverall ☐ sc1am/2 ☐ sc3lam ☐

Maximal number of scales:

scale1 ☐ scale2 ☐ scale3 ☐ scale4 ☐

scale5 ☐ scale6 ☐

Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

OPTIONAL:

- Right click on refinement
- Select/Listing tab
- Skip reflections 15
- Yes+Start

The screenshot shows the 'Refinement commands' dialog box in Jana 2020, with the 'Select/Listing' tab selected. The 'Indicate/Select reflections' section contains the following settings:

- Unobserved reflections: I < 3 *sig(I) ☒ Use unobserved reflections
- Not matching reflections: |F(obs)-F(calc)| > 3 *sig(F(obs))
- ☒ Skip reflections |F(obs)-F(calc)| > 15 *sig(F(obs))
- ☐ Apply sin(th)/lambda limits
- sin(th)/lambda: 0 max. 10

The 'How to handle weak reflections:' section has the following settings:

- Print of reflections: ☒ before the first cycle and after the last cycle
- ☐ after the last cycle
- Print correlation larger than: 0.9
- ☒ Not matching reflections
- ☐ All reflections
- ☐ Print twin/overlap details
- ☒ Print statistics

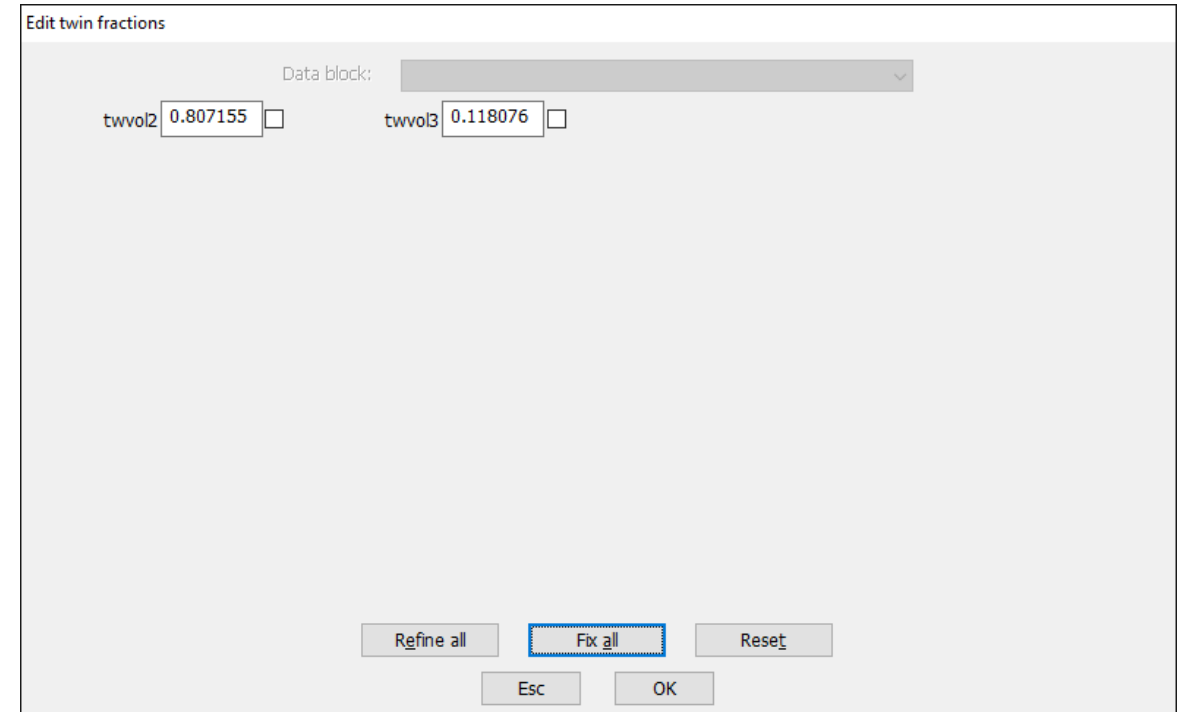
The 'Listing commands:' section is empty.

At the bottom, there is a button labeled 'Define datablocks used in the refinement' and two buttons: 'Esc' and 'OK'.

Overlaid on top of the dialog is a smaller dialog box titled 'Do you want to save new commands?'. It contains four buttons: 'Yes', 'No', 'Yes+Start' (which is highlighted with a blue border), and 'No+Start'.

Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

- Edit structure parameters > Edit twin phase/volume fractions
- Fix all
- Edit structure parameters > Edit scale parameters
- Fix all
- Re-run refinement



Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

Define/Edit atom parameters

Define Edit Multipole parameters Modulation parameters **Magnetic parameters**

3 Select atom(s) from list Atom name: Tb3 Atomic type: Tb

Mx0 8.585977 ☒ My0 -1.238635 ☒ Mz0 0 ☐

Refine all Fix all Reset Show p/sig(p)

Apply site symmetry Show symmetry restrictions

Esc OK

Information from refinement:

RFactors overview

R factors : [308=308+0/5], Damping factor: 0.1000
GOF(obs)= 7.75 GOF(all)= 7.75
Number of reflections excluded due to refinement options: 169+0
R(obs)= 7.53 wR2(obs)= 16.50 R(all)= 7.53 wR2(all)= 16.50
R factors for nuclear reflections : [297=297+0]
R(obs)= 7.58 wR2(obs)= 16.60 R(all)= 7.58 wR2(all)= 16.60
The R-values without taking magnetic contributions:
R(obs)= 36.37 wR2(obs)= 74.87 R(all)= 36.37 wR2(all)= 74.87
R factors for magnetic reflections : [11=11+0]
R(obs)= 5.69 wR2(obs)= 13.43 R(all)= 5.69 wR2(all)= 13.43
Last wR2(all): 17.06 17.11 17.00 17.00 16.95 16.76 16.57 16.54
Maximum change/s.u. : -3.3810 for Uiso[Ga1]

Refining procedure - cycle 86/100

End Parameters Cancel

Tb₂Ir₃Ga₉: Jana 2020 Single Crystal Refinement Tutorial

OPTIONAL:

- Draw only independent atoms
- Fill unit cell
- JanaDraw options
 - Bohr to angstrom ratio is 0.1

JanaDraw options

☒ Display coordinate system

☒ Direct coordinate system
☐ Reciprocal coordinate system

Background:
☐ White
☐ Light blue
☒ Black

Bonds between atoms derived from atomic radii and typical distances expanded by: %

Don't draw bonds being shorter than % of distance derived from atomic radii and typical distances

Atom radius [Ang]: Bond thickness [Ang]: Atom label shift [Ang]:

Atoms having site occupancy less than will be skipped

☒ Reduce atomic radii by occupancy factor Bohr to angstroem ratio:

Delay time for drawing atom labels and visualization of selected atoms: milliseconds

Note: For most graphic cards this delay can be 0. In case of problems try to find the smallest value.

Shine: 0.70

Gloss: 0.00

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