Practical: Refinement of powder diffractogram and extraction of single crystal intensities for $\text{ZnCr}_2\text{Se}_4$

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and

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Goals:
★ Obtain **possible** magnetic structure(s), which are **in agreement** with the symmetry imposed by the propagation vector group and **fit** the powder pattern.
★ Predict intensities for a single crystal experiment.

Methods:
★ Representational analysis for list of possible solutions (SARAh, MODY, pencil).
★ Your favourite refinement package (Fullprof Suite).

It is good to know in advance:
★ The nuclear structure? XRD
★ Any constrains on the moments? Magnetization, Mössbauer,...
★ Order of the transition? Latent heat?
★ Some crystallography... we are solving a structure after all
Normal spinel

\[ \text{A}^\text{T}\text{B}^\text{O}_2\text{X}_4 \]

- Cubic Fd-3m, \( a \sim 10.5 \, \text{Å} \), \( \text{ZnCr}_2\text{Se}_4 \) 10.43-10.52Å nonstoichiometric?
- B-site trigonal antiprism "octahedral" \((u=0.25)\).
- A-site always ideal tetrahedron.
- 2 settings: setting 1 Fd-3mS and setting 2 (standard) Fd-3mZ
What we know about ZnCr$_2$Se$_4$

\[ \mu_{\text{eff}} = 5.5 \mu_B, \quad \mu_{\text{sat}} = 5.7 \mu_B, \quad \Theta_{\text{CW}} \approx 100K \]
\[ \text{Cr}^{3+} \quad (S=3/2) \]
\[ \mu_{\text{eff}} = 5.46 \mu_B, \quad \mu_{\text{sat}} = 6.0 \mu_B, \]

Neutron Scattering Study of ZnCr$_2$Se$_4$
with Screw Spin Structure

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***Brookhaven National Laboratory, Upton, New York, U.S.A.

(Received May 13, 1977)

Neutron scattering experiments have been carried out on a single crystal and powdered specimens of ZnCr$_2$Se$_4$, a helimagnet, above and below the transition at $T_N=21.2$ K. Below $T_N$, crystal belongs to $D_{4h}^{18}$. Atomic parameters of Se can be expressed by a single parameter, $u=0.3847$. Rather small magnetic moment (1.71 $\mu_B$ at 0 K) and the linear dependence of the length of the screw vector, $Q_0$, on the lattice deformation were observed. Anisotropic critical scattering ($J_x/J_y=3$) was measured around $(4-Q_0, 0, 0)$ above $T_N$ and $Q$ dependence of the exchange interaction constant, $J(Q)$, near $Q_0$ was discussed. Recent studies by Plumier et al. were critically referred.

$T_N = 21K$, NTE $< 100K$
$I4_1/ amd, c/a=0.9995$

Ordered moment $1.71 \mu_B$!
Distortion and NTE - 1966!

Fullprof Suite survival kit:

Data
- Single or many patterns

PCR - instructions

Fullprof
- wfp2k, Fp2k

Output files (main)
- *.prf - Winplotr
- *.out - Text
- *.hkl - Text
- *.fou - Text
- *.fst - FPStudio
- *.cif - Text

EdPCR
- (cif, ins, self)
Getting started – preparing nuclear structure (from scratch)

- New PCR
- Import CIF
- Import SHELXL *.ins

FullProf
PCR Editor
Getting started – nuclear structure from scratch

Important things to type-in (General, Pattern, Phases):

1. One experimental pattern (Zn000001c.dat).

2. Wavelength 2.0782Å.

3. Background type – start with 6 order poly
   (we shall convert it later later)

3. CW-neutron powder diffraction

4. One phase:
   a) Nuclear only,
   b) Rietveld,
   c) Gaussian profile.
Pattern

![Pattern Information Window](image)

- Pattern: 1/1
- Weight: 1.0000

Options:
- Data file/Back shape
- Background Type
- Excluded Regions
- Geometry/IRF
- User Scatt. Factors
GSAS, XY or whatever you have

**Pattern/ Data file**

This is needed only if you click "Calculation"
Representational Analysis and Magnetic Structures
3rd August 2011
Georgetown University
Goergetown, DC, USA

Pattern: Geometry

Works better for me

Absorption - may help if your B<0
Always asymmetry.
Title for your phase. If not active add one first.

**Add phase**

**Rietveld nuclear**

Add phase
Phase/s: contribution

This section connects Patterns with Phases. We connect current Phase (1) with previously entered Pattern (1). Pattern (1) was CW-neutron. Make sure the correct one is selected!

BT-1 and our sample. Ask your instrument scientist! Check after 1 cycle if FP accepted it. (May go wrong)

Warning! This will automatically change to "HKL file" during LeBail fits. Reset to "Automatic" if you have problems.
Phase: Nuclear Symmetry

Should work for most standard symbols

Type-in your SG
And hit Symm Op.

This should fill operators.

Check with ITC, when in doubt.

Need this number for SOF,
Getting started – nuclear structure from scratch

Lattice parameter/s (10.48) and atomic positions. (Roughly – refine later).

Atoms
Zn 0.125, 0.125, 0.125 (8a) chemical SOF = 1, we type 0.04167
Cr 0.5 0.5 0.5 (16d) chemical SOF=1, we type ___
Se 0.26, 0.26, 0.26 (32e) chemical SOF=1, we type ___

Chemical SOF = like in GSAS, FOX, RIETAN
FP, Shelxl – crystallographic SOF

CrystSOF = Chem SOF * Site_Multi / General_Multi
Zn_SOF = 1 * 8 / 192 = 0.041666667 ← FP will round it

Make sure it's correct – otherwise your moment will not scale properly.
Getting started – nuclear structure from scratch

Atoms
Lattice and profile
Warning!
Make sure you choose an atom with chemical SOF=1 as Atom #1. Otherwise FP will "correct" you and you will get wrong scaling factor.
Profile

Profile Parameters: Phase 1  Pattern 1

Factors

<table>
<thead>
<tr>
<th>Scale</th>
<th>Overall B-factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10000E-02</td>
<td>0.00000</td>
</tr>
</tbody>
</table>

Coefficients

<table>
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<th>b</th>
<th>c</th>
<th>alpha</th>
<th>beta</th>
<th>gamma</th>
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<tbody>
<tr>
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<td>10.480000</td>
<td>0.00000</td>
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<td>0.00000</td>
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</table>

Your job

Cell Parameters

FWHM / Shape Parameters

FWHM Parameters

<table>
<thead>
<tr>
<th>U</th>
<th>V</th>
<th>W</th>
<th>I0</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.004133</td>
<td>-0.007613</td>
<td>0.006258</td>
<td>0.000000</td>
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</table>

Shape Parameters

Coefficients

Refine FWHM for second wavelength

<table>
<thead>
<tr>
<th>U2</th>
<th>V2</th>
<th>W2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Default but you can write your own if known.

Save yours or use ZnCr2Se4_start.pcr
Refinement............. your job

Try to choose initial scale by hand so you can see peaks!
Then refine a constant background.

Than is your call.
Indexing – was yesterday

Try your luck – that’s an example, which doesn’t look good.

<table>
<thead>
<tr>
<th>Kx</th>
<th>Ky</th>
<th>Kz</th>
<th>R-factor</th>
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</thead>
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<tr>
<td>0.410000</td>
<td>0.175000</td>
<td>0.125000</td>
<td>0.105909</td>
</tr>
<tr>
<td>0.410000</td>
<td>0.125000</td>
<td>0.175000</td>
<td>0.105909</td>
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<td>0.105909</td>
</tr>
<tr>
<td>0.175000</td>
<td>0.125000</td>
<td>0.410000</td>
<td>0.105909</td>
</tr>
<tr>
<td>0.125000</td>
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<td>0.175000</td>
<td>0.105909</td>
</tr>
<tr>
<td>0.125000</td>
<td>0.175000</td>
<td>0.410000</td>
<td>0.105909</td>
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<td>0.155000</td>
<td>0.285000</td>
<td>0.330000</td>
<td>0.273651</td>
</tr>
<tr>
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<td>0.330000</td>
<td>0.285000</td>
<td>0.273651</td>
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<td>0.273651</td>
</tr>
<tr>
<td>0.285000</td>
<td>0.330000</td>
<td>0.155000</td>
<td>0.273651</td>
</tr>
</tbody>
</table>

In fact k is close to (0,0,0.46)

Or if you use only first you may get
k2 = (0.24, 0.24, 0)

Check everything!
Insert second phase and do pattern matching but first lets look into PCR file

There are several blocks that you need to know.

General info – number of phases, type of background, number of excluded regions, input and output options.
Not a good idea to change them by hand except
Nph – number of phases,
Nex – excluded regions,
Nba – type of background.

Pattern properties:
Wavelength, zero shift, background but also number of parameters and cycles.

Phase properties

I am going to change Nph to 2 and copy the lines in light purple.
Second phase and do pattern matching

If everything went fine you should have pcr file with 2 identical phases. Also variables like lattice and shape are constrained to be the same.

Now we are going to:
1) change the name of the phase, **Phases – select Phase 2**, 2) change dropdown list "Calculation" type to "Pattern matching with constant scale factor," 3) remove constrains by unclicking and clicking again. 4) add k-vector and -k since they are not equivalent. 5) and try to refine. It will do Rietveld on Phase 1 and LeBail on satellites.

Pcr file after this stage ZnCr2Se4_Phase2.pcr.
Second phase and do pattern matching

At this point it should be evident that the background is not correctly modelled and the first peak (000)+q has nasty asymmetry.

Phases – Background type

- Linear Interpolation between a set background points with refinable heights

It inputs 10 default lines in the pcr file. Check them under Refinements->Background.
Second phase and do pattern matching

It inputs 10 default lines in the pcr file. Check them under **Refinements- >Background.**

Less is better but use as much as is necessary

ZnCr2Se4_Phase3.pcr
Representational analysis

At this point we have propagation vector and good atomic positions. What may go wrong? Use Basireps first.

Output:

=> Number of Space group: 227
=> Hermann-Mauguin Symbol: F d -3 m
=> Hall Symbol: -F 4vw 2vw 3
=> Table Setting Choice: 2
<cut>
=> Number of elements of G_k:  8
=> Number of irreducible representations of G_k:  5
<cut>
=> Dimensions:  2 1 1 2 2
<cut>
=> The input propagation vector is:
K=(  0.0000  0.0000  0.4600 )
=> K .. IS NOT .. equivalent to -K
<cut>
-> GAMMA(Magnetic):   3 Irep_k(1) + 1
            Irep_k( 2) + 2 Irep_k(3) + 3 Irep_k(4) + 3
            Irep_k(5)

Wrong!
ORDERS OF THE REPRESENTATIONS:

1: 2
2: 1
3: 1
4: 2
5: 2

DECOMPOSITION OF THE MAGNETIC REPRESENTATION INTO IRs OF Gk:

3 TIMES THE REP 1 OF ORDER 2
1 TIMES THE REP 2 OF ORDER 1
2 TIMES THE REP 3 OF ORDER 1
3 TIMES THE REP 4 OF ORDER 2
3 TIMES THE REP 5 OF ORDER 2

Identical to Basireps and Wrong!
Sarah/Kovalev

Used to be correct

ORDERS OF THE REPRESENTATIONS:

1: 1
2: 1
3: 1
4: 1
5: 2

DECOMPOSITION OF THE MAGNETIC REPRESENTATION INTO IRs OF Gk:

1 TIMES THE REP 1 OF ORDER 1
2 TIMES THE REP 2 OF ORDER 1
2 TIMES THE REP 3 OF ORDER 1
1 TIMES THE REP 4 OF ORDER 1
3 TIMES THE REP 5 OF ORDER 2

but since yesterday got overflow

?  

Correct!
MODY/Kovalev
But use setting 1!

Chromium position 0.625, 0.625, 0.625

Number of representations: 5
- REPRESENTATION tau 1, dim 1, occurring 1 time ****
- REPRESENTATION tau 2, dim 1, occurring 2 times ****
- REPRESENTATION tau 3, dim 1, occurring 2 times ****
- REPRESENTATION tau 4, dim 1, occurring 1 time ****
- REPRESENTATION tau 5, dim 2, occurring 3 times ****

Correct! $1+2+2+1+6 = 12 = 4 \times 3$ OK
Let us use SARAh/Kovalev

Look into the end of *.lst2

IR # 1, BASIS VECTOR: # 1 (ABSOLUTE NUMBER:# 1)
1: ( 2 2 0) + i( 0 0 0)
2: ( -2 -2 0) + i( 0 0 0)
3: (1.492 1.492 0) + i( 0 0 0)
4: (-1.492 1.492 0) + i( 0 0 0)

IR # 2, BASIS VECTOR: # 1 (ABSOLUTE NUMBER:# 2)
1: ( 2 2 0) + i( 0 0 0)
2: ( -2 -2 0) + i( 0 0 0)
3: (-1.492 1.492 0) + i( 0 0 0)
4: ( 1.492 1.492 0) + i( 0 0 0)

IR # 2, BASIS VECTOR: # 2 (ABSOLUTE NUMBER:# 3)
1: ( 0 0 4) + i( 0 0 0)
2: ( 0 0 4) + i( 0 0 0)
3: ( 0 0 2.984) + i( 0 0 0)
4: ( 0 -2.984) + i( 0 0 0)

IR # 3, BASIS VECTOR: # 1 (ABSOLUTE NUMBER:# 4)
1: ( 2 2 0) + i( 0 0 0)
2: ( -2 -2 0) + i( 0 0 0)
3: (1.492 1.492 0) + i( 0 0 0)
4: (-1.492 1.492 0) + i( 0 0 0)

IR # 3, BASIS VECTOR: # 2 (ABSOLUTE NUMBER:# 5)
1: ( 0 0 4) + i( 0 0 0)
2: ( 0 0 4) + i( 0 0 0)
3: ( 0 2.984) + i( 0 0 0)
4: ( 0 -2.984) + i( 0 0 0)

IR # 4, BASIS VECTOR: # 1 (ABSOLUTE NUMBER:# 6)
1: ( 2 2 0) + i( 0 0 0)
2: ( -2 -2 0) + i( 0 0 0)
3: (-1.492 1.492 0) + i( 0 0 0)
4: ( 1.492 1.492 0) + i( 0 0 0)

Can we make spiral with moments only in ab plane with one IRREP? YES
Let us use SARAh/Kovalev

Select "promissing" basis functions from IRREP5.

Press "Generate model PCR" for powder.

It will create file Sarah.pcr in the folder with your 227.MAT file.
Let us use SARAh/Kovalev

We need only part of it between the beginning and profile parameters. I am going to replace this fragment in the second phase of our pcr file.
We now have pcr file with 2 phases

When you check - the second phase is now Rietveld (magnetic)
with symmetry given by basis functions.

SARAh can do it for you but you have to know what is going on.

There are 3 corrections that need to be made.
1. Magnetic phase uses now CHEMICAL OCCUPANCIES!!!
2. There is 1 unique chromium atom in magnetic cell and SARAh
writes its SOF as 4. But in fact it is taken into account by the
centering in SG symbol F -1. Since we use F-centering – change
the occupancy to 1.
3. In the Refinement window find Convergence Criterion and
change it back to None.
4. Constrain scale.
It will not refine since it is not yet a spiral.

! Real(0)-Imaginary(1) indicator for Ci
0 0 0 0
!
SYMM X, Y, Z
BASR 2 0 0 0 2 0 0 0 0 0 0 0 0 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0 0 0
SYMM -X+3/4, -Y+3/4, Z
BASR 2 0 0 0 2 0 0 0 0 0 0 0 0 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0 0 0
SYMM Y+1/4, -X+1, Z+1/4
BASR 0 1.332 0 -1.332 0 0 1.492 0 0 0 -1.492 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0
SYMM -Y+1, X+1/4, Z+1/4
BASR 0 1.332 0 -1.332 0 0 1.492 0 0 0 -1.492 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0
!
!Atom Typ Mag Vek X Y Z Biso Occ C1 C2 C3 C4 C5 C6 C7 C8 C9 MagPh
CR1 MCR3 1 0 0.50000 0.50000 0.50000 0.00000 1.00000 0.000 0.000 0.000
0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000
0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00
0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00

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Let us normalize the functions (/2) to see what they are

! Real(0)-Imaginary(1) indicator for Ci

0 0 0 0

SYMM X, Y, Z

BASR 1 0 0 0 1 0 0 0 0 0 0 0 0 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0 0 0

SYMM -X+3/4, -Y+3/4, Z

BASR 1 0 0 0 1 0 0 0 0 0 0 0 0 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0 0 0

SYMM Y+1/4, -X+1, Z+1/4

BASR 0 0.666 0 -0.666 0 0 0.745 0 0 0 -0.745 0 0 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0 0 0

SYMM -Y+1, X+1/4, Z+1/4

BASR 0 0.666 0 -0.666 0 0 0.745 0 0 0 -0.745 0 0 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0 0 0
BASI 0 0 0 0 0 0 0 0 0 0 0 0 0 0

1 = cos 0
0 = sin 0
\( \alpha = 41.759 \)
Sin \( \alpha \) = 0.666 = sa
Cos \( \alpha \) = 0.746 = ca

! Atom Typ Mag Vek X Y Z Biso Occ C1 C2 C3

CR1 MCR3 1 0 0.50000 0.50000 0.50000 0.00000 1.00000 0.00000 0.00000 0.00000
0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000
0.00 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000
Let us normalize the functions (/2) to see what they are

! Real(0)-Imaginary(1) indicator for Ci
0 1 1 0

1=cos 0
0 = sin 0

Sin (α+0) = 0.666 = sa
Cos (α+0) = 0.745 = ca

α=41.759deg
This is the exact angle bewteen Cr planes or
Or
α=2Pi * 0.46/4

If your precision is not good enough i.e.
\( \sin^2\alpha + \cos^2\alpha \neq 1 \)
Extra peaks!!!!
Now we just need coefficients to refine

<table>
<thead>
<tr>
<th>Atom</th>
<th>Typ</th>
<th>Mag</th>
<th>Vek</th>
<th>X</th>
<th>Y</th>
<th>Z</th>
<th>Biso</th>
<th>Occ</th>
<th>C1</th>
<th>C2</th>
<th>C3</th>
<th>C4</th>
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<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td></td>
</tr>
</tbody>
</table>

We want a perfect helix with equal components, so

\[ |C1| = |C2| = |C3| = |C4|, \]
\[ C1 = C2 \text{ and } C3 = C4, \quad C1 = -C3 \]

Which gives

C, C, -C, -C and file ZnCr2Se4_withIRandC.pcr

Warning! If you refine propagation vector – change basis functions.

Make sure that sum of squares your values of sin and cos add to 1.
After several cycles

! Real(0)-Imaginary(1) indicator for Ci
  0 0
!
SYMM X, Y, Z
BASR 1 0 0 0 0 0
BASI 0 0 0 0 1 0
SYMM -X+3/4, -Y+3/4, Z
BASR 1 0 0 0 0 0
BASI 0 0 0 0 1 0
SYMM Y+1/4, -X+1, Z+1/4
BASR 0.7470 0 0 0 0.6648 0
BASI -0.6648 0 0 0 0.7470 0
SYMM -Y+1, X+1/4, Z+1/4
BASR 0.7470 0 0 0 0.6648 0
BASI -0.6648 0 0 0 0.7470 0
!

!Atom Typ Mag Vek X Y Z Biso Occ C1 C2 C3 C4 C5 C6 C7 C8 C9 MagPh
CR1  MCR3 1 0 0.50000 0.50000 0.50000 0.30000 1.00000 2.820 -2.820 0.000
  0.00  0.00  0.00  0.00  0.00  11.00 -11.00  0.00

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Now, how to get single crystal intensities?

Tell FP to write them in different formats available from those 2 tabs.

Write to *.out
Now, how to get single crystal intensities?

Then select more specific information. We want reflection file *.hkl

FP will create

Pcrname.hkl
Pcrnamex.hkl
## Output file

**Pcrname.out** – depending on the choice either integrated or profile integrated.

<table>
<thead>
<tr>
<th>No.</th>
<th>Code</th>
<th>H</th>
<th>K</th>
<th>L</th>
<th>IVK</th>
<th>Mult</th>
<th>Hw</th>
<th>(2\theta/\text{TOF})</th>
<th>(I_{\text{calc}})</th>
<th>(I_{\text{obs}})</th>
<th>Sigma</th>
<th>HwG</th>
<th>HwL</th>
<th>ETA</th>
<th>H1</th>
<th>H2</th>
<th>H3</th>
<th>d-hkl</th>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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<td>5.280</td>
<td>10389.2</td>
<td>10847.0</td>
<td>492.508</td>
<td>0.369009</td>
<td>0.153233</td>
<td>0.409600</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
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</tr>
<tr>
<td>2</td>
<td>1</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>2</td>
<td>1</td>
<td>0.455681</td>
<td>5.280</td>
<td>10389.2</td>
<td>10847.0</td>
<td>492.508</td>
<td>0.369009</td>
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<td>0.5352</td>
<td>6.936775</td>
<td>1.0000</td>
<td></td>
</tr>
</tbody>
</table>

**Pcrname.hkl** – does not list explicitly which k vector and gives multi * \(F^2\).

<table>
<thead>
<tr>
<th>h</th>
<th>k</th>
<th>l</th>
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<th>(2T)</th>
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<th>(F^2)</th>
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**Pcrnamex.hkl** – does explicitly list k vector and gives multi * \(F^2\).

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Number of propagation vectors: 2

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<th>l</th>
<th>n</th>
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<th>(F^2_{\text{calc}})</th>
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<th>Qy</th>
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Problems

Try to find satellite intensities of 551 and 711. Due to perfect degeneracy of d-spacings (similar thing happens in tetragonal groups for 431 and 501) FP makes a mistake.

**Pcrname.hkl**

Seems to be fine but the same F^2.

-7 -1 1 2 0.33803 89.2835 1.04261 1.010 0.2557
-7 1 1 2 0.33803 89.2835 1.04261 1.010 0.2557
-5 -5 1 2 0.33803 89.2835 1.04261 1.010 0.2557
-5 5 1 2 0.33803 89.2835 1.04261 1.010 0.2557

**Pcrname2.hkl**

```
7 1 1 2 24 7.975 12.114 7.0000 1.0000 0.5352 1.47914
4 4 4 1 8 22.766 20.802 4.0000 4.0000 4.4648 1.45548
7 1 1 1 24 9.829 11.639 7.0000 1.0000 1.4648 1.45253
```

Only 711 satellites are listed with intensity and multiplicity including 511 but separate 511 entry.
Remarks:

☆ Check SOF and ATZ! - Scaling of moments.

☆ Before removing a phase fix all variables.

☆ If a variable does not change its value during refinement and you are sure it should, check the "code" if it is not damped.

☆ "Sum Iobs=0, do you really have reflections" – check scale and lattice parameters, then profile, B and SOFs.

☆ www.cryst.ehu.es - Bilbao Crystallographic server

☆ Use FPstudio to get moments!
Final remarks:

🌟 Trust no one (software).

🌟 Have fun!