FullProf tutorial on crystal structure and commensurate magnetic structure

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• Orthorhombic structure: *Pnma (No. 62)*

a= 9.143086, b= 3.784552, c=13.416915;

- Octahedra CrSe_{6;}
- Magnetic transition 75 K:

Files provided for this tutorial

Time-of-flight diffraction data at POWGEN: PG3_42702-2_300K.dat PG3_42704-2_10K.dat

Instrumental resolution file: PG2018B_HighRes_60Hz_b2_Ddep.irf

- cif file for crystal structure CrSbSe₃.cif
- Final PCR files PG3_42702-2_300K.pcr PG3_42704-2_10K.pcr (representation analysis)
- Supporting information
- FullProf_CM.PDF (step-by-step instructions)



POWGEN Peak Profile

TOF profile has Convolution of back-to-back exponentials with pseudo-Voigt (a linear combination of Gaussian and Lorentzian)

6 refinable parameters: position, intensity, α , β , σ and γ Position:

TOF(microseconds) = Zero + Dtt1 * D + Dtt2 * D^2 + Dtt_1overD)/D

Exponentials: Alpha largely affects the sharpness of the leading TOF edge of each peak; larger values mean sharper front edges. Beta terms affect the trailing TOF edge in the same way.

$$\alpha = \alpha_0 + \frac{\alpha_1}{d} + \frac{\alpha_q}{\sqrt{d}}$$
 and $\beta = \beta_0 + \frac{\beta_1}{d^4} + \frac{\beta_q}{d^2}$

Gaussian and Lorentzian width: Sig terms affect the Gaussian shape component of the peak profiles; larger values result in broader peaks. The coefficients describe the sig and Gamma values as follows:

$$\sigma^{2} = \sigma_{0}^{2} + \sigma_{1}^{2} d^{2} + \sigma_{2}^{2} d^{4} + \begin{pmatrix} \sigma_{q}^{2} \\ d^{2} \end{pmatrix} \longrightarrow \sigma_{q}^{2} d \text{ GSASII used}$$

$$\gamma = \gamma 1 * d + \gamma 2 * d^{2} + \gamma 0$$

HIGH FLU>

- All these coefficients marked as green were implemented in the 2021 versions of FullProf by Juan rodriguez-Carvajal recently.
- > In other TOF instruments and previous versions of FullProf, there lack Dtt_loverD, β_q and σ_q .

Formats of the instrumental resolution files

Traditional Instrumental resolution files for old version FullProf

(2011 B- 2021 A cycle)

Instru	umental resolut	ion fun	ction f	or POWGEN/	SNS 2018_B cycle ireso: 5
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! Тур	pe of profile f	unction	: back-	to-back <u>ex</u>	pon * pseudo- <u>Voigt</u>
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END					

New Instrumental resolution files for new version FullProf (2021 B cycle-future)

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20		1	.54497	844.20258	19.59661	0.11365	0.15412	0.00000	
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Listed the global coefficients without Dtt_loverD, β_q and σ_q for old version FullProf.

Provides d-dependent numerical coefficients for new 2021 versions of FullProf (used in this tutorial)

Allows a more accurate determination of instrumental resolution.



Comparison of the refinement quality using old and 2021 versions of FullProf and IRFs

Old versions of FullProf and old IRFs until 2020

2021 versions of FullProf and new IRFs



- New 2021 versions of FullProf and IRFs improved the refinement quality
- Old PCRs can be read automatically by the new 2021 versions of FullProf and converted to the new format PCR files

Recommend using the new version FullProf to refine POWGEN data for getting an improved refinement on the peakshape

Exercise steps

- I. Import the cif file and create a PCR file for a single datafile at 300 K (T>Tm);
- II. Refine the data to get accurate structural parameters including the lattice constants, atomic positions, temperature factors and peak profile parameters at 300 K;
- III. Save the PCR file at 300 K as a new PCR to refine the structural parameters at 10 K (T<Tm).
- IV. Identify magnetic peaks/contributions and determine the propagation vector.
- V. Symmetry analysis to obtain irreducible representations and Basic vectors using SARAh.
- VI. Select a magnetic model and add it as the 2nd phase in the PCR.
- VII. Refine the magnetic phase to obtain the magnetic structure and ordered moment.
- VIII. Display the magnetic structure using FpStudio and Vesta.



I. Import the cif file and create a PCR file for a single datafile at 300 K (T>Tm)



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II. Refine the data to get accurate structural parameters at 300 K

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Pattern-Datafile/Peak shape tab



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Phase-tab





Use Winplotr to get the BG of the data

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Automatic search of background points



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Refinement-Profile tab

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=> Phase: 1
=> Bragg R-factor: 15.01
=> RF-factor : 10.72
=> Normal end, final calculations and writing...

=> CPU Time: 0.898 seconds => 0.015 minutes

=> END Date:04/11/2021 Time => 15:52:18.456



20000 40000 60000 80000 100000120000140000160000180000220000 TOF (usec)



Add the refinement on the peak profile coefficients Gam_2, Gam_1, and Gam_0

Profile Parameters: Phase 1 Pattern 1



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	- Information	
	Title, type of job: Rietveld, Integrated Intensities, Simulated Annealing,	General
	Type of Pattems, profile, background, diffraction geometry, user-given scattering factors	Patterns
Editor	Phase name, type of calculations (JBT), ATZ, contribution to patterns, symmetry,	Phases
	Number of cycles, relaxation factors, access to patterns and phases (atoms and profile)	Refinement
	Constraints definitions, adding, deleting, modifying	Constraints
	Fixing range of parameters, distances, angles, magnetic moments and linear restraints	Box/Restraints
17 21 20 29 33 37 41 45 49 20(*)	Output options for patterns and phases: Reflection lists, Fourier, distances, BVS	Output
Copyright (c) 2002-2005. JGP - JRC		

=> Phase: 1

=> Bragg R-factor: 7.922

=> RF-factor : 7.961

=> Normal end, final calculations and writing...

=> CPU Time: 3.594 seconds

=> 0.060 minutes

=> END Date:04/11/2021 Time => 15:49:06.360



20000 40000 60000 80000 100000120000140000160000180000220000 TOF (usec)

CAK RIDGE National Laboratory

SPALLATION NEUTRON SOURCE

Refinement-Atoms tab (to refine atomic positions and temperature factors B)

Ta Refinement Information	>
Cycles of Refinement: 5	
Stop Criterium of Covergence Forced Termination when shifts < 0.10 x E.S.D. Others: None	Relaxation Factors for Shifts Atomic 1.00 Anisotropic 1.00 Profile 1.00 Global 1.00
Reflections ordering C Only at the first cycle Pattern 1 Pattern 2 Pattern 3 Pattern 4 Pattern 5 Pattern (Bragg R-Factor excluding reflections limiting excluded regions Phase 1 Phase 2 Phase 3 Phase 5 Phase 6 P
Refinement weighting model Background Image: Comparison of the second	OK Atoms Prop. Vectors Cancel Patterns C 1 C 2 C 3 C 4 C 5 C 6 C 7 Profile Micro-Structure
Reduction factor of number of data points:	HKL Shifts Further Parameters

Refinement Information	>
Cycles of Refinement: 5 ÷	
Stop Criterium of Covergence Forced Termination when shifts < 0.10 x E.S.D. Others: None	Atomic 1.00 Anisotropic 1.00 Profile 1.00 Global 1.00
Reflections ordering	Braco R-Factor excluding reflections limiting excluded regions
Pattern 1 Pattern 2 Pattern 3 Pattern 4 Pattern 5 Pattern	Phase 1 5 Phase 3 Phase 4 Phase 5 Phase 6 P.
Refinement weighting model Image: Construction of the second s	Atoms Prop. Vectors Cancel Patterns
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C Unit Weights Micro-Absorption	Profile Micro-Structure
Reduction factor of number of data points:	HKL Shifts Further Parameters

	-				-												
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Atom # 1	Sb	Sb		0.0295	V	0.25	000	0.657	86	1.0000		0.5000	D 🗌 Isc	tropic			
Atom # 2	Cr	Cr		0.1549	X	0.25	000	0.044	60	1.0000		0.5000	lsc	tropic			Refine B_isc
Atom # 3	Se3	Se		0.1718	30 🔽	0.25	000	0.484	50	1.0000		0.5000	lsc	tropic	_		
Atom # 4	Se2	Se		0.2848	30 🔽	0.25	000	0.212	80 🔽	1.0000		0.5000	lsc	tropic		~	Ketine B_aniso
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# # < Special Form #	Factors SASH-Typ Spherical	e I	Matrix	; j=1		=2	=3	N. Coeff.	Indice	s #1	#2	#3	#4	#5	#6	1~ ^	
# # # # \$pecial Form #	Factors SASH-Typ Spherical Spherical	ie I	Matrix	(j=1		=2 j	=3	N. Coeff.	Indice	s #1	#2	#3	#4	#5	#6	~	

- => Bragg R-factor: 8.640
- => RF-factor : 6.833

=> Conv. not yet reached -> [Max] Shift(Biso_Cr_ph1)/(eps*Sigma)= -1.08 abs> 1

=> Normal end, final calculations and writing...

=> CPU Time: 3.750 seconds => 0.062 minutes

=> END Date:04/11/2021 Time => 17:15:08.698



20000 40000 60000 80000 100000120000140000160000180000220000 TOF (usec)



III. Save the PCR file at 300 K as a new PCR to refine the structural parameters at 10 K (T<Tm).

Image: Constraint of PCR Files File Editor Image: Constraint of PCR Files Image: Constrating to PCR Files Ima	⁶ 響 ⁶ , • 。 ② ×	- 0 X	Title		×	
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∧ Hide Folders



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Save

Use the BG for PG3_42704-2_10K.dat



CAK RIDGE National Laboratory REACTOR SOURCE

Refinement-Profile tab: unselect scale factor, Gam_2, Gam_1 and Gam_0 to refine lattice constants, the atomic positions and B factors



IDGE HIGH FLUX ISOTOPE

National Laboratory REACTOR

SPALLATION NEUTRON

SOURCE

III. Identify magnetic peaks/contributions and determine the propagation vector





IV. Symmetry analysis to obtain irreducible representations and Basic vectors using SARAh

a). Use SARAh webRefine – FullProf:

http://fermat.chem.ucl.ac.uk/spaces/willsgroup/web-software/sarah-refine-fullprof/

SARAh webRefine – FullProf

Two pieces of advice for using SARAh webRefine : 1. change your browser settings <evaluate>, it will look like nothing is happening for a few seconds. Look in the tab

-Andrew (February 2022)



SARAh webRefine – FullProf

Two pieces of advice for using SARAh webRefine : 1. change your browser settings to allow you to select where you save downloads (and overwrite file: <evaluate->, it will look like nothing is happening for a few seconds. Look in the tab '4. Help and Strategies' for more information.

-Andrew (February 2022)

Wolfram

1. Conventional basis vectors (as projected)	2. Stationary v combinatio	rector 3. Exchange mul ns	iplets 4. Help and strategies
Method 1. Conventional Select command (go	te for magnetic	ed basis vectors	metic phase present):
\Box Cr1 $\Gamma_1 \psi_1$	\Box Cr1 $\Gamma_4 \psi_1$	\Box Cr1 $\Gamma_7 \psi_2$]
$\Box \operatorname{Crl} \Gamma_1 \psi_1$ $\Box \operatorname{Crl} \Gamma_2 \psi_1$	$\Box \operatorname{Crl} \Gamma_4 \psi_1$ $\Box \operatorname{Crl} \Gamma_5 \psi_1$	$\Box \operatorname{Crl} \Gamma_7 \psi_2$ $\Box \operatorname{Crl} \Gamma_8 \psi_1$	
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 Cr1 Γ₁ ψ₁ Cr1 Γ₂ ψ₁ Cr1 Γ₂ ψ₂ Cr1 Γ₃ ψ₁ 	$\Box \operatorname{Crl} \Gamma_4 \psi_1$ $\Box \operatorname{Crl} \Gamma_5 \psi_1$ $\Box \operatorname{Crl} \Gamma_6 \psi_1$ $\Box \operatorname{Crl} \Gamma_6 \psi_2$	$\Box \operatorname{Crl} \Gamma_7 \Psi_2$ $\Box \operatorname{Crl} \Gamma_8 \Psi_1$	

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The generated PCR with the same name has been downloaded!



b): Use two softwares:

Fill in space group, K-vector and Atoms in SARAh-Representational Analysis

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ATOM	1:	(	0	2		0)	+	i(	0	0	0)
ATOM	2:	(	0	-2		0)	+	i(	0	0	0)
ATOM	3:	(	0	2		0)	+	i(	0	0	0)
ATOM	4:	(	0	-2		0)	+	i(	0	0	0)
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ATOM	1:	(	2	0		0)	+	i(	0	0	0)
ATOM	2:	(	2	0		0)	+	i(	0	0	0)
ATOM	3:	(	-2	0		0)	+	i(	0	0	0)
ATOM	4:	(	-2	0		0)	+	i(	0	0	0)
IR #	2, 1	BASIS	VECTOR:	#	2	(ABS	OLU	TE NU	MBER:#	3)	
ATOM	1:	(	0	0		2)	+	i(	0	0	0)
ATOM	2:	(	0	0		-2)	+	i(	0	0	0)

****	*									
ATOM	4:	(	0	0	2)	+	i(	0	0	0)
ATOM	3:	(	0	0	-2)	+	i(	0	0	0)
ATOM	2:	(	0	0	-2)	+	i(	0	0	0)

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ATOM	1:	(	2	0	0)	+	i(	0	0	0)
ATOM	2:	(	2	0	0)	+	i(	0	0	0)
ATOM	3:	(	2	0	0)	+	i(	0	0	0)
ATOM	4:	(	2	0	0)	+	i(	0	0	0)

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ATO	M	3:	: (	0	0		2)	+	i(	0	0	0)
ATO	M	4 :	: (	0	0		-2)	+	i(	0	0	0)
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IR #	4,	BASIS	VECTOR:	#	1	(ABS	OLU	JTE NU	MBER:#	6)	
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ATOM	2	: (	0	-2		0)	+	i(	0	0	0
ATOM	3	: (	0	-2		0)	+	i(	0	0	0
ATOM	4	: (	0	2		0)	+	i(	0	0	0
****	**										

#### Magnetic space group

MAGNETIC SPACE GROUPS (THE BLACK AND WHITE SHUBNIKOV GROUPS)

BV#			Shubnikov Grou	up Group number
1	IR# :	1	Pnma	62.441
2	IR# :	2	Pn'm'a'	62.449
3	IR# :	2	Pn'm'a'	62.449
4	IR# :	3	Pnm'a'	62.447
5	IR# :	3	Pnm'a'	62.447
 6	IR# 4	4	Pn'ma	62.443
7	IR# !	5	Pn'ma'	62.448
8	IR# (	6	Pnm'a	62.444
9	IR# (	6	Pnm'a	62.444
10	IR# '	7	Pn'm'a	62.446
11	IR# '	7	Pn'm'a	62.446
12	IR# (	8	Pnma'	62.445



#### Select a magnetic model and add it as the 2nd phase in the PCR

Generate PCR of  $\Gamma_3$  using **SARAh refine**.  $\Gamma_3$  moment in the *ac* plane

					😻 SARAh-Refine	– 🗆 ×
File <u>QSAS Controls</u> <u>EullProf Controls</u> Jopas Controls <u>View bar</u>	Yew Features!	😻 SARAh-Refine		- 🗆 ×	Eile GSAS Controls EullProf Controls Topas Controls View basis vectors Tools Help	*Video Help* New Features!
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Generate/Edit.pcr file K-Vector Search	SARAh-Refine			V instantions		SARAh-Refine for FullProf A.S. Wills (Version: 9.0.13 May 2011)
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	A.S. Wills, Physica B 276,680(2000). Program available from	← → < ↑ 📴 > This PC > Windows (C:) > Program File:	s (x86) → SARAh	✓ Ŏ Search SARAh	SARAh- Generate/Edit FullProf 2K *.PCR File	B & Wille Dhusion B 276 680(2000) .
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		Tax	6/2/2021 9:54 AM MATLAB Data	30 KB	$\square A(1) \Gamma(6) \Psi(9)$	8) mm2 mmm' Pnm'a 62.444 9) mm2 mmm' Pnm'a 62.444
		JaxReturn	7/8/2019 3:50 PM MATLAB Data	4 KB	$\Box A(1) \Gamma(7) \Psi(10)$	10) 2/m m'm'm Dn'm'a 62.446
		travel	9/24/2021 12:39 PM MATLAB Data	51 KB	Select PCR Edit	it Fie
		通 相机上传 1 sarah225	2/28/2020 11:04 AM MATLAB Data	54 KB	Select Basis Vectors Make	FST file Click on the basis vector label in the main window to move to its Point Group and
		<b>。</b> 银行卡			Select All Unselect All	Shubnikov Group in this list.
		<ul> <li>OneDrive - Oak Ri</li> </ul>			Option	The symmetry of a basis vector will be highlighted when it is selected
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					Γ(1) Γ(2) Γ(5) Select	have a valid Point or Shubnikov Group. C/B indicates a change of is required. See Ist2 file for
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19 BASR 2 0 0 0 0	ATOM	2: (.34279, .75, .54469)				
20 BASI 0 0 0 0 0	•			$\sim$		
22 BASR 2 0 0 0 0	2 ATOM	3: (.84279, .75, .95531)				
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33 !> Profile Parameters for Pattern # 1						.9
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# Fix all the structural parameters and initialize nonzero C1 and C2 coefficients to the two basic vectors

## Fix all the parameters and only initialize nonzero C1 to the first basic vector only



Two reasons why the small AFM coefficient C2 should be zero:

1). The refinement quality is similar using small C2 or zero C2;

2). The nonzero AFM coefficient C2 produces pure magnetic peak (100). The POWGN data shows there is no detectable (100) intensity, indicating that C2 should be zero.



The fitting can be further improved by refining the scale factor, lattice parameters, profile parameters. Remember to constrain all of them to be the same for both nuclear and magnetic phases.

Bragg R-factor: 7.513

=> Normal end, final calculations and writing...

1.562 seconds

2

=> Magnetic R-factor: 9.653

5.997

RF-factor

=>

=>

=> Phase:

=> CPU Time:

=> 0.026 minutes

#### Modify PCR directly



#### Final refinement result:



20000 40000 60000 80000 100000120000140000160000180000220000220000 TOF (usec)



#### VIII. Display the magnetic structure using FpStudio or Vesta.

**FpStudio** Combined fst file FILE for FullProf Studio: generated automatically by FullProf 📖 🛄 🔞 🎆 🖓 🏭 💓 👶 👘 💹 🕺 🔘 2 !Title: CrSbSe3 nuclear phase Code File PG3_42704-2_10K Refine 3 SPACEG P n m a 4 CELL 9.150326 3.783540 13.331617 90.0000 90.0000 90.0000 DISPLAY MULTIPLE 5 BOX -0.15 1.15 -0.15 1.15 -0.15 1.15 6 ATOM Sb 0.02909 0.25000 0.65861 Sb 7 ATOM Cr Cr 0.15737 0.25000 0.04474 8 ATOM Se3 Se 0.17096 0.25000 0.48400 9 ATOM Se2 Se 0.28632 0.25000 0.21412 10 ATOM Sel Se 0.50108 0.25000 0.60829 12 { 13 LATTICE P 14 K 0.00000 0.00000 0.00000 15 SYMM x,y,z 16 MSYM u,v,w,0.0 17 MATOM CR1_1 Cr 0.15737 0.25000 0.04474 SCALE 1.0 GROUP

NU, dN4 J, 16 Jaffeel sand Proc your Unit			18         SRP         1         1           19         MATOM CR1_2         2         2           20         SRP         1         1           21         MATOM CR1_3         2         3	2.56154 Cr 2.56154 Cr 2.56154	0.00000 0.00000 0.34263 0.75000 0.00000 0.00000 0.84263 0.75000 0.00000 0.00000	0.00000 0.00000 0.54474 SCALE 1.0 GR( 0.00000 0.00000 0.95526 SCALE 1.0 GR( 0.00000 0.00000	0.00000 OUP 0.00000 OUP
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#### **CAK RIDGE** National Laboratory SPALLATION NEUTRON SOURCE

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POWGEN workshop "Getting the Most from Your POWGEN Data", June 15-17, 2023

FullProf tutorial on crystal structure and commensurate magnetic structure

Please contact me if you have any question or comments. Thanks!

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