GSAS-II tutorial on crystal and commensurate magnetic structure

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

ORNL is managed by UT-Battelle for the US Department of Energy



Time-of-flight powder diffractometer POWGEN

High resolution powder diffractometer to study crystal, magnetic and local structure of

polycrystalline materials

Freq (Hz)	WL center	WL min	WL max	dmin	dmax	Qmin	Qmax	Bank
60	0.533	0.15	1.066	0.075	7.50	0.82	83.45	0
60	0.800	0.27	1.333	0.134	8.00	0.76	46.88	1
60	1.500	0.97	2.033	0.485	13.00	0.48	12.95	2
60	2.665	2.13	3.198	1.070	21.00	0.30	5.87	3
60	4.797	4.26	5.33	2.140	38.00	0.17	2.94	4

Resolution: best resolution $\Lambda d/d$: 0.8x10⁻³



2018B POWGEN High Resolution 0.025 CW=0.8 Å 0.020 ₿ 0.015 CW=1de [ta] 0.005 0.000 d (Å) HR CWL 0.7 HR CWL 1.5 HR CWL 2.665

Dependent on wavelengths and d (or 2theta)

~ 30%: high profile publications: Nature materials, Nature energy, Nature physics, Nature chemistry, PRL, Nature Communications, PRX, Advanced materials, Angewandte Chemie International Edition, JACS, etc;

Total publication number and instrument H index of POWGEN are both the 1st place among all ORNL beamlines;



Autoreduced POWGEN data vs CW neutron data



Ready for Rietveld refinement or PDF analysis

TOF to D spacing (FullProf):

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

(D in Angstroms)



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 + Ott_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:

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

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

> In other TOF instruments, there lack Dtt_loverD, β_q and σ_q .



Overview of the Rietveld softwares to refine magnetic structures

Commensurate magnetic order

FullProf: representation analysis or magnetic space group;

GSAS-II: magnetic space group only;

TOPAS: representation analysis or magnetic space group;

JANA: representation analysis and magnetic space group;

Incommensurate magnetic order

FullProf: representation analysis or magnetic superspace group

JANA: representation analysis and magnetic superspace group



Basic information on CrSbSe₃



- H=0.1 T 30 **—∎—** H//a *⊢ H//b* -H//c M(emu/g) 20 1/2 (mol Oe/emu) /b H=0.1 W fitting T~200 K 20 10 100 200 T (K) 300 (100 200 300 400 0 T (K)
- Orthorhombic structure: *Pnma (No. 62) a*= 9.143086, *b*= 3.784552, *c*=13.416915;
- Magnetic transition 75 K from the magnetization

HIGH FLUX ISOTOPE

SOURCE

National Laboratory REACTOR

• Octahedra CrSe₆

Files provided for this tutorial

Time-of-flight diffraction data at POWGEN: PG3_42702_300K.gsa PG3_42704_10K.gsa

Instrumental resolution file:
 2018B_HighRes_60HzB2_1p5

 cif file for crystal structure CrSbSe₃.cif

Final PCR files CrSbSe3_N.gpx CrSbSe3_1 mag_3.gpx

Supporting information GSAS_II_QZhang.PDF (step-by-step instructions)



Data file for GSAS-II

INSTPRM

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‡ PI	Y. Qu										
ŧ 1 1	Histogi	ams									
ŧ Fi	le gene	erated	by Mantid								
‡ In	strumer	nt: POW	GEN								
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	1434	15.9621	37489	2	89.493	3223846	5		4.5	829991	100
	1435	57.4325	53787	3	318.844	801503	3		4.7	7235943	398
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1	#GSAS-II instrument parameter file; do not add/delete items POWGEN 2018B!
2	2:-0.465205938282
3	fltPath:63.183
4	beta-q:0.0
5	Bank:2.0
6	sig-1:-852.163368943
7	2-theta:90.0
8	sig-q:797.184446197
9	sig-0:-203.060186472
10	s1g-2:298.195588039
11	Zero:-26.810325593
12	airc:22597.8753723
13	X:-4.50393449632
19	Azimuth 10.0
15	110.34314063
17	alpin 0.12001030100
18	Deta-1.0.0059005250012
19	Turne PNT
20	difB:4.70563778582
21	difA:-4.59435624908
22	



Exercise steps

Identify magnetic peaks in CrSbSe₃;

- Refine the structure of CrSbSe₃ at 10 K (T<Tm);</p>
- > Refine the magnetic structure of $CrSbSe_3$ at 10 K;
- Check if small Mz (AFM component) is real;
- > To publish the magnetic structure and refinement plots;



Identify magnetic peaks in CrSbSe₃



At 10 K, the intensities at high *d* part and low *d* part both increased: different origins! Two ways to determine the structural parameters in $T < T_N$ (or T_C) before refining magnetic structure:

1). Refine the data in T >T_N (or T_C) firstly. Then use them as initial parameters and only refine the atomic positions, B factors, and lattice constants in T<T_N (or T_C).

2). Exclude the high-d part of data that involves magnetic contribution and only refine low and middle d data on the structural part in TOF<68,000 µs

Refine the structure of CrSbSe₃

I. Import datafile and set the limit



🐝 GSAS-II project: <unnamed project>





File-save project!

II. Import the phase

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III. Refine background and scale factor

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- Limits	
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Sample Parameters	v multiplier 1.0
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CrSbSe3	

🐺 GSAS-II project: CrSbSe3_July6tH	1.дрх		
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IV. Refine lattice constants

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-50 -

TOF, μs





V. Refine atomic coordinates and thermal parameters

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Restraints	2	Se3	Se		0.17206	0.25000	0.48457	1.0000	m(y)	4	1	0.0106	D	
Rigid bodies	3	Se2	Se		0.28529	0.25000	0.21334	1.0000	m(v)	4	1	0.0106	0	
PWDR PG3_42704_10K.gsa Ba	4	Se1	Se		0.50254	0.25000	0.60913	1.0000	m(y)	4	1	0.0089	D	
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National Laboratory REACTOR

HIGH FLUX ISOTOPE

SPALLATION NEUTRON SOURCE

VI. Refine peak profile coefficients

In "Instrument Parameters" tab:



HIGH FLUX ISOTOPE

ational Laboratory | REACTOR

NEUTRON

VII. Virtualizing crystal structure

Site

✓ Sb

✓ Cr

∽ Se

Sb

Cr

Se3

Se2

Se1

1.41

1.41

1.29 1.04

1.04

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1.04

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Use VESTA software (jp-minerals.org/vesta/en/download.html) to open it directly for publication:

Export cif file:



Let us add the high-TOF data to refine the magnetic structure!



Refine the magnetic structure of CrSbSe₃ I. Add the high TOF data back and refine background



II. Determine the magnetic propagation vector





Magnetic peaks sit on the nuclear peak positions!

$$\boldsymbol{k} = \boldsymbol{Q}_M - \boldsymbol{Q}_N = \boldsymbol{0}$$



III. Obtain all the possible magnetic models with *k*=0

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🐺 GSAS-II project: CrSbSe3_N.gpx

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beta

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90.000

SAS-II project: CrSbSe3_N.gpx

Data	Calculate Import	Export	Cell I	ndex/Refine	1	Help									
Project: (CrShSe3 N.gpx	Indexing	cont	rols:											
Note	book														
Cont	rols	Max Nc/	Nobs	4			- Sta	rt Volum	e 25			Use M20/(X	20+1)?		
- Cova	riance	Select Bra	avais	Lattices for i	ndex	ing:									
- Cons	traints	Cubic-	F		ubic-	I		Cubic-l	0		frigonal-R	🗌 Trige	onal/Hexag	jonal-P	
Restr	aints	Tetrage	onal-	l 🗌 Te	trage	onal-P		Orthor	ombic	-F 🗌 (Orthorhom	bic-l 🗌 Orth	norhombic	A	
Rigid	bodies	Orthor	rhom	bic-B 🗌 O	rthor	hombi	c-C	Orthor	nombic	-P 🗌 I	Monoclinic	-I 🗌 Mor	nọclinic-A		
PWD	R PG3_42704_10K.gsa	Mono	clinic	-C 🗌 M	onoc	linic-P		Triclinio							
	imits	Coll Test	o. n		Char	ي ادا يا ي			0.5	o/					
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	ample Parameters eak List	Unit cell:	a =	9.15209		÷	b=	3.78432		÷	c = 13	.33414	÷		
In	ndex Peak List	V	ol -	461 820		_									
R	nit Cells List eflection Lists														
- Phase	es	Magnetic	c sub	group cells f	rom E	Bilbao	k-SUBC	-SN	IAG for	Pnma	; kvec1=(0,	0,0):			
C	rSbSe3_1			Space Gp	Тгу	Keer		Λj	nSup	Trans	Vec	а	Ь	c	alpha
		1		Pn'm'a'	\checkmark	\checkmark	1	1 1	1	a,b,c	0,0,0	9.15209	3.78432	13.33414	90.00
		2	_	Pn'ma'			1	1 1	1	a,b,c	0,0,0	9.15209	3.78432	13.33414	90.00
		3		Pnm'a'		\checkmark	1	1 1	1	a,b,c	0,0,0	9.15209	3.78432	13.33414	90.00
🐮 GSAS	S-II project: CrSbSe3_July6th.	арх		1							-	- 🗆 X	-		
File Dat	ta Calculate Import Exp	port Selector	, c	e Help											
- Proje	ct: C:\Users\qzg\Dropbox (4 General Da	ta	Fourier map				Models	Map peak	es MC/S/	A RMC Tex	ture Pawley ref	1		
Co	ontrols	Phase name: CrS	bS	Search map Charge flipping				Space gro	ap: P	n m a	Modulated	?	?		
Co	ovariance	Refine unit cell:		4D Charge flippi	ing			c= [13.33416		Vol = 461.824				
⊕-Re	estraints	Density: 5.906		Clear map				Γ.							
Ri	igid bodies WDR PG3_42704_10K.csa Ba	Elements	[MC/SA				<u> </u>	1						
	Comments	Isotope	1	Multi MC/SA Transform				5	·						
	Limits Background	No. per cell	[Compare		-]						
	- Instrument Parameters	Atom weight	-	Select magnetic	/subgr	oup phas	e								
	Sample Parameters Peak List	Bond radii	1.50	Protein quality	26		1140		-						
	- Index Peak List	angle rauli	ii 2.20	1	.30		1.40		-						
	Unit Cells List Reflection Lists	fault color					1.50								
- Pł	hases CrSbSe3	Pawley controls:	Do P	awley refinement	? Paw	ley dmin:	1.0	Pawley o	- Imax: 100	0.0 Pa	wley neg. wt.:	0.0			
		Fourier map contr	ols: Ma	p type:	~	Reflecti	on sets:	✓ Select	eflection	sets					
		Map grid step: 0.	25	Peak	utoff 9	% 50.0									
		Charge flip contro	ls: Refle	ection sets: 🔍	Select	reflectio	n sets N	ormalizing e	lement:	None					
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		MC/SA schedule:	log	~ slop	ie: 0.9										
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						_									
Í -															
disabled	make in PWDR/Unit Colle														
			~												

Magnetic Space Group: P n' m' a' The lattice is centrosymmetric primitive orthorhombic The Laue symmetry is mmm The magnetic lattice point group is m'm'm' Multiplicity of a general site is 8 The inversion center is located at 0,0,0 The equivalent positions are: (1) X, Y, Z, (1) (2) 1/2-X, 1/2+Y, 1/2+Z, (mx)

Magnetic space group information

 \times



Make new magnetic phase	×	
Select magnetic space group		
(1) Pn'm'a': (a.b.c) + (0.0.0)	^	
(2) Pn'ma'; (a,b,c) + (0,0,0)		
(3) Pnm'a'; (a,b,c) + (0,0,0)		
(4) Pn'm'a; (a,b,c) + (0,0,0)		
(5) Pnma'; (a,b,c) + (0,0,0)		
(6) Pnm'a; (a,b,c) + (0,0,0)		
(7) Pn'ma; (a,b,c) + (0,0,0)		
(8) Pnma; $(a,b,c) + (0,0,0)$		
(9) Pn 21a; (a,b,c) + (0,0,0) (10) Pn 21'a'; (a,b,c) + (0,0,0)		
(10) F121 a, (a,b,c) + (0,0,0)	•	
ОК	Cancel	
* OAK RI		FLUX SPALLATION
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Ci Vacional La	our y TREAC	TOR TOORCE

Save as a new gpx file



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^	Name	Date modified	Type	Size			
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File game: CrSbSe3_1	mag_1						-
Save as type: GSAS-II pr	piect file (*.gox)						

Refine m_x and m_z , and peak profile coefficient X:

🐝 GSAS-II project: CrSbSe3_1 mag_1.gpx

<u>File</u> Data <u>Calculate</u> Import Export | Select tab Edit Atoms Compute | <u>H</u>elp



Visualizing the magnetic structure

In "Draw Atoms" tab:



Poor refinement quality

W GSAS-II project: CrSbSe3_1 mag_	1.gpx														
<u>File Data Calculate Import Ex</u>	port Select	tab E	dit Ator	ns Cor	npute	<u>H</u> elp									
Project: C:\Users\qzg\Dropbox (General	ata A	toms	Draw O	ptions	Draw Atoms	RB Mo	dels M	lap peaks	MC/SA	RMC	Texture	Pawley	reflect	ions
Notebook Controls		Name	Туре	refine	×	у	z	frac	Мх	Му	Mz	site sym	mult	I/A	Uiso
Covariance Constraints	0	Cr1	Cr+3	м	0.1564	9 0.25000	0.04390	1.0000	1.3911		-0.41	88 m'(y)	4	1	0.00176

🐺 GSAS-II project: CrSbSe3_1 mag_1.gpx

Eile Data Calculate Import Export | Select tab Edit Figure Compute Restraints Rigid body | Help



"Draw atom" tab will be updated

🐝 GSAS-II project: CrSbSe3_1 mag_1.gpx

<u>File</u> Data <u>Calculate</u> Import Export | Select tab Edit Figure Compute Restraints Rigid body | <u>H</u>elp

■ Project: C:\Users\qzg\Dropbox (General	Data	Atoms	Draw Opti	ons D	raw Atoms	RB Model	s Map p	oeaks	MC/SA	RMC	Texture	Pawle	y reflecti	ons
Notebook		Nam		~		-	Mv	My	Mz	Sum	0.	Style	Label	Color	1/4
Controls		Nam	e iype	^	y	2	INIX	iviy	IVIZ	Jyin	ΟP	Style	Laber	COIOI	WA.
Covariance	0	Cr1	Cr+3	0.15649	0.250	0.04390	1.3911	0.0000	-0.41	88 1	vd\	W balls			1
Constraints	1	Cr1	Cr+3	0.34352	0.750	0.54390	-1.3911	0.0000	-0.41	88 2+0,0	0 vd\	W balls			1
Restraints	2	Cr1	Cr+3	0.84352	0.750	0.95610	-1.3911	-0.0000	0.41	88 -1+1,	1,1 vd\	W balls			1
Rigid bodies	3	Cr1	Cr+3	0.65648	0.250	0.45610	1.3911	-0.0000	0.41	88 -2+1,	1,1 vd\	W balls			1
PWDR PG3_42704_10K.gsa Bi															



Canted AFM order with magnetic space group Pn'm'a' can be excluded.



V. Select magnetic space group Pnm'n' for the refinement





Save as a new GSAS-II file!



Refine the components Mx and Mz of magnetic moment

File Data Calculate Import Export Select tab Edit Atoms Dute Help Project: C:\Users\qzg\Dropbox (🐝 GSAS-II project: CrSbSe3 mag_3.	дрх												_		×
Image: Project: C:\Users\qzg\Dropbox (General Data Atoms Draw Options Draw Atoms RB Models Map peaks MC/SA RMC Texture Pawley reflections Notebook Notebook	File Data Calculate Import Ex	port Select	tab E	dit Ator	ns 🕐	pute	Help									
Notebook Name Type re2 3 y z frac Mx 3.y Mz site sym mult I/A Uiso Controls 0 Cr1 Cr+3 M 0.15659 0.25000 0.04370 1.0000 1.0000 m'(y) 4 I 0.001	Project: C:\Users\qzg\Dropbox (General D	ata A	toms	Draw O	ptions	Draw Atoms	RB Mod	lels Ma	ap peaks	MC/SA	RMC 1	Texture	Pawley	reflect	tions
Covariance 0 Cr1 Cr+3 M 0.15659 0.25000 0.04370 1.0000 1.0000 1.0000 m'(y) 4 I 0.001	Controls		Name	Туре	re 2	3	у	z	frac	Мх	3 .y	Mz	site sym	mult	I/A	Uiso
Constraints	Covariance	0	Cr1	Cr+3	М	0.15659	0.25000	0.04370	1.0000	1.0000		1.000) m'(y)	4	I	0.00111

Calculate-refine:





General	Data	Atoms	Draw O	ptions [Draw Atoms	RB Mo	dels 🕴 N	Aap peaks	MC/SA	RMC	Texture	Pawley	reflec	tions
	Nam	e Type	refine	x	у	z	frac	Mx	Му	Mz	site sym	mult	I/A	Uiso
0	Cr1	Cr+3	м	0.15649	0.25000	0.04390	1.000	2.6208		0.176	7 m'(y)	4	I.	0.00176

FM alignment along the a axis; AFM alignment along the c axis.



Check if small Mz is real

🗱 GSAS-II project: CrSbSe3_1 mag_3_test.gpx

File Data Calculate Import Export | Select tab Edit Atoms Compute | Help

Project: C:\Users\qzg\Dropbox (General	Data A	toms	Draw O	ptions	Draw Atoms	RB Mo	dels 🛛 N	lap peaks	MC/SA	RMC	Texture	Pawley	reflec	tions	
Notebook Controls		Name	Туре	refine	x	у	z	frac	Мх	My	Mz	site syn	a mult	I/A	Uiso	
Covariance	0	Cr1	Cr+3	М	0.15649	0.25000	0.04390	1.0000	2.6208		0.166	i9 m'(y)	4	I	0.001	76



a. Zero (100) magnetic peak: moment along the *a* axis.



b. Highest magnetization with H//a: moment along the *a* axis.

ACTION SUPERITOR SUPERITOR

— C

c. Similar refinement quality if Mz=0

	General	Data	Atoms	Draw O	ptions	Draw Atoms	RB Mo	dels M	ap peaks	MC/SA	RMC 1	exture	Pawley	refle	ctions	
Γ		Nam	e Type	refine	x	у	z	frac	Мх	-0	Mz	site sym	mult	I/A	Uiso	
	0	Cr1	Cr+3	м	0.15649	0.25000	0.04390	1.0000	2.6214		0.000) m'(y)	4	I	0.001	76



Hold phase variable	×
Select variables to hold (Will not be varied, even if vary flag is set) Name Filter:	
35) 1::A4 ase Cr5bSe3 mag_3 Reciprocal metric tensor component 4 36) 1::A5 ase Cr5bSe3 mag_3 Reciprocal metric tensor component 5 37) 1::A4 be Cr5bSe3 mag_3 Reciprocal metric tensor component 5 37) 1::A4 be Cr5bSe3 mag_3 Reciprocal metric tensor component 4 38) 1::A4 be Cr5bSe3 mag_3 Reciprocal metric tensor component 4 39) 1::A4 be Cr5bSe3 mag_3 Reciprocal metric tensor component parameter, x 39) 1::A4::0 Atom Cr_0 in Cr5bSe3 mag_3 Reciprocal metric meant parameter, z 40) 1::A1:so:0 Atom Cr_0 in Cr5bSe3 mag_3 Reciprocal metric coordinate, x 41) 1::A1:so:0 Atom Cr_0 in Cr5bSe3 mag_3 Atomic coordinate, x 43) 1::A4:0 Atom Cr_0 in Cr5bSe3 mag_3 change to atomic coordinate, y 44) 1::A1:so:0 Atom Cr_0 in Cr5bSe3 mag_3 change to atomic coordinate, z	
OK Cancel	

Refinement results, Rw =3.901							
1	Detailed results are in C:\Users\qzg\Dropbox (ORNL)\POWGEN\NXS\202\CrSbSe3_ mag_3_test_mz_zero.lst	_1					
	Final Reduced Chi^2: 5.298 (before ref: 5.298)						



. .



General	ral Data Atoms		ns	Draw O	ptions	Draw Atoms	s RB Models I		/lap peaks	p peaks MC/SA		Texture	Pawley reflections		tions
	Nar	ne Ty	pe	refine	x	у	z	frac	Мх	Му	Mz	site sym	mult	I/A	Uiso
0	Cr1	Cr	+3	М	0.15649	0.25000	0.04390	1.0000	2.6360		0.000	0 m'(y)	4	I.	0.00176

Μ=(2.64,0,0) μ_B



Visualizing the magnetic structure

In "Draw Atoms" tab:

Ferromagnetic order











To publish the results

I. 3D magnetic structure

Export to cif file: Export- phase as- quick cif



Cr

SPALLATION NEUTRON

SOURCE

II. Refinement plots Convert TOF to d



Click P button to open a new window

Publication plot creation ext size 11 🗸 🗸 Line widths 1 Tick size $_{6}$ \sim width $_{1}$ \sim Obs type + v size 5 v width 1 ~ obs calc bkg zero diff ••••• Show V V V V Include in legend V V V \mathbb{N} \square Color Hard copy Pixels/inch: 600 Width (in): 8.0 Height (in): 6.0 File format: Portable Document Format, pdf 600 - obs — calc — bkg 500 _____ diff CrSbSe3 CrSbSe3 mag 3 Normalized intensity 000 000 000 100 0 10 Δa 0 -10 10 2 8 AA Cancel Save



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

GSAS-II tutorial on crystal and commensurate magnetic structure

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

zhangq6@ornl.gov

