Basic Powder X-ray Diffraction Analysis

Import your data files

Powder data from:						
File name i3_ABDC.dat			Browse			
O MAC format						
GSAS format						
Riet7 formats		UXD format (Siemens/Bruker)				
 ILL D1A/D2B (Rietveld-Hewat for 	ormat)	 Jana2000 format 				
ILL D1A/D2B standard format		PANalytical XRDML				
O ILL D 1B/D20		O Rigaku formats				
O LLB G4.1		 Huber formats 				
O Saday format		Stoe raw data format				
O PSI format		O Free format of I values				
○ <u>1</u> 1-BM		• Free format of 2th, I ,[sig(I)]				
	Show details about	the selected format				
Debye-Scherrer method						
Bragg-Brentanno method - Fixe	d Divergence Slit					
Bragg-Brentanno method - Varia	able Divergence Slit					
<u>A</u> nother/unknown method						
	Back	Next	Cancel			

Important! Rename your .xyd file to .dat

Input your "guessed" lattice parameters

Complete/correct experimental parameters						
Cell parameters:	eters: 25.7262 25.7262 90 90 90					
Target dimension:	3		Info about metrics parame	ters		
⊙ <u>X</u> -rays X-ray to	ube		Polarization cor	rection:		
O Neutrons			 Circular polarization 			
O Electrons			O Perpendicular setting	g Info		
Kalpha 1/Kalpha 2 doub	let		Parallel setting	Info		
Wave length 1.5405	1		O Guinier camera			
			Linearly polarized be	am		
			Monochromato	r parameters:		
			Perfectness	0.5		
Temperature 293			Glancing angle	13.28815	Set glancing angle	
			Back Next		Cancel	
		_			cuncer	

Edit your M50 file

	Define/modif	y basic structural parameter	5:
ell Symmetry	Composition	Multipole parameters	Magnetic parameters
ucture			
l parameters 25.7	7262 25.7262 25.7262 90	90 90	
.d.'s 0.00	00427 0.002121 0.000481	1000	
nension 3			
	•		

Important!

Change the space group to the one from the .cif file

Cell Symmet	ry Composition	Multipole parameters	Magnetic parameters
Space group	3m		Select from li
Origin shift 0 0 0	9		
	The operat	ors derived from the group symbol	
(1) x y z		▲ <u>L</u> oad ->	
(2) -x -y z			<- Add <- Rewrite
(3) -x y -z		Delete or	perator Clean out
(4) x -y -z			
(5) z x y			
(6) z -x -y		Cell centering	
(7) -z -x y			
(8) -z x -y			Complete the set
(9) y z x			
(10) -y z -x		Defin	e local symmetry operators
(11) y -z -x			
(12) -y -z x		T	

Edit your powder diffraction profile

Important! Refine your lattice parameters Important! Refine your peak shape

Important! Refine your background

Powder options	Powder options	Powder options		
Cell Radiation Profile Asymmetry/Diffractometer Sample/Experiment Corrections Various	Cell Radiation Profile Asymmetry/Diffractometer Sample/Experiment Corrections Various	Cell Radiation Profile Asymmetry/Diffractometer Sample/Experiment Corrections Various		
Cell parameters	Peak-shape function	Background		
a 25.7262 🖌 b 25.7262 🖌 c 25.7262 🖌	C Gaussian Cutoff 8 *FWHM	Egendre polynomials Number of terms 20		
alpha 90 beta 90 gamma 90	□ Lorentzian GU □ LX □ □	Edit background		
	Pseudo-Voigt GV O Lxe	bdkg1 0 🗸 bdkg2 0 🗸 bdkg3 0 🗸		
	O Modified Lorentzian GW 5 ✔ LY 0 ✔	bdg4 0 🗸 bdg5 0 🖌 bdg6 0 🗸		
		bdkg7 0 🗸 bdkg8 0 🗸 bdkg9 0 🗸		
	Anisotropic particle broadening Broadening direction	bckg10 0 🗸 bckg11 0 🗸 bckg12 0 🗸		
	Anisotropic strain broadening	bdig13 0 🗸 bdig14 0 🗸 bdig15 0 🗸		
	Mone	bdig16 0 🗸 bdig17 0 🗸 bdig18 0 🗸		
	O Axial method	bckg19 0 V bckg20 0 V		
	Insor method Edit tensor parameters	<u>Kenne ali</u> <u>Fix ali</u> <u>Kešet</u> Snow <u>Disig(p)</u>		
		Esc Ok		
Fer Ok	Esc Ok	Esc Ok		

Edit your refinement profile

	Refine o	ommands	
Basic	Select/Listing	Various	Modulation
lumber of cycles	100 🚔 📃 Use Marguart techni	que 💿 Sigma weight	t Instability factor 0.01
amping factor	0.1 Fudge facto	🖌 Use Wil	son's modification
		O Unit weight	
✓ Use dynamical	LS method => if Rw is increased by 10°	% reduce the damping b	y a factor 2
	After 3 🚔 cyd	es try to enlarge it back.	
 Check for cont 	vergence => stop if max(change/s.u.) \leq	0.05 in 1	consecutive cycles.
 Disable atoms 	having too large isotropic ADP parameter	=> ADP(iso) limit for disablin	g 0.2
✓ Automatic refi	nement keys	Apply Berar's correction	
 Automatic syn 	nmetry restrictions 1	Frequency of	e Bail decomposition
Refinements of	on F(obs)**2		
Apply electron			
Simulation run			
After last cycl	e call Fourier		
Correct for la			
Randomize at	omic coordinates Random seed	Maximal random displa	acement in Ang
Waring: the ra	andomize procedure will apllied just once duri	ing the next run	
	Esc	Ok	

Ready to start refining!

Refinement

At the end of the refinement check your ptofile



Profile R factors :[2937/23+1], Damping factor: 0.1000 GOF = 11.53 Rp = 29.32 wRp = 36.08 Last wRp: 58.03 54.22 50.81 47.74 44.96 42.44 40.13 38.02 Maximum change/s.u. : 17.9576 for LY

Refining procedure - cycle 15/100			
End	Parameters	Cancel	

Refinement (modify your profile and repeat)

Powder options						
Cell Radiation Profile	Asymmetry/Diffractometer	Sample/Experiment	Corrections	Various		
Background						
Legendre polynomials	Number of terms 20					
Chebyshev polynomials	Use 1/x term					
Cos-ortho background	Edit background					
Cos-GSAS background						
Import manual background						
Define excluded regions						
Shift parameters						
shift 0	sycos 0		sysin 0			
			-			
	Esc	k				

When your powder pattern is close to the experimental one refine the shift (Edit Profile Icon)

Use your data

Report your statistics after refinement converges GOF, Rp and wRp

> Profile R factors :[2937/24+1], Damping factor: 0.1000 GOF = 5.05 Rp = 10.60 wRp = 15.81 Last wRp: 15.81 Maximum change/s.u. : 0.0457 for bckg2

> > Regular end of REFINE program
> >
> >
> > Open the listing?
> >
> >
> > Yes
> > No
> >
> >
> > It remains
> > 4 sec - press space for pause

Report your final lattice parameters (Edit M50 Icon)

Plot your data (Profile Viewer Icon)





X-ray diffraction: The basic principle



Miller Indices (hkl) of the atom planes



The concept of Dimensional Reduction



C. C. Stoumpos, M. G. Kanatzidis, Acc. Chem. Res. 2015, 48, 2791.

Androulakis, J., Peter, S. C., Li, H., Malliakas, C. D., Peters, J. A., Liu, Z., Wessels, B. W., Song, J.-H., Jin, H., Freeman, A. J. Kanatzidis, M. G. (2011), Adv. Mater., 23: 4163–4167



Powder patterns of 2D materials



C. C. Stoumpos, D. H. Cao, D. J. Clark, J. Young, J. M. Rondinelli, J. I. Jang, J. T. Hupp, M. G. Kanatzidis, Chem. Mater. 2016, 28, 2852

Preferred growth orientation Intermediate Members



D. H. Cao, C. C. Stoumpos, O. K. Farha, J. T. Hupp, M. G. Kanatzidis, *J. Am. Chem. Soc.* **2015**, *137*, 7843 C. C. Stoumpos, D. H. Cao, D. J. Clark, J. Young, J. M. Rondinelli, J. I. Jang, J. T. Hupp, M. G. Kanatzidis, *Chem. Mater.* **2016**, *28*, 2852