Ultima IV Powder XRD

MPU-4 Standard Configuration

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Do NOT Remove from XRD lab!

1. Turn On the Diffractometer

 Turn on the Haskris (cooling water for X-Ray tube).
 Flow rate: ~4 L/min;
 Temperature: 65 – 69 K (the compressor will turn on when reaching 69 K). 2) Turn on the power of Ultima IV (90° clockwise turn). The OPERATE LED will be flashing and remain green after ~20 seconds.



3) Switch the X-Ray enable key ON (90° clockwise turn).





2. Aging the X-Ray Tube

- 1) Login on the PC (pw: bworld).
- 2) Double-click XG Operation Icon (desktop).
- 3) Option \rightarrow Control (Control Mode).

- 4) Option \rightarrow Property \rightarrow choose 'morning' \rightarrow OK.
- 5) Click 'Execute aging' button to start aging.
- 6) The instrument will be ready in about 1 hour.

		Post-aging XG condition	- Aging da	ata		R.	
-ray control	- Shutter ctrl	The present condition	morning			New	Deleti
				kV [mA	Time (sec) 🔺	
		Aging information		20	5	300	
March apprend		kV minimum step : 1 kV	2	25	10	120	
Execute agin	9	mA minimum step : 1 mA		30	20	120	
	0 60	kV setting range : 20 - 60 kV	5	40	20	120	
		mA setting range : 2 - 60 mA	6	40	25	120	•
	0 60	Maximum kW : 2.20 kW	7	40	30	120	
			8	40	35	120	
Load : 0.04 KM	0 2.20	1	9	40	40	120	
			10	40	44	120 🔻	

2. Aging the X-Ray Tube – Status

When aging finished:



Option \rightarrow Monitor (Monitor Mode):



3. Prepare and Load Samples

- Prepare the samples using Sample Plates or directly load your samples if they have special shapes (samples with rough face may affect the quality of the XRD pattern).
- Press the DOOR LOCK button (flashing and beeping), then slide open the door.
- Load the samples onto the Center of the MPU-4
 Sample Holder. For samples with a thickness
 between 0-4mm , use the thicker Sample Holder;
 for 4-8mm samples, use the thin Sample Holder.
- Slide close the door and Press the DOOR LOCK button again.



4. Automatic Alignment (1)

- 1) Double-click Automatic Alignment to start the software.
- 2) Tick the Theta Alignment and click Execute to start.
- 3) A pop-out window will ask you to install a sample and make sure proper Slits is chosen. Click OK to proceed.





4. Automatic Alignment (2)

- 4) A second pop-out window will ask you to install sample and absorber. Do NOT click OK!!!
- 5) Get three 0.3mm Cu absorber and put them into the absorber holder. Open the door and load the absorber into the Filter Holder (refer to picture on next page).

VBScrip	t Alignment confirmation						
i	Warning!						
V	Please install sample plate.						
	Please install absorber to the Filter holder.						
	Please adjust the number of sheets of the absorber so that X-ray intensity becomes 3,000-150,000 cps of range. (Reference value : 40kV-40mA>Cu absorber 0.9mm) (Reference value : 40kV-40mA> Al absorber 0.4mm + DHL 2.0mm)						
	OK Cancel						
*							

4. Automatic Alignment (3)

- 6) Remove the 10mm DHL slit. Close and Lock the door.
- 7) Click Ok to start the Automatic Alignment. It will take about 10 20 mins, depending on samples.
- 8) After completion, click Save to load the alignment results. Exit the software.
- 9) Important: replace the Cu absorber with Cu K_{β} filter, and insert the 10mm DHL slit.



5. Set up the Experiments (1)

- 1) Double-click Standard Measurement.
- 2) File \rightarrow Open to open the mcd file for your group.
- 3) Edit the Folder name and File name.
- 4) Double-click Condition # to open measure condition.

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	2								
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1000									
No.	Use	Print	Folder name	File nan	ne	Sample Name	Attach.	Condition	Next
1	No	No	C:\Windmax\Data\ume0	H3P04-offset.ra	Browse		1	9	Cont
2	No	No	C:\Windmax\Data\jiz258	ZeroBackground	Browse		1	8	Cont
3	No	No	C:\Windmax\Data\Takon	Si-Al-20-2.raw	Browse		1	7	Cont
4	No	No	C:\Windmax\Data\jiz258	Si-MPU4-BB-Kbe	Browse		1	8	Cont
	Yes	No	C:\Windmax\Data\jcb14	IE079_Fe05Zr08	Browse		1	8	Cont
5					Browse				Cont
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5			Click the No and	a then the	COPYP				
5			Click the No and	a then the	Browsell				Cont.
5			Click the No and		Browse				Cont.,

5. Set up the Experiments (2)

- 1) An example measuring condition shown below.
- 2) You may use the Default Condition button to create default measuring conditions for Inorganics or Organics.
- 3) Make sure power level at 40 kV and 44 mA.

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J	No. Use	₽	Comment	4	Start angle	Stop angle	Sampling W.	Scan speed	EL kV	mA	
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6. Start the Measurement

- 1) Click 'No' under 'Use' to change it to 'Yes'.
- 2) Start the measurement by clicking the top left button.

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Attack	hment ;	: М	lultipurpose attachment 4 fc	or Standard						
Endin	iq proci	ess :	Init position	XG ending:after P	Present conditi	ion 🔻				
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No.	Use	Print	Folder name	File nam	ne	Sample Na	me	Attach.	Condition	Next
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6. Start the Measurement – Status

- 1) The measuring diffraction pattern will be opened on a new window.
- 2) Do NOT attempt to open the cabinet door during the measurement.



7. Turn Off the Diffractometer

- 1) Close all the windows, except the XG operation.
- 2) Go to the XG Operation window:
 - Option \rightarrow Control
 - Option \rightarrow Property \rightarrow Choose 'Shutdown'
 - Start aging. It will take ~10 mins.
 - Close the XG Operation window.
- 3) Turn the X-Ray enable key back to upright position.
- 4) Turn off the main power of the diffractometer.
- 5) Turn off the Haskris.

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Post-aging XG condition		-Aging dal	:a			
Xray off		Shutdowr	n		New	Delete
		Г	kV [mA	Time (sec)	
Aging information			40	44	60	
kV minimum step : 1	kV	2	40	40	60	
mA minimum sten : 1	mΔ	3	40	35	60	
		4	40	30	60	
kV setting range : 20 - 60	k٧	5	40	25	60	
mA setting range : 2 - 60	mΑ	6	40	20	60	
Maximum kW : 2.20	kW	7	35	20	60	
		8	30	20	60	
		9	25	15	60	
		10	20	10	60 🔻	

8. Convert RAW file to ASCII

🛗 Binary -> ASCII Conversion	- [×								
File Help										
	Execute	Exit								
File setting Conversion General ASCII Type										
Input File Name Folder: B:\XRD\Data-PXRD\jiz258\2018-03										
Si-ASC10-BB.raw										
Output File Name Folder: B:\XRD\Data-PXRD\jiz258\2018-03										
Si-ASC10-BB.TXT										
Newline Windows/DOS (<cr+lf>)</cr+lf>	•									
Conversion conditions										
Field separator TAB (*** <tab> ***)</tab>	-									
Divided data Vertical line output	•									
Separator None 💌										
Electric information (Gonio, Attachment, Monochro	mator)									
Measurement info (Scan avis Scan mode Slit VV	må Start Ston Star	n angle)								
Profile data (2theta,intensity) Inte	ensity format: Auto) 🔻								
Select contents to convert										

1) Open Rigaku folder (on desktop)

→ Bianry–ASCII Conversion.

- 2) Choose General ASCII Type.
- 3) Open the Input RAW File(s).
- 4) Choose the contents to be included in the TXT file.
- 5) Click Execute to finish.