

# **PANalytical X'Pert<sup>3</sup> Powder**

Version: February 12, 2023

#### SAFETY INSTRUCTIONS

Radiation exposures from **X-ray** diffraction units can be extremely hazardous. Any part of the body temporarily placed in the beam would receive enough radiation to cause serious radiation burns. X-rays cause the most damage to rapidly growing, undifferentiated cells. Thus, women those are pregnant or suspect that they may be pregnant, should take special care to avoid X-rays exposure.

The maximum permissible dose exposures in one year for radiation workers without limiting the activities are: Whole body 1 mSv ; Lens of the eye 15 mSv ; Skin  $(1 \text{ cm}^2)$  50 mSv. A cabinet X-ray system must contain sufficient shielding and be located so that exposure rates in unrestricted areas do not exceed 0.02 mSv/h and 1 mSv/year.

1 millisievert (mSv)= 100 millirem (mRem).

All unused ports must be securely closed to prevent accidental opening and the X-ray beam must be terminated within the enclosure at all times. All interlocks on the X-ray machine must be functional and in operation for X-ray production. X-ray diffraction machines must be operated in accordance with the basic X-ray safety guidelines in this laboratory instruction.

No individuals other than the operator(s) should be in the X-ray room while the X-ray diffractometer is prepared for operation. During operation all individuals must leave the X-ray room.

The **high voltage** power supply of X-ray machines can be particularly hazardous. Personnel must never tamper with high voltage equipment. Only properly trained personnel are permitted to install, repair, or modify high voltage equipment.



# **Technical info**

Instrument name	:	PANalytical X'Pert <sup>3</sup> Powder
X-ray source	:	Cu K <sub><math>\alpha</math></sub> , $\lambda$ =1.5418 Å
Operating range	:	$10-70$ $^{\mathrm{o}}2 heta$
Temperature	:	Room temperature
Humidity	:	Ambient
Sample amount	:	App. 10 mg



Consult lab supervisor if other sample holders,  ${}^{o}2\theta$ , temperature (temperature chamber available) or humidity (humidity chamber available) are required.

Measuring software data collector is always ON (if off then username and password is needed). When Measurement status showing "No program executing" means no measurement is currently running. Also as an indicator for no measurement running is the status of the MPPC generator is off. Umeå universitet, Institutionen för fysik, Linnaeus väg 24, 901 87 Umeå User manual, PANalytical X'Pert<sup>3</sup> Powder

## **1. STANDARD FLAT STAGE**

 Check the filter used. the X-ray source filter (5.0), FASS filter (2°) and FDS (1/4°) filter. You may exchange only the FDS filter to manipulate the detected intensity (reduce or raise the intensity). Do not touch other filters.



- 2. Check, the door of the XRD must be closed.
- 3. Turn ON cooling-water circulation switch(s), then turn ON the HT key.





4. Start software Data Collector, and login, if it was not already on the screen.

		Login	>
<u>U</u> ser:	Alexey		
<u>P</u> assword:	******		
		OK	Cancel

Username:  ${\bf Alexey}$  , Password:  ${\bf Parashut}$ 

5. Go to **Instrument** menu, then **Connect**. Do not change anything just press **OK** on the popup windows. A column with diffractometer setting commands will appear on the left side.

B De Dit See Mount Determini Joch Starfelinge Seine Beinge heinen Manimuurs Stater Brit D 🕲 🗃 🖨 X 🖓 Seenet 🔮 🕸 🖏 🖗	Data Collector (Alexey Riechikov)		- 6 -
N .			
Instrumen	t		
Connect			
🖉 Sti	ll no water flow	indicator	
			- • 12 Sector
ŭ	Connect	click	×
Configuration		_	OF
Name	Description	Date Owner	
MHC trans_Temperature	MHC trans_Temperatur	10/26/20 User-1	- Lancel
Stage HTK 16N	Anton Paar	5/9/2016 User-1	Help
Stage SAXS / WAXS Stage Spinner	Factory Settings Reflection / Transmission	5/9/2016 User-1 5/9/2016 User-1	
Stage TTK 450 Stage TTK 450 LNC	Anton Paar Anton Paar	5/9/2016 User-1 10/13/20 User-1	
L			_
Data Colleg	tor 2/2/2022 0.10.2	and click	
Data collec			
Assuming incident beam radius:240 mm	d divergence slit	и ок	
A Incident Anti-scatter slit. Assuming Fixed	d slit 2*.	Cancel	
Assuming diffracted beam radius: 240 m	nn Celerator with fixed anti-scatte	er slit	
1			
*			
<u>File Edit View M</u> easure Instrument	Tools User Settings Syst	em Settings System Mainten	ance <u>W</u> indow <u>H</u> elp
🗋 🖄 🖬 🍠 🗶 🛍 🖷 🔿 🖡	· · • • • •	<u>x</u> ?	
Stage Flat Sample	×		
Instrument Settings   Incident Beam Optics   Diffrac	ted Beam Optics	🖗 X-rau	
Positions		Generato	
Goniometer: PW/3050/60 (Theta/Theta)			is = Un
Sample stage: PW/3071/xx Bracket		- Iens	ion = U kV
Sample mode = Heflection		📃 🦾 🗛 Curre	ent = 0 mA
Generator: MPPC			
Internation = 0 kV			
	430 033 7310x)		
Status = Closed			

6. Double click on **Status**, click on **Generator on**, then press **Apply**, values 30 kV, 10 mA will load automatically. Wait until the values are confirmed, then press **OK**.

S Data Collector (Henry Keth) Die Die Vere Menore Internet Date Datation Schweitung Schweitung Schweitung Schweitung 그 그 과 4월 X 등 8 등 주 ₽ ← 4 © 35 등 7 T	1 Instrument Settings
Restriction	Position         Sample Stage         ∑ing         1           X-ray generator
🗄 🔧 X-ray	Breed
🚊 🖉 Generator: MPPC	Shutter open
Status = On	
Tension = 30 kV	
🔜 🗛 Current = 10 mA	2 2
Water flow: 4.3	OK Cancel Apply Help

Umeå universitet, Institutionen för fysik, Linnaeus väg 24, 901 87 Umeå User manual, PANalytical X'Pert<sup>3</sup> Powder

A test measurement before a series of sample measurements is recommended (optional). This is to test the XRD performance in general and test the detected intensity. By using the same FDS filter one must get approximately the same intensity as in previous series of measurements. This test measurement is performed by a Silicon disc which has a double peak located on  $2\theta=27^{\circ}$ , intensity about 20000 counts when  $1/4^{\circ}$  FDS filter is used. The Si disc, or powder sample holder disc are too fragile. Do not drop.

7. Press Unlock doors button to open the XRD doors. Push down the disc fixation clamp and load the Si disc on position. Then lock the doors.



8. Double click on **Tension**, then exchange values to **45 kV** and **40 mA**, then **Apply**. Wait until the values are confirmed, then press **OK**.

A Data Colecci (Alway Cicci Da Set Yee Beaux Johnner Dot Declaring Sciencizing Science/Pressure Beau Data Colecci (Alway Cicci Data Colecci (Alway Cicci	1 Instrument Settings
Reference to the second	Position         Sample Stage         ¥tyr.         45           x-ray generator
È	Shutter © Lire focus Shutter open
Water flow: 4.4	OK Cancel Apply Help

 Go to Measure menu then Program, select 0\_Si\_Standard\_Flat stage, press Open. A new window will pop up, there you have to give file name and destination for saving. Here you will also can see the 2θ measurment range 13.5-27. Finaly press OK. Measurement will start and stop automatically after about 2 minutes.

	Ever	uto Brogram	- = ×	
7	Exect	ute Program		
Measurement type		15	Start	
All	· ]	Program Name:	C:\PAN\0_Si_Standard_Flat stage.xrdmp	
Name 0_Si_Standard_Flat stage	Measurement Type Der Absolute scan	Type: Description:	Absolute scan	
1_Powder standard stag	ge Absolute scan	File Name:	2023 02 03 Si Standard Flat stage xrdml	
		Eolder:	C:\XRD Data\Alejandro	
		Sample		~
		ID: Name:		
		Prepared by:		
Open Browse	]	Position Diffractometer 2Theta (*): Omega Offset   Omega (*):	27,006         Phi [']:         △ (nm):           0,000         Cbj [']:         △ (nm):           13,503         Phi Offset [']:         △ (nm):	
Use the dro You can sor	p-down list to filter on measurem t the available measurements by c	Reflection Unit cell:	h <u>k</u> l:	
Use the dro You can sor	p-down list to filter on measurem t the available measurements by c	Reflection	h k l:	Help
Use the dro. You can sor	p-down list to filter on measurem t the available measurements by o eng. Sanctong Somethymese Both Both 영영 및 및 기기 (Prend 2000) (avec 184	Reflection	hk1: OK Cancel	Help
Use the dro You can sor You can sor	p-down list to filter on measurem the available measurements by c the available measurements by c the second secon	Reflection Unit cell: Data Cherce (Meer Di College Che	h <u>k</u> 1: OK Cancel	Help
Use the dro You can sort Vou can sort	p-down list to filter on measurem the available measurements by c	Reflection Unit cell: Data Calcular (Annuel Annuel	h js 1: DK Cancel	Нер
We the dia     Watch and a	p-down list to filter on measurement by c	Reflection Unit of the second	h h h h h	Help
	p-down list to filter on measurement by c	Referction	h h h h h h h h h h h h h h h h h h h	Hep

Test or performance measurement is done.

 For measurement on your sample; set tension to 30 kV and Current to 10 mA, then Apply and OK. Unlock the XRS doors. Take out the Si disc and replace by the disc for powders. Load powder and lock the doors.



11. Go to **Open** menu 
then select **1\_Powder** standard stage, press **Open**.

۲ <u>۵</u>		Open Progran	n	-	×
Measurement type All	•				
Name	Measurement Type	Description	Creation Date	Created by	Modificat
0_Si_Standard_Flat stage	Absolute scan		6/14/2017 6:48:21 PM	User-1	2/17/2022
1_Powder standard stage	Absolute scan		5/7/2018 1:58:51 PM	Alexey Klechikov	10/28/202
					•
Open Browse					Close
Use the drop- You can sort t	down list to filter on mea he available measuremer	isurement type. hts by clicking on th	e column headers.		



Here you can change the setting for **Start angel**, **End angle** and **Time per step**.

Prepare Absolute Scar	n [1_Powder standard stage]	
Configuration	Scan properties Repetition	Description
Stage Flat Sample 🗸 🗸 🗸	◯ Step	Comment
Scan Axis	<ul> <li>Continuous</li> </ul>	Settings
Gonio 🗸	Pre-set counts	o crangs
Other angle	Start angle (*): → 10.001	
Use actual angle at start	End angle (*):	
Offset (*):	Step size (*): 0.0065652	
	Time per step (s):	
	Net time per step (s): 58.395	
	Scan speed (*/s): 0.027937	
	Pre-set counts (counts): 10000	
	Number of steps: 9139	
	Total time (h:m:s): 00:37:52	

12. Input Start angel, End angle and Time per step then close this window

For best quity rsults; the recommended values are: 10, 70 and 130 s, this will give a total scan time of about 1 hour.

- 13. To measure do as given in steps 8 and 9. Exchange values to 45 kV and 40 mA, then Apply and OK. Go to Measurement menu then select program, select 1\_Powder standard stage, press Open, give file name and destination for saving, press OK. Measurement will start, do not disturb until it is finished.
- 14. To measure on a second sample, do as given in step 10 (Set tension to 30 kV and Current to 10 mA, then Apply and OK. Unlock the XRS doors. Take out the first sample and replace by the second sample, lock the doors). Run measurement as in step 13 again (Exchange values to 45 kV and 40 mA, then Apply and OK. Go to Measurement menu then select program, select 1\_Powder standard stage, press Open, give file name and destination for saving, press OK).
- 15. When all measurements are done? Double click on **Status**, unclick **Generator on**, then press **Apply**, values 0 kV, 0 mA will load automatically. Wait until the values are confirmed, then press **OK**.
- 16. Turn OFF the HT key, then turn OFF coolingwater circulation switch(s). Do not shut down software or PC.

ъ	Instrument Settings ×
Position Sample	Stage Xray
X-ray genera	tor
<u>T</u> ension (kV):	0 Generator on
<u>C</u> urrent (mA):	0
X-ray tube	
≚-ray tube:	Empyrean Cu LFF HR (9430 033 7310x) DK43 👻
	Breed
Shutter	Line focus     Point focus
	Shutter open
UK	Uancel Apply Help

### 2. DATA ANALYSIS

- 1. Start software HighScore , no login or password are required.
- 2. To save your data file as text file to be used in other data processing programs. Go to **File**  $\Rightarrow$  **Open**  $\Rightarrow$  choose your data file  $\Rightarrow$  **Open**. Then go to **File**  $\Rightarrow$  **Save As...**  $\Rightarrow$  Save. The data will be save as ASC file in the same destination as the original file.
- 3. To determin background, you have to loads the wanted data in HighScore software. Go to **Treatment**  $\Rightarrow$  **Determine Background...**  $\Rightarrow$  adjust the Bending factor until you satisfied  $\Rightarrow$  **Substract**  $\Rightarrow$  **Replace**  $\Rightarrow$  **File**  $\Rightarrow$  **Save As...**  $\Rightarrow$  Save.



4. Some other things can be done by this software, like **Treatment**  $\Rightarrow$  **Search Peaks** or **Analysis**  $\Rightarrow$  **Search** & **Match...**, these can be used to identify the peaks and extract some info about the peaks but the results are not accurate. Better to be done in other software based on your experience.