

COLUMBIA UNIVERSITY IN THE CITY OF NEW YORK COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

Agilent SuperNova SCXRD Standard Operating Procedure



These instructions are intended for reference only, and will *not* replace the thorough training required for proper system operation. Contact staff/superuser with questions or to report a system problem. Written by Daniel Paley.

Updated by Manju Rajeswaran (Oct, 2021)





IN THE CITY OF NEW YORK

COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

·	Г	LV0110		
1.	Enable the tool in BADGER	Window Equipment Actions Reservation Enable Disable Columi Shutdown > Clear Columi > Make Comment Qualify User UID Mag Browse Manual TCA A Gilent 8453 UV-vis. spectrop C Aglient 8453 UV-vis. Spectrop A Broker Dimension FastScan AF Malvern Nano-ZS zeta potenti. PANalytical XPert3 Powder XRL A Renishaw inVizor Woldiam Alpha-SE ellipsometr Phi XPS		
2.	If no window reading "CrysAlisPro (online)" is open, then start one from the desktop.	38.41 (online) CrysAlisPro		
3.	If the Cryostream is not running (display reads "Shutdown"), then press START and wait 10 seconds to initialize.			
4.	Click "Cryo." Click "Set," "Cool," enter 100 K, click OK. From room temperature, the Cryostream takes about 20 min to cool down.	START/STOP Shutter Closed Cu Cu Cu 4 x 4 Cu Cu 4 x 4 Cu Cu Cu Cu Cu Cu Cu Cu Cu Cu Cu Cu Cu		





IN THE CITY OF NEW YORK

COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

5.	Click "Xray." Click "Set kV,mA,X- ray". Choose Auto-ramp and click OK.	Firmware version: 2.2.4.109 - Final_Rev676_PCB2 Power [W] Temperature 0.60 1.9.7 C X-ray off X-ray off X-ray off Stop Set kV,mA,X-ray X-RAY Cu
6.	Prepare a microscope slide with 1 drop of oil. Do not spill. Turn on the light.	
7.	Transfer a few crystals to the oil.	



8.	Use a knife to separate a small, single-crystalline fragment.	
	The best crystals are 0.05 to 0.2 mm in size and regularly shaped.	
	The full scale bar is 10 mm (1x	0 10 20 30 40 50 50 70 80 90 100
	200m); 2.5 mm (4x 200m).	D T
	Push the crystal out of the oil	
	easily.	
9.	Pick a goniometer tip. Use your oil droplet to clean it off, then remove all excess oil by touching	
	the glass.	
	Mount the crystal neatly and without any excess oil. The	
	crystal should be easily visible so	
	you can center it on the diffractometer	
	diffactometer.	
10.	In CrysAlis, click "Start/Stop" and	CrysAlisPro: Experiment (1.13)
	"Mount."	Start new
		Start new (no pre-experiment)
		Resume all / pre-experiment; recalculate





IN THE CITY OF NEW YORK COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

11.	Open the cabinet with the large key. Using your right hand, place the magnetic crystal mount on the goniometer. Do not touch anything besides the goniometer. The X-ray sources, camera and Cryostream head are carefully aligned; the detector window is made of toxic beryllium.	
12.	In the mounting window, set phi=180.	Goniometer control Use mouse or keyboard shortcut O: -35.50 T: -8 Port: 134.00 P: 15:00 DD: 95.21 180 Arrow Up 90 Arrow Left Ctrl Arrow Right Page Up Ctrl Arrow Right Page Down
13.	Using the small key and your right hand, adjust the crystal up/down with the upper screw and left/right with the lower screw.	



IN THE CITY OF NEW YORK COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

14.	Rotate to phi=90 and adjust the crystal left/right again.				
15.	Rotate the phi axis to 2 positions separated by 90 degrees and ensure the crystal is well centered. The x-ray beam is half the diameter of the circle on the video display.				
16.	Exit the mounting window and close the cabinet. Click the small arrow next to "Screening." Set theta=-35, exposure time about 2-5 seconds. Click "Ok & Screen."	Screening options (1.0.0) Screening position Approx. theta -35.0 Max res.: 1.282 Phi offset to current position: 0 20 C 40 Exposure time Exposure time Pratures of screening Frames used 10 frame(s) Reset unit cell Hardware settings Generator Information Restore defaults Cancel OK & Screen OK			



17.	Evaluate the diffraction. The spots should be round and reasonably intense with sharp edges. If the diffraction is not good, you can screen more crystals.	
18.	If the diffractometer is still cooling down, wait for it to reach 100 K. Use the "Mount" window to ensure your crystal is still centered.	CAL OPEN ZAL SILENCE LOWER CM
19.	Use the pre-experiment slider to set an appropriate frame time. Click "Start Pre-Exp." This will collect 30 frames in 6 different positions. The wide angle frames are exposed for 5x the time you select.	Experiment - ac-2-17-2 Name: exp_1413 User=Dan, Detector=41.0mm, Res. = 1.100Ang, I/sig.=8.0, width=1.0deg, Movie, cryo off, Strategy: Complete data (default mode), Exposure: 1.0s 5.0s Exposure time: 1.0 s Start Pre-Exp. (3 min) Edit
20.	When the pre-experiment finishes, a strategy window will launch. Set the wide-angle exposure time for predicted individual I/sigma around 6 to 10 and the small-angle time for 1/5 of the wide angle. Set "Scan width" to 1.	Time prediction based on data to 0.800 Ang exp time individual (uncorrelated): I/sigma: I/s





IN THE CITY OF NEW YORK

COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

21.	Under Strategy Parameters, choose "Other," "hemisphere." Check that "Resolution" is set to 0.8. Use "Complete Data" mode with 100% completeness. Click "Calculate Strategy."	Strategy parameters Resolution Theta ZTheta 0.800 Laue group Other hemisphere Detector Distance 41.00 Strategy mode Complete data (default mode) limit 100.0 UCr limit Max 99.98 % Generates runs that reach completeness limit		
22.	To stop cryo after data collection, In strategy parameters - select Autochem/ Movie/Cryo/Red			
23.	In the experiment options window select "Auto cryo/hot device shutdown on experiment completion option". Click OK.	Experiment options (1.0.5) X Parameters Expected chemical formula: Import Attempt ActoChem AutoChem settings Record movie during dc. Step in deg: 6 Auto cryo/hot device shutdown on experiment completion 6 Auto cryo/hot device shutdown on experiment completion 25 # of frames Restart full auto analysis during data collection: 6 after 1/2 of data faiter 2/3 of data fait the end of DC Use external process RED during concurrent data reduction (G+Dit: only) Information Chemical formula is empty! 0K		





24.	Click "Start named experiment." Use "Browse" to find your data folder. Enter a name (preferably a notebook page #.) Enter the elements expected in your crystal. Click Start.	Special data collection (2.0.0) Special collection File name and path Name: dwp-5-100 Experiment: dwp-5-100 Path is ok Browse root folder Expected chemical formula: c74 o8 h100 Comment: test Information Path is ok!
25.	Ensure that the microscope light is off and the area is clean. If you leave samples, they will be thrown away.	
26.	Adjust your tool reservation in badger with your predicted finishing time.	
27.	When your experiment is finished, you can use Olex2 to solve your structure.	0 139 Ger / y 304 J11 151 151 1000 0.700 Co 0 rest raits











IN THE CITY OF NEW YORK

COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

31	Select "Standby" and "OK"	Read values Firmware version: 2.7.0.0 - Final_Rev786_PCB2		
51.	Sciect Standby and OK	Voltage [kV]	SUPERN set values	× Temperature
		50.00	Voltage (0-50 KV) 12.000	31.4
		Set values		
		Voltage [kV]	Current (0.0-0.8 mA) [0.050	Ramp On (50.0kV 0.8mA)
		Current [mA]	C X-ray ramp options	
		Emergency X-RAY OFF	Standhy	Set kV,mA,X-ray
		L Status: Device conne	Default	X-BAY Cu
		Faults	Autorramo	
		K/ EPP	Liser C	rature limit 43.0 deg. C
		mA EPR Tomporatura EPR		nerror O
		Safety shuffer EPR	Stand-by mode for inactive tube	tatus
		Safety shutter lamp ERR		e time 21657 h
		X-ray lamp ER-R		er of cycles 6//
			art lon	Settings
-				
32.	BADGER LOGOUT: Disable the			
	CCVDD in Dodgor	W	Equipment Actions	Reserva
	SCARD IN Badger.		Enable	
			Disable	
			Disable	_
		um	Shutdown	
			Report Problem	
		CLL	Make Comment	
		SMC	Make Comment	_
			Qualify User	UID Mag
			Provise Manual	тса
			browse Mariual	
		4	👍 Agilent 8453 UV-vis.	spectrop
			👍 Agilent SuperNova SC	CXRD
			A Bruker Dimension Fa	stScan AF