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)
1. Enable the tool in BADGER

2. If no window reading “CrysAlisPro (online)” is open, then start one from the desktop.

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.
5. Click “Xray.” Click “Set kV,mA,X-ray”. Choose Auto-ramp and click OK.

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 zoom); 2.5 mm (4x zoom).

Push the crystal out of the oil droplet so you can pick it up 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.

10. In CrysAlis, click “Start/Stop” and select “Start new.” Click “Mount.”

Start new
Start new (no pre-experiment)
Resume all / pre-experiment; recalculate
<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>11.</strong></td>
<td>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.</td>
</tr>
<tr>
<td><strong>12.</strong></td>
<td>In the mounting window, set ( \phi = 180 ).</td>
</tr>
<tr>
<td><strong>13.</strong></td>
<td>Using the small key and your right hand, adjust the crystal up/down with the upper screw and left/right with the lower screw.</td>
</tr>
</tbody>
</table>
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.”
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.

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.

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.
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.”

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.
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.

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.
28. When you are done, before you disable, please check to make sure that X-ray power is down to kV: 12.00 mA: 0.05 or it is ramping down. If not, please ramp down X-ray power manually.

29. Select “X-ray”

30. Click on “Set kV, mA, X-ray”.
31. Select “Standby” and “OK”

32. BADGER LOGOUT: Disable the SCXRD in Badger.