SAXS1 Operating Procedure for Single Capillary
Anton-Paar SAXSess mc²

Start-Up

1. Turn on the water chiller if not already on (located behind the side door).
2. Turn on the computer if not already on.
3. Turn the X-ray generator key to standby, wait a few seconds, then turn to on.
4. Turn on the TSC control box.
5. Turn on the Detector control box.
6. Turn on the Vacuum control unit.
7. Open SAXSquant software.
8. Select Measurement button in lower left corner.
9. Click on Manage Devices in left window (if not already open).
10. Right click on Generator: ID 3003 and click Set Online.
11. Double click on Generator: ID 3003 to open the generator menu.
12. Check the Perform PowerUp box.
13. Verify settings of 40kV and 50mA. Change to these setting if not correct.
14. Click Apply button.
15. Wait for generator to finish ramping to 40kV and 50mA. This may take up to
   20 minutes if generator has been powered off for a while. Time to power up
   will be displayed in the widow on the generator box (next to the key).
16. Uncheck the Perform PowerUp box and click the Apply button and close box.
17. Right click on Detector: 1D Diode Array and click Set Online.
18. Right click on Controller TSC Unit and click Set Online.

Alignment and Resolution Check

19. Pump down the chamber by pressing Start/Stop button on the Vacuum
    control unit. Make sure the vent valve is close. Turn clockwise to close
    (finger tight).
20. Wait until pressure is below 2 mBar to continue.
21. Open the X-Ray shutter by pressing F6 on generator control panel.
22. Perform Beam-Stop alignment verification:
   a. Double click on Detector: 1D Diode Array to open the detector menu.
   b. Set Exposure time to 5 seconds and No. of Frames to average to 100
   c. Enter Output Filename of beamstop_xxyy20zz (where xx=month,
      yy=date and zz=year)
   d. Select output directory of C:\My Documents/SAXS
      Data/User/Calibration
   e. Click Apply and then click display detector read-out button to view
      results.
f. The residual beam should be less than 1/10 size of primary beam. The position of residual beam defines the resolution (qmin).
g. Stop here if the results do not look good and notify SMIF staff.
h. When the data collection is finished, click the closed button.

23. Determine zero-angle position:
   a. Double click on Detector: 1D Diode Array to open the detector menu (insert graphic of this menu screen)
   b. Set Exposure time = 10 seconds and No. of Frames to average = 10
   c. Enter Output Filename of zero_xx_yy20zz (where xx=month, yy=date and zz=year).
   d. Select output directory of C:\My Documents/SAXS Data/User/Calibration
   e. Under Zero Angle Use, select “Find New Value” from the pull down menu. Note the current Zero Value.
   f. Set the search range from 140 to 170.
   g. Click Apply and wait for results to finish
   h. The new zero position will be displayed in the Current Zero Angle box. Record this value in the SAXS1 log book.
   i. Stop here if the new zero value is significantly different from the previous value and notify SMIF staff.
   j. When the data collection is finished, click the closed button.
   k. Press the F6 button on generator control panel to close the X-ray shutter.

Experiment Set Up
(See SAXSquant manual page 27-30)

24. Under Define Experiments in the navigation pane and click on “Create new definition file”.
25. To define a new experiment, click on + icon (add single experiment)
26. Enter experimental setting into “add new experiment” window (insert graphic that shows window).
   a. Enter output file name and select your directory in data folder (C:\My Documents/SAXS Data/User/ your folder).
   b. In the After Acquisition pull down menu, select “close shutter”.
   c. Enter Width of Detection Area as 8mm.
   d. Set exposure time and No of Frames to average (e.g., 60 seconds and 15 frames = 15 minute measurement)
   e. Check Use Despiking box
   f. Make sure that the Use Sampler box is unchecked.
If using the temperature control function to set temperature of sample during measurement follow the below steps:

1. Check *Use temperature control* box.
2. Fill in the *Set Temperature To* box.
3. Check *Wait for Temperature* box.

27. See attached protocol for filling and cleaning the capillaries. More information is also found in the SAXSess manual page 59-61 for handling of liquid samples. **Note: It is advisable to run the buffer blank first, the samples and then the buffer again.**

28. Vent the sample chamber by pressing *Start/Stop* button on the Vacuum control unit.
29. Open the vent value and remove the TSC cap.
30. Insert capillary, with the pin up, into the TCS sample-holder unit. Turn cap of TCS clockwise to close.
31. Close the vent valve. Pump down the chamber by pressing *Start/Stop* button on the Vacuum control unit.
32. Wait until pressure is below 2 mBar to continue.
33. Start automated measurement (*See See SAXSess manual page 35*)
    a. Select the experiment you created by clicking on the experiment name.
    b. Transfer the experiment to the measurement queue by clicking on the transfer icon.
    c. Start the measurement by clicking on the arrow icon.
34. When measurement is completed, vent the chamber and remove the quartz capillary.
35. Following attached protocol to flush out the sample/buffer and clean the capillary. Then load next sample or buffer.
36. Insert capillary, with the pin up, into the TCS sample-holder unit. Turn cap of TCS clockwise to close.
37. Close the vent valve. Pump down the chamber by pressing *Start/Stop* button on the Vacuum control unit.
38. Wait until pressure is below 2 mBar to continue.
39. Edit the filename in the experiment that was created above to indicate that this is a different sample.
40. Repeat steps 33-39 for all of the samples.
41. When the last sample/buffer has finished, vent the chamber and remove the quartz capillary.
42. Clean the capillary following attached protocol.
43. Close the vent valve. Pump down the chamber by pressing *Start/Stop* button on the Vacuum control unit.

**Shut Down procedures**
44. Right click on *Detector: 1D Diode Array* and click *Set Offline.*
45. Right click on *Controller TSC Unit* and click *Set Offline.*
46. Double click on *Generator: ID 3003* to open the generator menu.
47. Check the \textit{Perform PowerDown} box and click \textit{Apply}.
48. When it is finished uncheck the \textit{Perform PowerDown} box and click \textit{Close}.
49. Right click on \textit{Generator: ID 3003} and click \textit{Set Offline}.
50. Turn off the TSC control box.
51. Turn off the Detector control box.
52. Turn off the Vacuum control unit.
53. Close the SAXSquant software.
54. Turn the X-ray generator key to standby, wait a few seconds, then turn to off.
55. Turn chiller off and close the hall door.