NOVA 800 User Instruction Manual

Zaera Research Group

prepared on 8/21, updated on 9/1, 2023

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1. Introduction

Safety Signs

Wear protective gloves.

Wear safety goggles.

Use protective clothing.

Overview

Instrument	Analysis stations	Degas stations	Design features	Analysis results
NOVA 800	4	4	• Long sample cells and two-liter Dewar (40+ hours) enable the most detailed pore size analyses • Flexible software for complete surface area, pore size and pore volume analyses, and data reduction • Moderate sample throughput with 2 analysis Stations • Highest sample throughput with 4 analysis stations	• **Surface area**: BET, NSA, STSA, Langmuir • **Pore size**: BJH, DFT • **Pore volume**

Abbreviations: *BET - Brunauer-Emmett-Teller; NSA - Nitrogen Surface Area, STSA: Statistical Thickness Surface Area., BJH - Barrett, Joyner & Halenda; DFT - Density Functional Theory.*

Summary & Keynotes

- All users should **make reservations ahead, with the FACES system**. You should consider drying time for sample cells after washing.

- Maximum heating temperature for degassing: 425 ℃.

(Where degassing temp. ≥ 350 ℃, **quartz sample cell** should be used instead of standard borosilicate sample cell)

- If **any types of issues** happen, please let the manager know about it.

(Issues including broken sample cells/rods, unexpected abort, problem with pump etc.)

- If you notice **LN_2** is running out after your analysis, please let the manager know about it for the next user.

2 & 3. Sample Loading & Degassing

The recommended amount of powder samples for the analysis can be found as follows:

(1) Total surface area (m^2) in the cell: 2 – 5 m^2

e.g., Sample A has 100 m^2/g as BET surface area.

BET surface area (m^2/g)	Loaded mass (g)	Toal surface area in the cell (m^2)	Result
100	0.005	100 * 0.005 = 0.5	<< 2, not promising
	0.1	100 * 0.1 = 10	>> 5, not promising
	0.05	100 * 0.05 = 5	Just right

(2) Minimum of sample mass in the cell: 25 mg (after degassing)

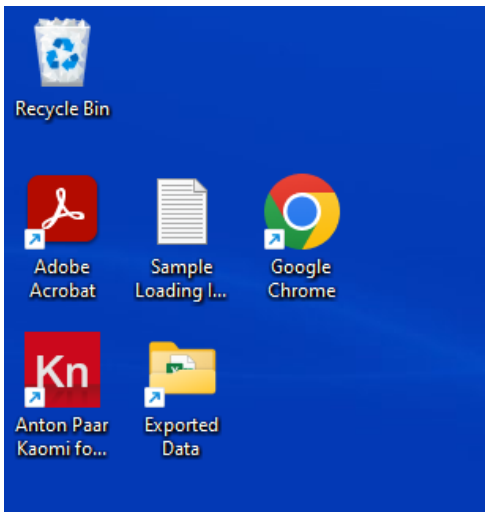
You should consider and expect sample loss during (a) transferring after weighing out and (b) degassing.

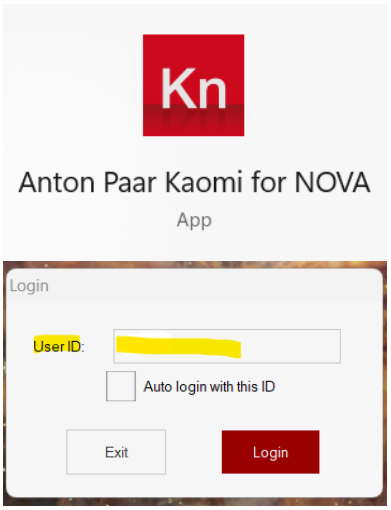
a. Take out clean cell(s) from the oven and cool down to room temperature.

a.1. Two types of sample cells

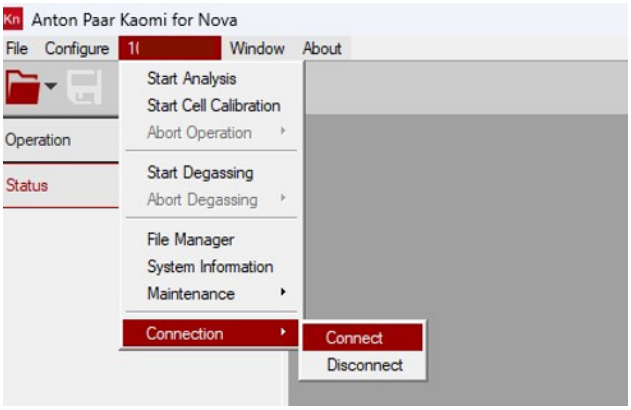
Type B Long cell, 9 mm large bulb	Type B Long cell, 9 mm no bulb
	

b. Weigh out the clean cell(s). Repeat this step until reasonable reproducibility is achieved. Note those numbers in your own lab notebook.
c. Weigh out the sample (~ 30 mg, *reference recommended amount above this page*). Add this to the weighed clean cell. Follow the transferring method from BET manual video on lab website.
d. Weigh out the cell(s) containing the powder sample. Repeat this step until reasonable reproducibility is achieved. Note those numbers in your own lab notebook.

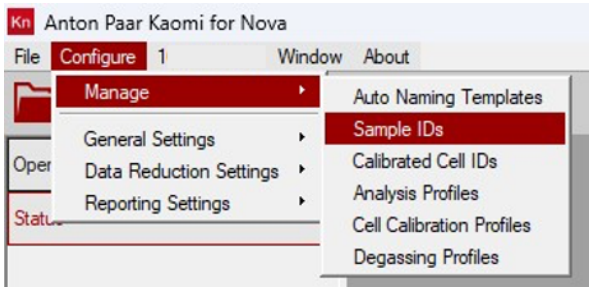


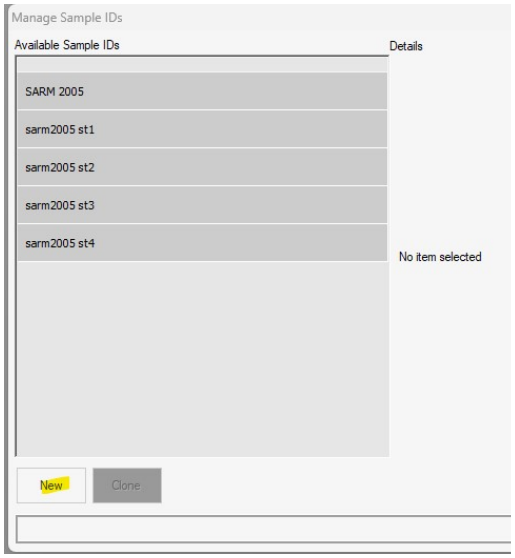


e. Run ‘Anton Paar Kaomi for NOVA’ program, insert your UCR NetID at ‘User ID’ and click Login button.

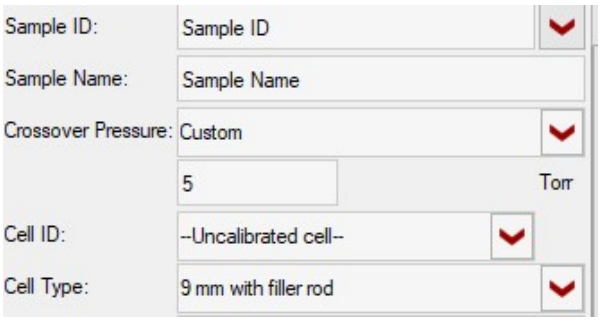


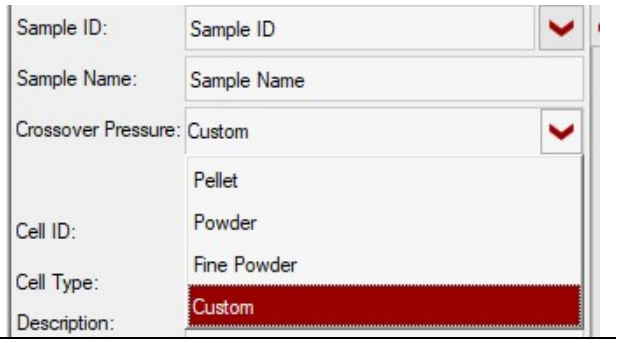
f. Connect with instrument. (S/N (1*********) > Connection > Connect)





g. Make **new sample IDs** via Kaomi program. (Configure > Manage > Sample IDs > New)

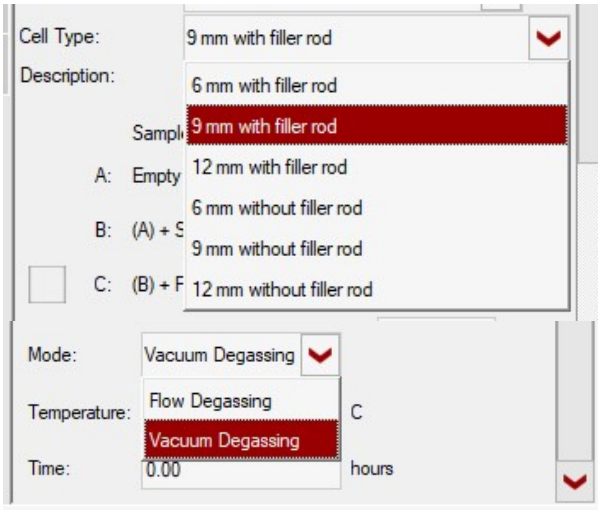




Sample ID & Name; whichever you can remember and distinguish between samples after analysis

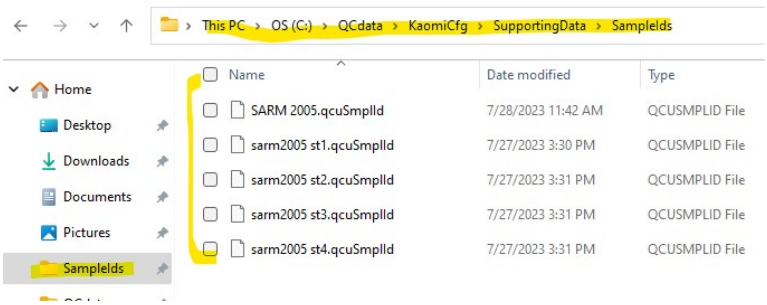
Crossover Pressure;

Powder	Fine Powder	Custom (Recommended)
50 Torr	20 Torr	5 Torr



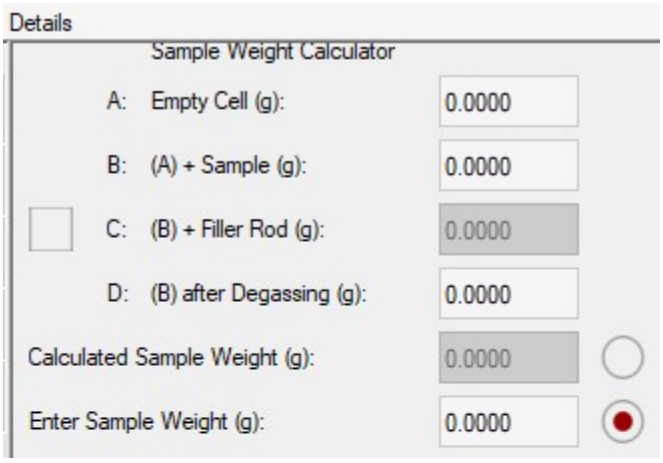
Cell type: 9 mm with filler rod (fixed)
(only available ones in our lab)

Mode: Vacuum Degassing (fixed)
(Inform operator when 'Flow Degassing' is required for your samples)



You can delete your Sample IDs at location of SampleIDs folder (C: > QCdata>KaomiCfg>SupportingData>SampleIds).

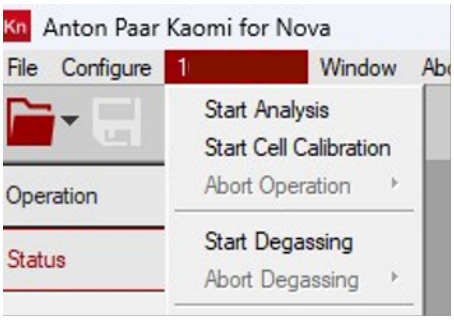
c.1. Insert the values for each part as follows:



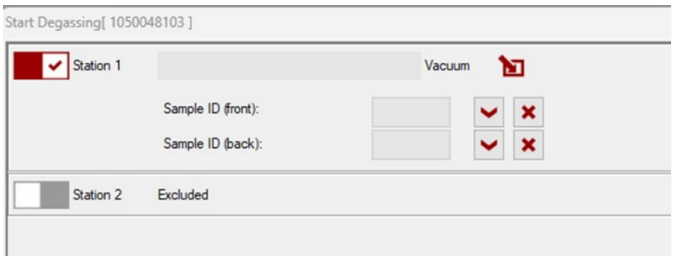
A. Empty Cell (g)	Measure the weight of the empty cell w/o the rod from **step b**
B. (A) + sample (g)	Measure the weight of (empty cell + powder sample) w/o the rod from **step d**
Check 'Calculated Sample Weight (g)'	

Do not use the rod during degassing step to avoid sample loss.

h. Load the sample cell on the degas station (②).

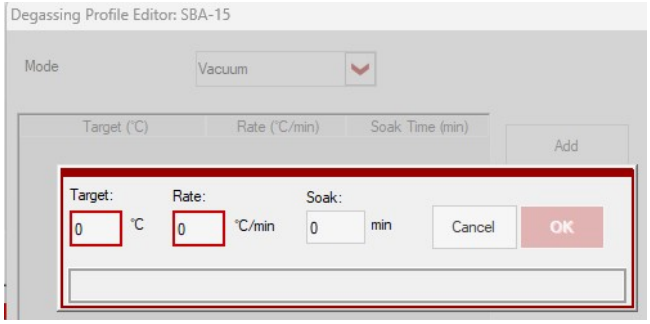


i. Start Degassing (S/N > Start Degassing).

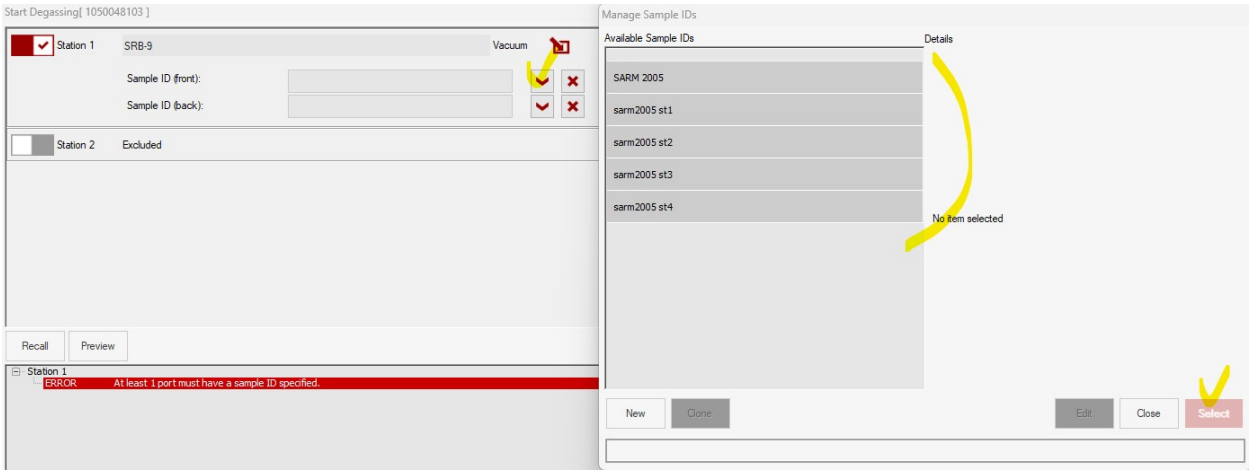


e.1. Enable Station 1 (or 2) by selecting its on/off control () (make sure the check box is checked).

e.2. Select the degas profile. If needed, click ‘New’ and make new degassing method.



Max. heating temp. : 425 ℃

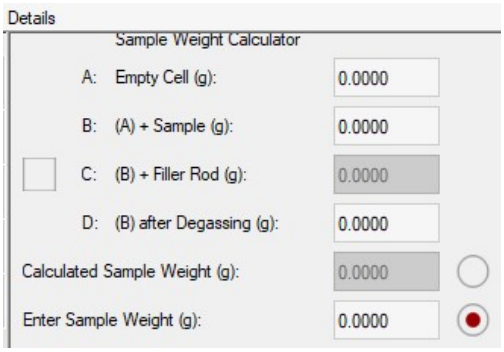


e.4. Open the Sample ID drop-dwon list.

e.5. Click ‘Start’ to begin the degassing process. Keep track of progress by selecting Status from the Kaomi for Nova Control Center/ Sidebar, or the Log View icon on the touchscreen.

e.6. Always use **Software method** to run degassing. Do not use Touchscreen method.

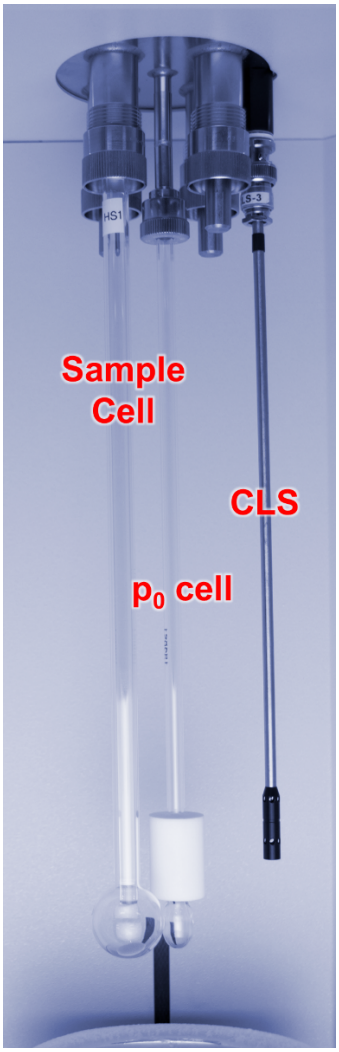
j. Stay near to the instrument and wait until the vacuum is switched from fine vacuum to the rough vacuum. If you notice your powder is being sucked during this step, contact to manager immediately.
k. After degassing is complete, the sample cell is cooled down, and backfill is complete, remove the sample cell from the degassing station.
l. Re-weigh the sample cell to determine the net weight for the analysis.



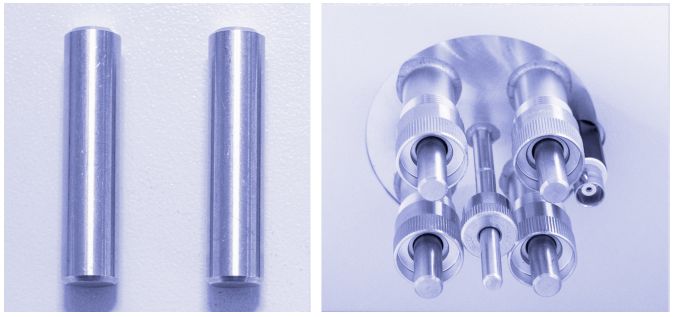
D. (B) after degassing	Measure the weight of (empty cell + powder sample) w/o the rod after degassing

m. Update the Sample ID.

4 & 5. Sample Cell Setting and Start Analysis

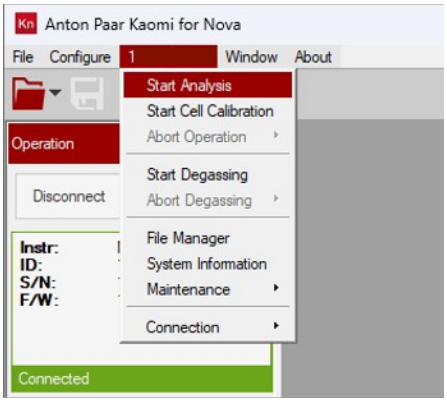


- Fill the Dewar with LN_2 to the level indicator and allow the cryogen to settle. If needed, add more cryogen to ensure it is at the level indicator.
- To set up and run an analysis, ensure the **p_0 cell** and **CLS** are installed and attach the sample cell you have been working with to Analysis Station 1 as shown in left hand side figure.
- Inform the manager, if there is any issue with compartments (e.g., O-ring and/or fittings)
- Ensure all unused stations are sealed with dowel pins.

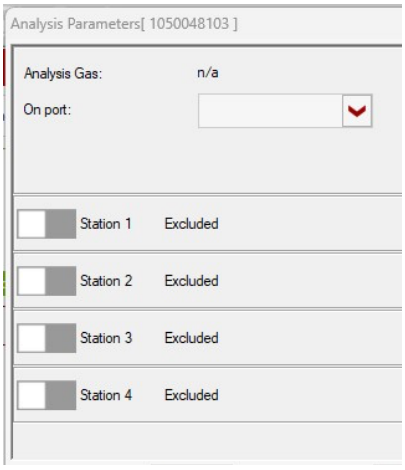


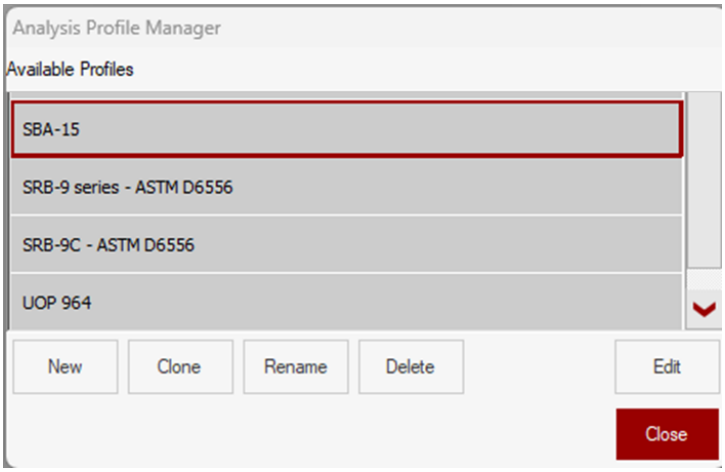
(*left: image of 'dowel pins', right: as inserted in analysis stations*)

- Note: Make sure to use only one O-ring when installing the sample cell into the fitting (using two or more O-rings may cause erroneous results).



a. Start Analysis (S/N > Start Analysis).





b. Select on using Station and corresponding analysis profile (e.g., [SBA-15]).

b.1. If needed, make a new profile for your own sample.

c. Fill in the remaining fields, such as:

c.1. On port: set to nitrogen.

c.2. Sample ID as same as used for degassing

c.3. File name for the results file (your choice if not using the auto-naming templates feature)

d. Click Start to begin the analysis process.
e. Keep track of progress by selecting Status from the Kaomi for Nova Control Center/Sidebar (program from desktop).

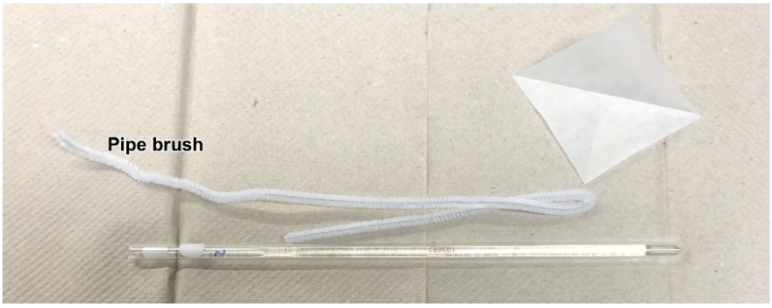
※ Because of the limited LN_2 volume in the Dewar, you should consider how long the analysis will take your sample. (It can stay up to 40 h once it is refilled.)

e.g., *Student A* planned to analyze 4 samples, but it turns out that 2 samples would already take 30 hours. → Analyze 2 samples first (Station 1 and 2 are on first), refill LN_2 after two samples and analyze the rest of the samples.

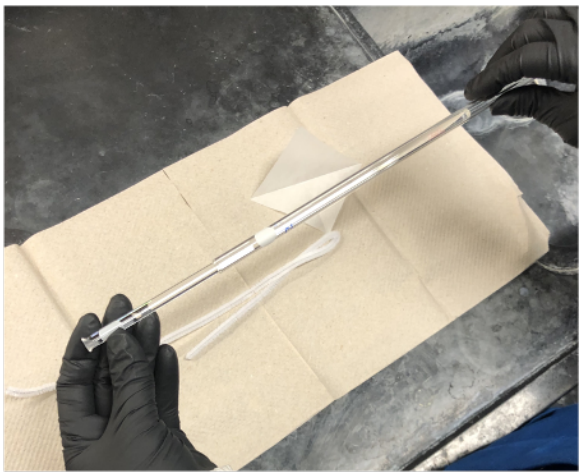
6. After Analysis

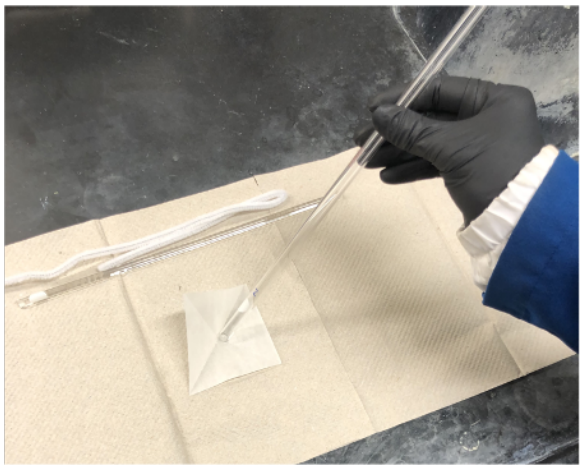
a. Remove the sample cells from the Instrument. Make sure all unused stations are sealed with dowel pins.
b. Clean up the used cell.

b.1. Prepare following tools: pipe brush and container for collected powder (vial or waste)

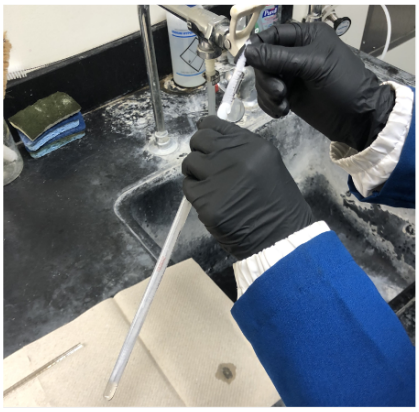


b.2. Separate the rod carefully and collect used powder samples.

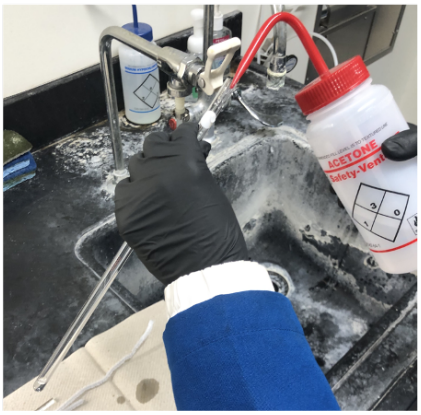


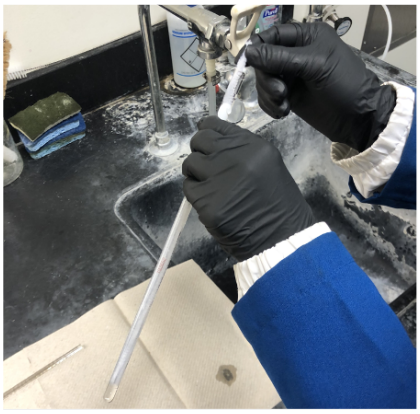


b.3. Clean up the cell with deionized water by using a pipe brush. Repeat 3-4 times if needed.



b.4. Clean up the cell with acetone by using a pipe brush. Repeat 1-2 times if needed.

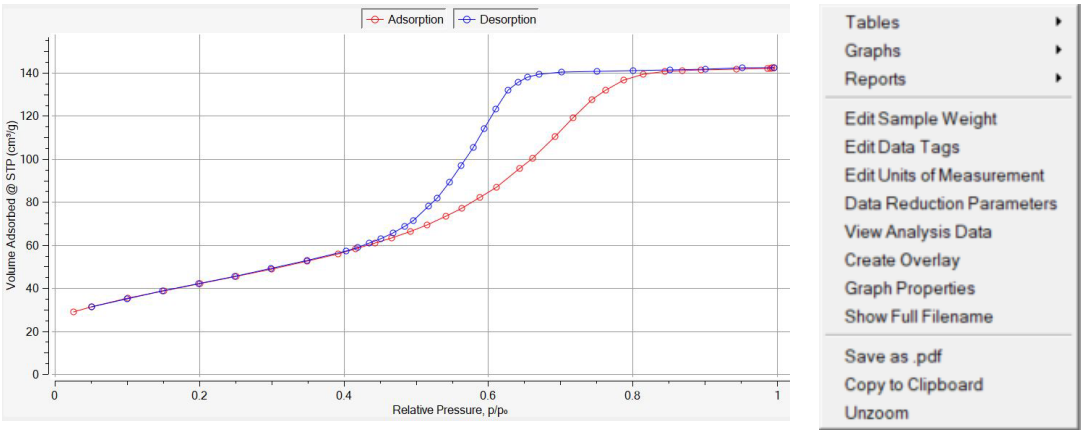




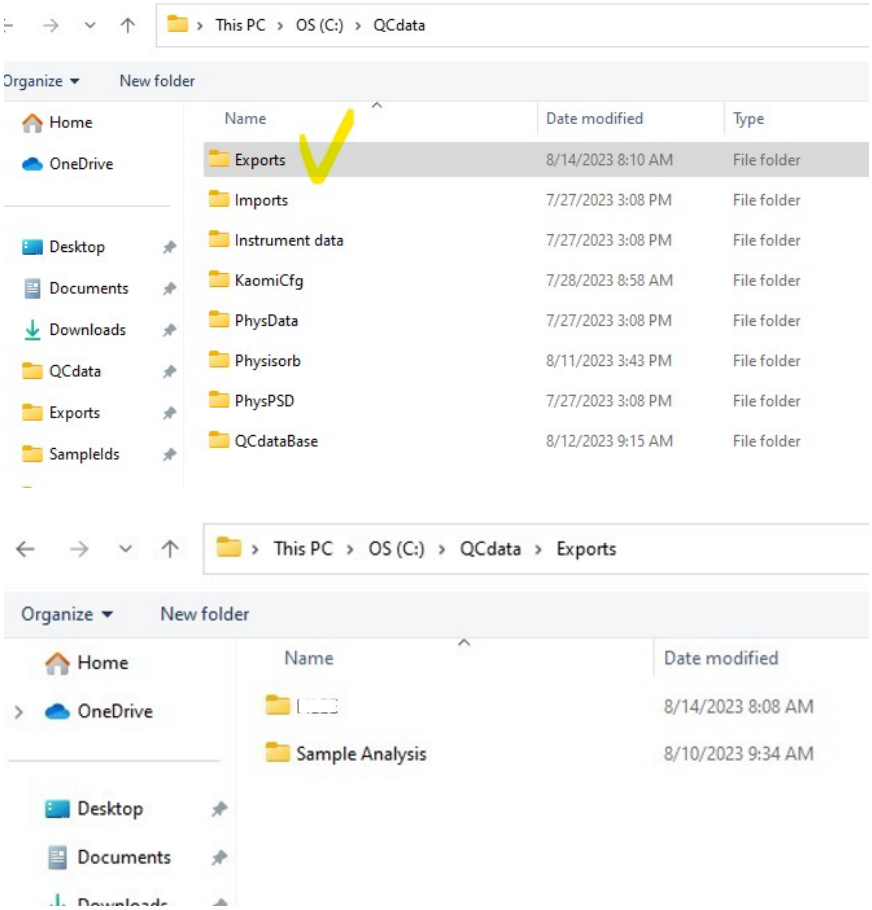
b.5. Clean up the rod in a such way. Wipe down with Kimwipes.

b.6. Dry the cleaned ones inside of the oven upside-down.

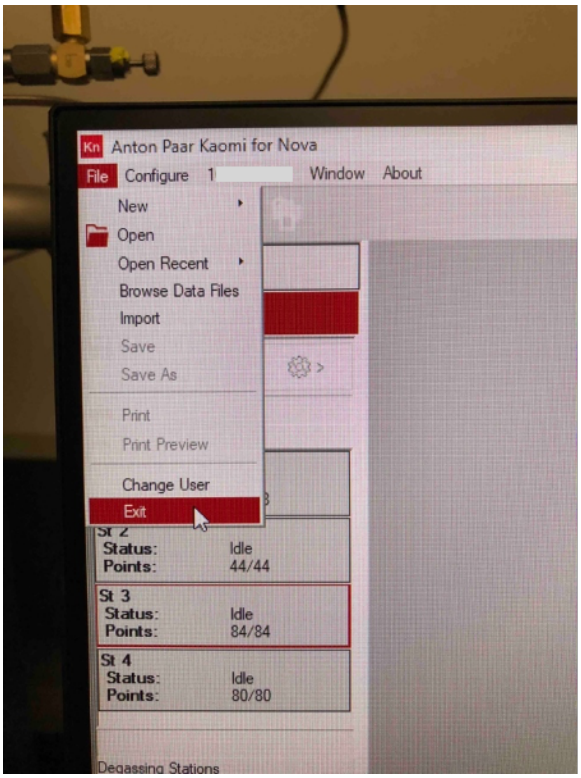
c. Inform the manager, if any types of replacement is needed.



d. Right-click the displayed figure displayed in Kaomi software for reporting options.
e. Save your data in your designated folder only. If you don't have your own folder, please make one in This PC > OS (C:) > QCdata > [your name] as you see below,



f. Close the results windows
g. Click the File menu and select Exit to finish the program.
h. Clean the bench for the next user.



Step by Step Guide

1. Take a clean cell from the oven
2. Cool the cell down to room temperature
3. Weigh the empty cell
4. Weigh your sample (~30 mg)
5. Fill the cell with your sample
6. Weigh the sample cell containing the sample
7. Run [Anton Paar Kaomi for NOVA]
8. Type your UCR NetID in [User ID]
9. Click [Login] button
10. Select [1*********] > [Connection] > [Connect]
11. Select [Configure] > [Manage] > [Sample IDs] > [New]
12. Fill Sample ID and Sample Name
13. Select [Custom or Fine Powder] on Crossover Pressure
14. Choose [9 mm with filler rod]
15. Click [Vacuum Degassing] on Mode
16. Type the weight of empty cell (Line A) (weight from step 3)
17. Type the weight of the sample cell (Line B) (weight from step 5)
18. Check [Calculated Sample Weight]
19. Save the Sample ID
20. Load the sample cell onto the degas station
21. Select [1*********] > [Start Degassing]
22. Enable [Station 1]
23. Select the degas profile, [SBA-15]
24. Open the Sample ID drop-down list
25. Choose the right Sample ID
26. Click [Select] button
27. Click [Start] button
28. Stay nearby until the ramping starts
29. Wait until the sample cell is fully cooled down after degassing is complete
30. Take a clean rod from the oven and cool down to room temperature
31. Remove the sample cell from the degassing station
32. Re-weigh the sample cell without the rod
33. Ensure P0 cell and CLS are installed
34. Put a clean rod into the sample cell
35. Attach the sample cell to the analysis station (1 to 4)
36. Ensure any unused station is sealed with dowel pins
37. Fill the Dewar (use cryogenic gloves) with LN2 to the level indicator
38. Allow LN2 to be settled.

39. Place the LN2 Dewar on to the lift drive
40. Select [1*********] > [Start Analysis]
41. Set Nitrogen to on port
42. Check all the stations you attached your sample cells
43. Select SBA-15 analysis profile
44. Type the weight of the degassed sample cell (Line D)
45. Type the net sample weight in Sample Weight entry field
46. Click [Save] button
47. Click [Select] button
48. Click [Start] button
49. Wait until all the analysis is done
50. Mouse right-click on the result figure for reporting options
51. Select [Graphs] for image data
52. Select [Tables] for numeric data
53. Close the results window
54. Click [File] > [Exit]
55. Send your results by using (1) R'Mail or (2) Google Drive (Do not use USB drives)
56. Take LN2 Dewar out of the analysis station
57. Remove the sample cells from the analysis station
58. Put the dowel pins back to the ports
59. Have a pipe brush and a vial
60. Separate the rods from the sample cells
61. Collect your powder samples from the cells and place them in the vials
62. Rinse the cells and rods with deionized water by using a pipe brush (3-4 times)
63. Clean up the cells and rods with acetone by using a pipe brush (1-2 times)
64. Put the cleaned cells and rods (upside-down) back into the oven