NOVA 800 User Instruction Manual



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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 °C. •

(Where degassing temp. ≥ 350 °C, **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 LN₂ 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 (m²) in the cell: $2 - 5 \text{ m}^2$

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

BET surface area (m²/g)	Loaded mass (g)	Toal surface area in the cell (m ²)	Result
	0.005	100 * 0.005 = 0.5	<< 2, not promising
100	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. Run 'Anton Paar Kaomi for NOVA' program, insert your UCR NetID at 'User ID' and click Login button.



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



No item selected

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

Details			Details			
Sample ID: Sample Name:			Sample ID: Sample Name:		~	^
Crossover Pressure:	Fine Powder	~	Crossover Pressure:	Pellet	~	
	20	Torr		Pellet		
1			Cell ID:	Powder		
			Cell Type:	Fine Powder		
			Description:	Custom		
ample ID & N	ame; whichever y	ou can remember	Crossover Pre	essure;		
nd distinauish	between samples	s after analysis		Fine		

Powder

50 Torr

6 mm with filler rod ppli 9 mm with filler rod ty 12 mm with filler rod 6 mm without filler rod 9 mm without filler rod		
by 12 mm with filler rod 12 mm with filler rod 6 mm without filler rod 9 mm without filler rod		
hty 12 mm with filler rod 6 mm without filler rod 9 mm without filler rod		
6 mm without filler rod 9 mm without filler rod		
6 mm without filler rod 9 mm without filler rod 12 mm without filler rod		
12 mm without filler rod		
ow Degassing C		
acuum Degassing		
	+ F 12 mm without filler rod icuum Degassing ow Degassing acuum Degassing 00 c hours	

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

Powder

(Recommended)

20 Torr

Custom

e.g., 10 Torr

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

Home		Name	Date modified	Туре
Deckton		SARM 2005.qcuSmplld	7/28/2023 11:42 AM	QCUSMPLID File
Desktop	*	🔲 🗋 sarm2005 st1.qcuSmplld	7/27/2023 3:30 PM	QCUSMPLID File
Uownloads	*	sarm2005 st2.qcuSmplld	7/27/2023 3:31 PM	QCUSMPLID File
Documents	*	sarm2005 st3.qcuSmplld	7/27/2023 3:31 PM	QCUSMPLID File
Pictures	*	Sarm2005 st4.gcuSmplid	7/27/2023 3:31 PM	OCUSMPLID File
SampleIds	*			

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

c.1. Weigh out and insert the values for each part as follows:

S	ample Weight Calculator	1.121.011.011		A. Empty	Measure the weight of
A: E	mpty Cell (g):	0.0000		Cell (g)	
B: (A	() + Sample (g):	0.0000			
C: (E	3) + Filler Rod (g):	0.0000		B. (A) + sample (g)	(empty cell + powder
D: (E)) after Degassing (g):	0.0000			sample) w/o the roo
Calculated Sar	nple Weight (g):	0.0000	\circ	D (P) offer	Measure the weight of
Enter Sample Weight (g):		0.0000		degassing	sample) w/o the rod after degassing

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

c.2. Reference the video on our lab website for sample transfer from weighing paper to the sample cells.

c.3. Two types of sample cells





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



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

Start Degassing[1050048103]		Available Profiles
Station 1	Vacuum 🛅	ASTM B922
Sample ID (fi Sample ID (b	ront):	ASTM D 1993
Station 2 Excluded		New Clone Rename Delete Edit

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.

Degassing Profile Editor: SBA-15	
Mode Vacuum 🖌	
Target ('C) Rate ('C/min) Soak Time (min) Add	Max. heating temp. : 425 °C
Target: Rate: Soak: 0 "C 0 "C/min 0 min Cancel OK	
Start Degassing[1050048103]	Manage Sample IDs
Station 1 SRB-9 Vacuum Sample ID (front): Sample ID (back):	Available Sample IDs Details SARIM 2005 serm 2005 st1 sarm 2005 st2 sarm 2005 st3 serm 2005 st4 No hem selected
Recall Preview ESTATION 1 FROR At least 1 port must have a sample ID specified.	New Cone Edit Close Select

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, if you are not familiar with Touchscreen method.

- f. After degassing is complete, the sample cell is cooled down, and backfill is complete, remove the sample cell from the degassing station.
- g. Re-weigh the sample cell to determine the net weight for the analysis.

Details Sample Weight Calculator A: Empty Cell (g): B: (A) + Sample (g): C: (B) + Filler Rod (g):	0.0000 0.0000 0.0000	D. (B) after degassing	Measure the weight of (empty cell + powder sample) w/o the rod after
Calculated Sample Weight (g):	0.0000		
h. Update the Samp	le ID.		

4 & 5. Sample Cell Setting and Start Analysis



- Fill the Dewar with LN₂ 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).

Analysis Parameters[1050048103]	Analysis Paramet	ers[1050048103	Analysis Profile Manager	
Analysis Gas:	n/a	Analysis Gas:	n/a	Available Profiles	
On port:		On port:		N2 STSA	^
				N2 full Isotherm with micropore BET points(2)	
				N2 full Isotherm with micropore BET points	
Station 1	Excluded	Station	1 Excluded	N2 full isotherm	
Station 2	Excluded	Station	2 Excluded	SARM 2001	
Station 3	Excluded	Station	3 Excluded	SARM 2005	
Station 4	Excluded	Station	4 Excluded	SARM 2007	
				SARM 2009	
				SBA-15 88 Pts (Weng)	

- b. Select on using Station and corresponding analysis profile.
- 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. Net sample weight (calculated earlier) in the Sample Weight entry field.
- c.4. 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, or the Log View icon on the touchscreen.

***** 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. \rightarrow Analyze 2 samples first (Station 1 and 2 are on first), refill LN₂ 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,

Home	Name^	Date modified	Туре
OneDrive	= Exports	8/14/2023 8:10 AM	File folder
	imports	7/27/2023 3:08 PM	File folder
Desktop 📌	📒 Instrument data	7/27/2023 3:08 PM	File folder
Documents 🖈	📒 KaomiCfg	7/28/2023 8:58 AM	File folder
Downloads 🖈	늘 PhysData	7/27/2023 3:08 PM	File folder
QCdata	늘 Physisorb	8/11/2023 3:43 PM	File folder
Exports 🖈	PhysPSD	7/27/2023 3:08 PM	File folder
Samplelds 🔹 🖈	CCdataBase	8/12/2023 9:15 AM	File folder
- → ∨ ↑ Drganize ▼ Nev	This PC > OS (C:) > QC v folder	data > Exports	
- → ∨ ↑ Organize ▼ Nev	This PC > OS (C:) > QC v folder Name	data > Exports Date	modified
- → ✓ ↑ Drganize ▼ Nev ↑ Home ● OneDrive	v folder	data > Exports Date 8/14,	modified /2023 8:08 AM
- → × ↑)rganize ▼ Nev ☆ Home ● OneDrive	This PC > OS (C:) > QC v folder Name Sample Analysis	data > Exports Date 8/14, 8/10,	modified /2023 8:08 AM /2023 9:34 AM
- → ✓ ↑ Organize ▼ Nev	This PC > OS (C:) > QC v folder Name Sample Analysis	data > Exports Date 8/14, 8/10,	modified /2023 8:08 AM /2023 9:34 AM
- → ∨ ↑ Drganize ▼ Nev ↑ Home ● OneDrive ■ Desktop ■ Documents	This PC > OS (C:) > QC v folder Sample Analysis	data > Exports Date 8/14, 8/10,	modified /2023 8:08 AM /2023 9:34 AM

- 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.

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Ka Anton Paar K	aomi for N	lova		
File Configure	1	Window	About	
New Dopen Open Recent Browse Data Import Save Save Save As Print Print Preview	Files	©>		
Change User	r 3			
St z Status: Points:	Idle 44/44			
St 3 Status: Points:	Idle 84/84			
St 4 Status: Points:	Idle 80/80			
Denassing Station	D.º			

Step by Step Guide

- 1. Take a clean cell from the oven
- 2. Cool the cell down to room temperature
- 3. Run [Anton Paar Kaomi for NOVA]
- 4. Type your UCR NetID in [User ID]
- 5. Click [Login] button
- 6. Select [1********] > [Connection] > [Connect]
- 7. Select [Configure] > [Manage] > [Sample IDs] > [New]
- 8. Fill Sample ID and Sample Name
- 9. Select [Fine Powder] on Crossover Pressure
- 10. Choose [9 mm with filler rod]
- 11. Click [Vacuum Degassing] on Mode
- 12. Weigh the empty cell
- 13. Type the weight of empty cell (Line A)
- 14. Fill the cell with your sample
- 15. Weigh the sample cell
- 16. Type the weight of the sample cell (Line B)
- 17. Load the sample cell onto the degas station
- 18. Select [1*******] > [Start Degassing]
- 19. Enable [Station 1]
- 20. Select the degas profile, [SBA-15]
- 21. Open the Sample ID drop-down list
- 22. Choose the right Sample ID
- 23. Click [Select] button
- 24. Click [Start] button
- 25. Wait until the sample cell is fully cooled down after degassing is complete
- 26. Remove the sample cell from the degassing station
- 27. Re-weigh the sample cell
- 28. Type the weight of the degassed sample cell (Line D)
- 29. Fill the Dewar (use cryogenic gloves) with LN2 to the level indicator
- 30. Allow LN2 to be settled.
- 31. Ensure P0 cell and CLS are installed
- 32. Put a clean rod into the sample cell
- 33. Attach the sample cell to the analysis station (1 to 4)
- 34. Ensure any unused station is sealed with dowel pins
- 35. Select [1*******] > [Start Analysis]
- 36. Check all the stations you attached your sample cells
- 37. Select SBA-15 analysis profile
- 38. Set Nitrogen to on port

- 39. Type the net sample weight in Sample Weight entry field
- 40. Name your data file if not using the auto-naming templates feature
- 41. Click [Start] button
- 42. Wait until all the analysis is done
- 43. Mouse right-click on the result figure for reporting options
- 44. Select [Tables] for numeric data
- 45. Select [Graphs] for image data
- 46. Close the results window
- 47. Click [File] > [Exit]
- 48. Remove the sample cells from the analysis station
- 49. Put the dowel pins back to the ports
- 50. Have a pipe brush and a vial
- 51. Separate the rods from the sample cells
- 52. Collect your powder samples from the cells and place them in the vials
- 53. Rinse the cells and rods with deionized water by using a pipe brush (3-4 times)
- 54. Clean up the cells and rods with acetone by using a pipe brush (1-2 times)
- 55. Wipe the cells and rods with Kimwipes
- 56. Put the cleaned cells and rods (upside-down) back into the oven