Micromeritics BET Surface Area and Porosity Analyzer

Instrument Information and Generalized Standard Operating Procedure

Micromeritics ASAP 2020 Surface Area & Porosity Analyzer Location: W108 Plant Science
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0. Obligatory things to consider PRIOR to beginning analysis

The ASAP 2020 analyzer is equipped with two independent vacuum systems — one for sample preparation and one for sample analysis. Having two separate systems, as well as separate preparation ports, allows sample preparation and sample analysis to occur concurrently without interruption. Inline cold traps are located between the vacuum pump and the manifold in both the analysis and the degas systems.The sample saturation pressure (Psat) tube is located next to the sample analysis port. Gas inlet ports and cable connections are located conveniently on the side panel of the analyzer for easy access.

The ASAP 2020 is equipped with an elevator that raises and lowers the analysis bath fluid Dewar automatically. A removable shield to enclose the Dewar is also included for safety purposes.

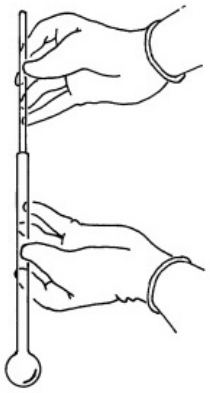


Figure 1 (a) *Photo of Micromeritics Instrument capable of analyzing surface and porosity characteristics of powders or solids. Instrument has two degassing units and isothermal jackets on the left, a cyrotrap dewar for condensing excess moisture in the center (preventing moisture from reaching the $3,000 turbo vacuum pumps) and the elevating analysis dewar on the right for maintaining isothermal conditions when determining specific surface areas (SSA).* ***(b)*** *A sketch of the glass sampling tube with glass rod to fill excess void space.*

Energy Conservation Practices: Turn on the ASAP 2020 instrument first (on/off switch on the right side of instrument) and make sure the two preliminary vacuum pump are plugged in BEFORE turning on the nearby computer. Otherwise ASAP software won't recognize network correctly with the instrument. The pump plugs exit the back from inside the instrument panel and the pumps themselves can not be seen unless you open the front panel of the instrument. It is ok to leave the instrument and vacuum pumps on during days or weeks of analysis. However, please turn off the instrument and computer, and unplug the two preliminary vacuums during long periods without use. (see steps 1, 2 & 29 in procedures)

The **ASAP 2020 Operator's Manual is provided as a pdf file on the desktop** if further reference or trouble shooting is needed.

Table 1 *File types that can be found within the "param folder" (parameters) of the ASAP 2020 data folder*

File tag ending	Description
.SMP	Raw sample files will all method parameters and measured sample data only readable by ASAP software
.ADP	adsorptive property files of phsyisorb gas for method selection on adsorptive properties tab
.ANC	analysis method files for choosing BET SSA only, Full Ads/Des Isotherms, or other specific processing methods
.REP	report files of tabulated and plotted surface characteristic, only read by ASAP software
.pdf	Same as above .rep file, yet able to be read by any pdf reader software
.xls	transferring comma delimited tabulated data for use on spreadsheet software such as MS Excel

How much solid sample is needed?

- The bulb shaped, glass sample tube holder can hold volumes from 1cm^3 to 20cm^3 of your solid sample to be characterized, yet mass of sample needed depends upon material density and expected SSA (see literature for an estimate).
  Typically 0.5g – 1.0g for samples of high SSA > 100m^2/g
  And expect 1.0g – 8.0g for SSA < 100m^2/g
- The ASAP 2020 Operator's Manual recommends 40m^2 to 120m^2 of total surface area per sample for best surface area analysis results to be achieved, although detection down to 10m^2 total surface area is attainable as well. Any surface area above 120cm^2 total unnecessarily extends analysis time. This means if your expected specific surface area is only 8m^2/g be sure to include at least 5.0 grams of sample in order to achieve 40m^2 of total surface area.
- **Limit of detection** is said to be ~ 10 m^2 total surface area within sample holder when using N_2 as adsorptive gas. However, I have been able to achieve accurate, repeatable SSA with N_2 gas with LOD $\leq$ 0.24 m^2 total SA using 15.1g of TiO_2 material with SSA of 0.16 m^2/g. For material with expected SSA < 0.01 m^2/g krypton (Kr) should be used as the adsorptive gas. The limitations of Kr are that it can not be trusted for porosity measurements and it is very expensive ($380 per small lecture bottle of ~10L compared to tens of dollars for same amount of liquid N_2).
- Be sure to **record mass** of glass sample tube, with glass filler rod and seal frit for weight without sample and after sample is inserted. But it is important to get both mass measurements AFTER degassing and He backfill has occurred for empty and sample filled measurements. This gives the **truest value for the mass of your sample** alone (without hydration weight, etc. which is used to quantify all other calculations for SSA and porosity. (see steps 13 & 18 in procedures)

What sample preparation and analysis time can I expect?

- **Your sample material surface is sufficiently dry when the evacuation rate is < 5µmHg/min on the meter during the degas step.** (see steps 11-12 in Degas procedures)
- It is important to dehydrate sample material as much as you can prior to degassing on instrument. This could be done using an oven if sample is not at risk of surface transformations at high heat. If there is any concern for surface characteristic alterations with heat, then use a dessicator or fume hood instead. (see step 3 in procedures)
- **Try to find examples in the literature of degassing times and temperatures**. If degas times can not be found in the literature for your sample type, set to a conservative low temperature so as not to inadvertently transform your surface properties and start with a large temperature hold time, like 3000 min. Then keep checking by clicking "Check" button, wait 30 seconds to a minute to get a stable evacuation rate of moisture (and other volatiles if present), until evacuation rate is < 5µmHg/min. See the screen shot in Degas Tab Procedures in Figure 8. (steps 11 & 12 in Degas procedures)
- The degassing step is required to evacuate any moisture (or VOCs) in your sample which will negatively impact the surface interaction with the N_2 gas. **Plan for 6 to 8 hours** (360 to 420 min) of degassing even for "dry" samples (could be 48 hrs or more for hydrated samples). Fill the cryotrap (condensation trap) dewar in the center of instrument with liquid N_2 prior to degas step in order to protect the $3000 turbo vacuum pumps from water damage. (steps 9-18 in Degas procedures).
- Determine what details of your surface characterization are needed for your analysis because option 1 can save up to 5 hrs for the analysis step.
  Option1: Short analysis times of 2-3 hrs.
  Determination of **specific surface area (SSA)** only need to ramp the relative pressure of N_2 (or Kr if needed for very low SSA < 0.01 m^2/g) through the linear range of the adsorption isotherm (e.g 0.2 – 0.6 p/p^0 in Figure 3 plot); Therefore the 6pt, 7pt, or 8pt BET SSA analysis methods (.anc file type) can be chosen and have shorter run times.
  Option 2: This analysis takes 5-8 hrs per sample depending on total SA present.
  If in addition to SSA, porosity characteristics are needed such as porosity diameter distribution, relative SA or pores compared to total SA, or estimated nanoparticle size then the a full adsorption and desorption isotherm is needed using method "**Full Ads/Des Isotherm N_2 @ 77K.anc**" with P/P_0 set to 0.995. For proper calculations of porosity and nanoparticle size the **Density of your material must be known** along with the mass of sample and entered on "Sample tube info" tab (so that method can determine volume V = m/D).

What equipment or physisorption gas should be used?

- **Use rubber gloves** or lint-free cloth when handing glass sample tubes or filler rods, so as not to contaminate the glass surfaces.
- Fill the cryotrap (condensation trap) dewar in the center of instrument with liquid N_2 prior to degas step in order to protect the $3000 turbo vacuum pumps from water damage. The large laboratory dewar in W108 Plant Science can be filled with 10L of Liquid N_2 from Chem Stock room, and expect an extra 1-2 L charge for cooling the empty warm dewar. The 10L can last for 4-5 days allowing for up to 10 samples if you can get two analyzed per day. A polymer dipstick can be used when filling ASAP 2020 analysis dewar prior to each analysis (see Figure 2 below). If there is any concern for possible moisture loss during analysis step, then the cyrotrap (condensation trap) dewar in the center of the instrument should also be filled to protect the $3000 turbo vacuum pumps from damage.

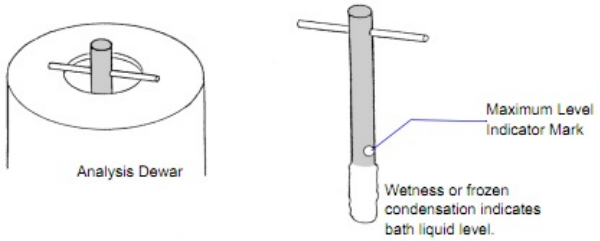


Figure 2 *Sketch of appropriate liquid N_2 level in dewar using manufacturer's dipstick*

- N_2 as the physisorption gas should be used anytime porosity measurements are also desired. The filler tube is used to ensure accuracy in low total surface area samples (less than 100m^2) by reducing free-volume space and preventing adsorption of physisorption gas to internal glass surface. Although, the operator manual does report that the filler tube can cause variability in micropore analysis due to interference with thermal transpiration correction. Filler tube is not necessary for total surface areas above 100m^2.
- Krypton can be used as physisorption gas for low specific surface area substances where greater than 10m^2 total surface area can not be achieved in the sample tube. However, Kr is not effective for porosity measurements. This need to be purchased prior to use as we do not normally stock this gas and it is very, very expensive at $350 per small lecture bottle of ~10L).

What can be gained from the full ads/des isotherm plots?

- **Langmuir Isotherm plots** are needed to determine any of the porosity or nanoparticle size calculations mentioned above in Option 2.
- **Langmuir Isotherm plots** like the linear plot shown below can be used to characterize one of the 4 main adsorption types and help suggest likely processes involved in the adsorption/desorption process (though **can not** provide **evidence of mechanism** as other investigations would be needed to confirm mechanism of ads/des). For example, my TiO_2 pellets exhibit TYPE IV ISOTHERM: with (a) mild hysteresis (delay) on the desorption isotherm due to capillary condensation (see plot below), (b) typically indicates moderate physisorption abilities with mesoporosity of somewhat irregular organization. More irregular "ink bottle type" pores with narrow necks and wide bodies can cause much larger desorption hysteresis.

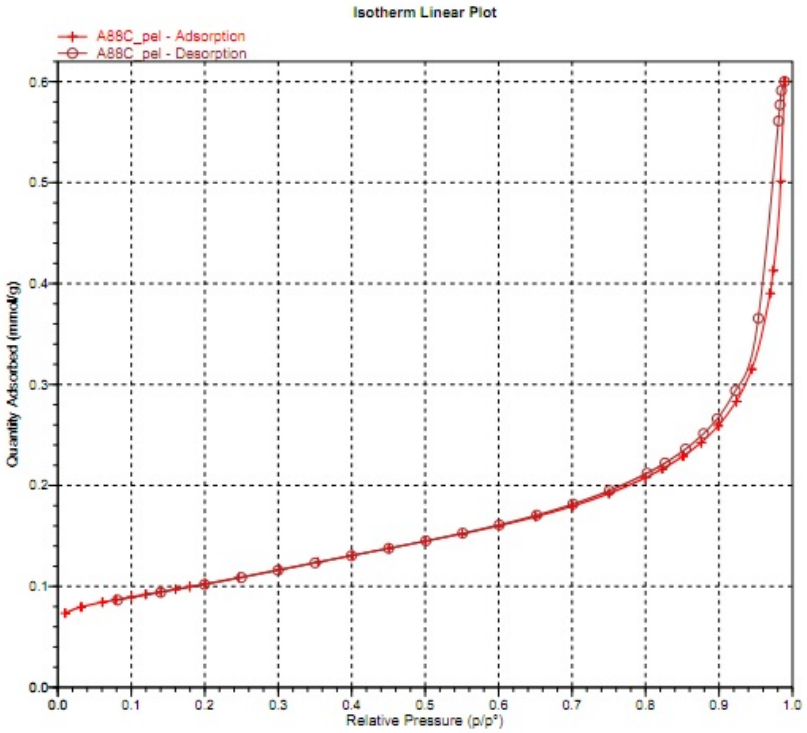


Figure 3 *Linear plot of Langmuir Adsorption/Desorption Isotherm. The plot shape or type can be indicative of ads/des processes that may be at work. The linear range of data is what is used for creating the BET transform plot and calculating BET surface area. Log plots can also be performed.*

Micromeritics ASAP 2020 BET Surface Area and Porosity Analyzer

Standard Operating Procedure

Energy Conservation Practices: In an attempt to conserve energy and preserve the life of our vacuum pumps, the instrument and computer must be turned on and the two preliminary vacuum pumps must be plugged in prior to each use. (see Steps 1 & 2) This means that during periods of time without instrument use, the instrument & computer should be turned off and vacuum pumps unplugged. (see step 29)

I. Instrument Preparation, Sample Preparation, and entering Sample Information into ASAP 2020 Software

1. Turn on the ASAP 2020 instrument first on the right side of the instrument and make sure the two preliminary vacuum pump plugs are plugged in. These plugs exit the back from inside the instrument panel and the pumps themselves can not be seen unless you open the front panel of the instrument. You will hear preliminary vacuum pumps turn on once plugged in.Check that the green light on upper left comes on with the instrument AND both green lights on the upper right come on indicating that both turbo pumps (additional vacuum pumps located inside the upper portion of the instrument) are functioning. The instrument and vacuum pumps should remain on for at least 3 hours prior to use to establish an appropriate vacuum. It is best to let it do this overnight if you have time.
2. Now, the computer can be turned on to communicate with the instrument. **User name:** BorchLabUsr and password: envirochem2014 (or look at sticky note under keyboard or on back of computer monitor is password details have changed). The **ASAP 2020 Operator's Manual is provided as a pdf file on the desktop** if further reference or trouble shooting is needed.
3. Prepare sample by drying it well, in fume hood, prior to degassing with instrument. The degassing step will take a lot longer if sample is still wet and too much moisture could damage the vacuum pumps.
4. Fill the cryotrap (condensation trap) dewar in the center of instrument with liquid N_2 prior to degas step in order to protect the $3000 turbo vacuum pumps from water damage. The large 10L laboratory dewar in W108 Plant Science can be filled with 10L of Liquid N_2 from Chem Stock room.
5. Confirm that the N_2 gas regulator is set to 10-12 psig (gauge pressure as read on the regulator dial). Confirm that the He gas (used for backfilling sample tubes) is set to 10-16 psig.
6. Open ASAP 2020 software on the desktop. File → Open → Sample Info (F2) → Dbl Click on [..] to browse C drive directory → Create a file folder with your name, e.g. "Jeramy" Folder and save your sample data in this folder. This folder can be created or found later at My Computer → C drive → ASAP 2020 folder → data folder → Jeramy.
7. Leave initial numerical sample name, as it is our way of tracking total samples run on instrument, e.g. 000-045.SMP → then click Create new file, OK → on the next window in the "sample info" tab you may now rename this data in a way you will recall its content. The file will be saved as a .SMP file with your specific name and the sample # in the data folder specified earlier.
8. Most tabs in the Sample Information window remain with the default settings except:

   Sample Information tab: You will open this again later to **insert mass** of sample later (for specific surface area analysis)…and **insert density** as well if you want accurate nanoparticle size and porosity measurements (e.g. BJH ads or des porosity). This is because these latter measurements require a known volume of sample in the tube, thus the software uses Vol = mass/density to get this value.

   Degas Conditions tab: As a starting point, evacuation target temp 30°C and hold time 10 min are probably good values to use for initial moisture evacuation without heat. Heating target temp can be set from 28°C to 300°C and 1000's of minutes. You want to be sure that the temperature is below any threshold that may cause physical and/or chemical changes that could affect your sample surface characteristics. Look in the literature for appropriate heating temperatures and target times. If no literature protocols can be found, start with a conservative temperature, even as low as 28°C - 35°C, and a long holding time like 3000 minutes and just keep checking the degas progress intermittently. This could take 5 hours with an extremely dry sample or multiple days with wet samples.
   This is done by clicking Check below the Degas Schematic (as shown below in screen shot), waiting a minute for a stable evacuation rate reading, then Continue until a gas evacuation rate <5µmHg/min is achieved. When your sample has

achieved this low evacuation rate, then click continue, followed by Skip or Cancel, then agree to cancel remaining degas time when asked.

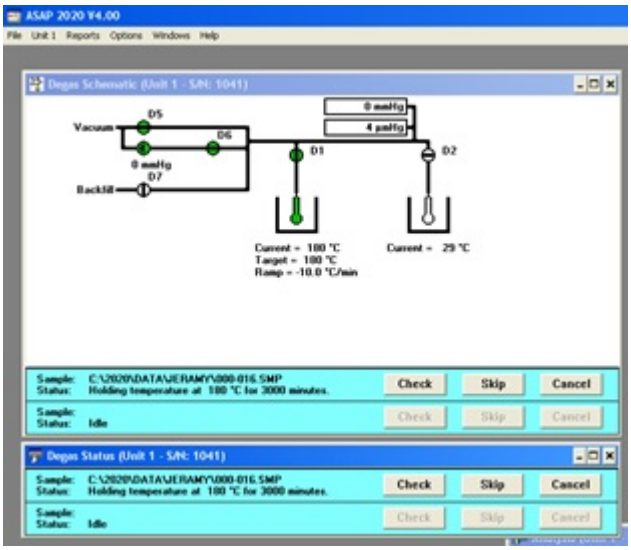


Figure 4 *Screen shot of Degas Schematic Tab*

Note: We use 180°C target temperature and 420 minutes hold time for my TiO_2 pellets since this temperature is low enough to prevent any transformation from anatase to rutile phase which happens near 600°C. We use only 60°C for target temperature for ferrihydrite coated silica sand to remain safe.

Analysis Conditions: The default analysis method conditions should be programmed to automatically appear for standard physisorption analysis (Langmuir adsorption isotherms and BET surface area analysis). If not, click Replace and search in parameter folder for "**Full Ads/Des Isotherm N_2 @ 77K.anc**" with P/P_0 set to 0.995 →Click Save. You may also choose other analysis method files if needed.

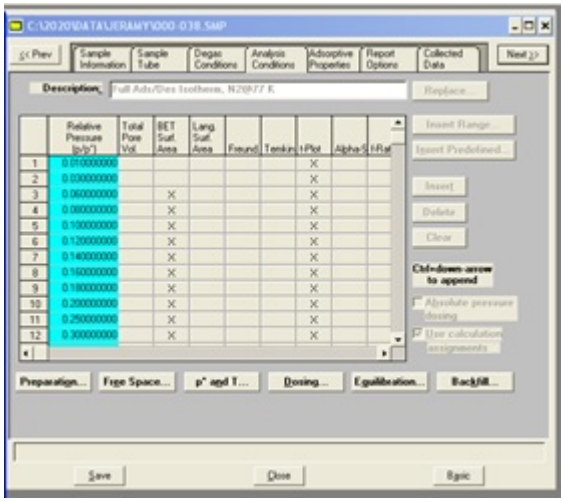


Figure 5 *Screen shot of Analysis Conditions Tab*

Adsorptive Properties: The default adsorptive conditions should be programmed to automatically appear for **Nitrogen** gas physisorptive gas under "**Ideal Gas Law**" Conditions between checked. Check to see that the parameters shown below in the screen shot Figure 6 (a) match those in your adsorptive tab, such as hard sphere diameter of 3.860Å, molecular cross-sectional area of 0.162 nm^2 and Ideal Gas law being checked. If they don't, you may need to click Replace and search in parameter folder for "**Nitrogen.adp**" file (not the N2 Standard.adp file which uses real gas conditions checked) →Click Save. You may also choose other or create new .adp files if needed, such as the one that we use for krypton physisorption gas shown below "Krypton.adp" in Figure 6 (b) with hard-sphere diameter of 3.810Å and molecular cross-sectional area of 0.210 nm^2, and Ideal Gas law checked.

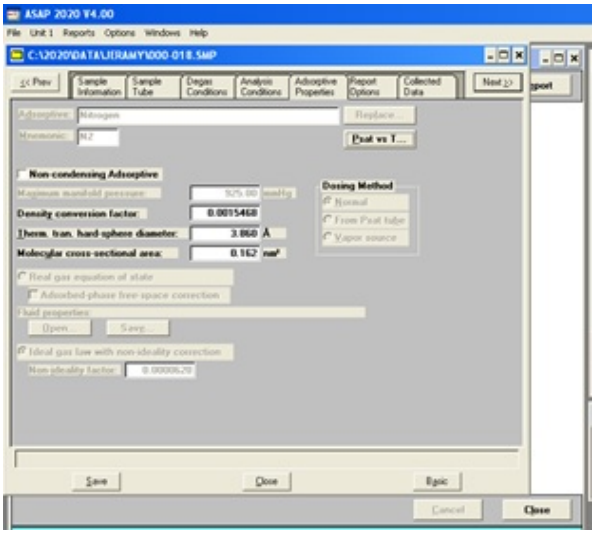
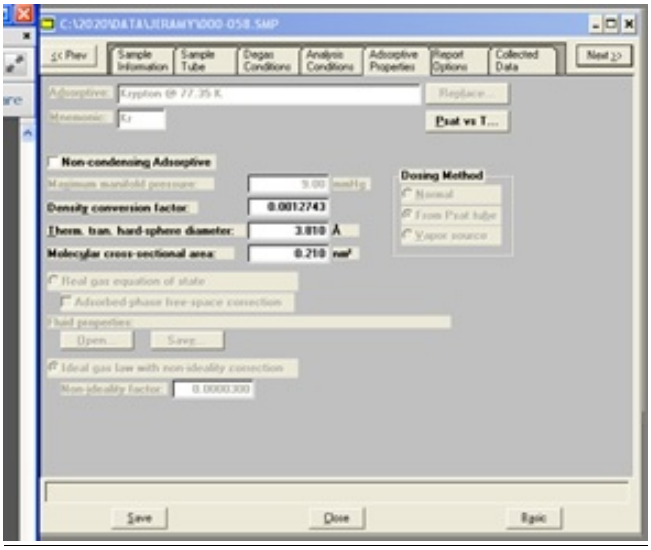



Figure 6 *Screen shot of Adsorptive Properties Tab for (a) Nitrogen.adp file and (b) Krypton.adp file*

Report Options: double click on Summary report and any other reports you think are relevant. If none are chosen, you can later go to File → Sample Info → find your data file and then add reports of interest to you at that time.

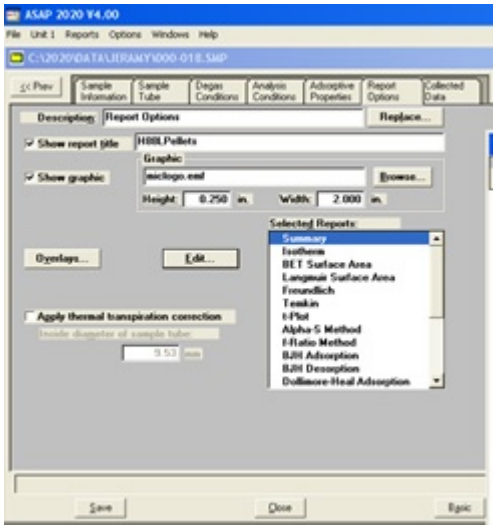


Figure 7 *Screen shot of Report Options Tab*

Calculated Data: Data will appear hear after analysis is complete. You will find that you can double click on X's to choose which data points to use or eliminate when generating calculations and reports.

II. Degassing Steps for EMPTY and SAMPLE-FILLED sample tubes → Record masses of each

Empty glass sample tube

9. It may be helpful to make a copy of the ASAP Series Sample Data Sheet in instrument binder or print a copy from page 379 (pg. A-3) of the ASAP 2020 Operator's manual to visually see what data is important to record.
10. Install EMPTY glass sample tube, (filler rod if used,) and seal frit onto either Degas port D1 (on left) or D2 (on right side) or both. A filler rod is typically used when total surface area of sample is less than $100m^2$. Add heating jacket with clip around the base of your EMPTY glass sample tube, (filler rod if used,) and seal frit. Add connector nut, metal ferrule (ring structure), and O-ring then screw tightly into port fitting for degassing. You must engage the spring inside when attaching and be careful to not break the glass sample tube by tilting during insertion.

    *Note: Instrument is capable of degassing two samples at once and doing one surface area analysis simultaneously. Degas and backfill with Helium gas. You may use automatic glass degas setting which evacuates then heats at 300°C until evacuation < 5µmHg/min. It will then cool and backfill with He gas.

11. Click Unit 1 Tab → Start Degas → Top D1 is Left Port and Bottom D2 is right port → You may choose the standard Glass Degas file or browse to find your sample name and leave with "degas conditions" → Ok.

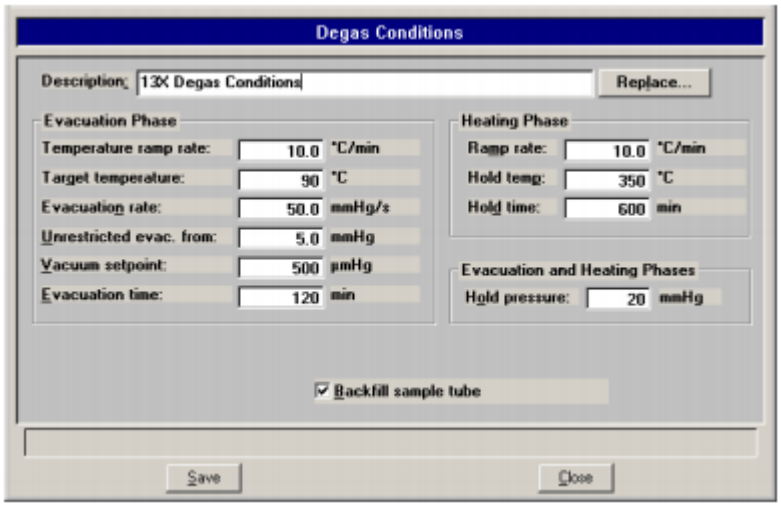


Figure 8 *Screen shot of Degas Conditions after clicking Start Degas*

12. Periodically click "Check Button" to see if evacuation rate < 5µmHg/min, click OK which returns to previous screen. Leave as is or Click Skip if want to skip remaining evacuation time → Yes to stop, Yes to backfill with He. Allow to cool to room temp. Remove warming jacket. Leave seal frit in and be careful to keep it sealed as your remove sample holder to be weighed.
13. Allow to cool to room temperature and backfill with He gas before removing warming jacket. Using gloves, Record the mass of the sample tube, (filler rod), and seal frit. Use styrofoam donuts to use as a holder apparatus to keep sample tube holder on mass balance. Accurate mass measurements are essential for proper BET surface area calculations.

Sample-filled glass sample tube

14. Now use gloves to remove seal frit and filler rod from empty sample tube. Add your solid or powder sample, trying to prevent sample material from sticking to upper 3 inches of sample holder for best accuracy. You may need to use the flexible, plastic insertion tube or I find creating your own tube from paper (in the shape of a straw) is even better…since it has less static electricity adsorption problems.

    *Note: Read obligatory section 0 above to help make your best estimate of expected specific area to determine required mass of sample required (range of approximately 1-15 grams), trying to achieve 40m^2 to 120m^2 total sfc area (10m^2 absolute minimum) in sample holder for best results. *Note: My ultra-fine TiO_2 powder would build up static electricity when using plastic insertion tube so I would create my own insertion tube of rolled printer paper which worked better.
15. Installed SAMPLE-FILLED glass sample tube, (filler rod if used,) and seal frit onto either Degas port D1 (on left) or D2 (on right side).
16. Click Unit 1 Tab → Start Degas → Top D1 is Left Port and Bottom D2 is right port → You may choose the standard Glass Degas file or browse to find your sample name and leave with "degas conditions" → Ok
17. Periodically click "Check Button" to see if evacuation rate < 5µmHg/min, click OK which returns to previous screen. Leave as is or Click Skip if want to skip remaining evacuation time → Yes to stop, Yes to backfill with He. Allow to cool to room temp. Remove warming jacket. Leave seal frit in and be careful to keep it sealed as your remove sample holder to be weighed.
18. Record the mass of sample and sample tube holder after degas and backfill. Subtract the sample holder mass recorded earlier and record mass of sample on data sheet. Now Is the time to add the mass (and density if known) of your sample into **Sample Information Tab**.

III. Analysis Steps: Adsorption/Desorption Analysis with N_2 gas for surface area AND porosity measurements

19. Using gloves, add white isothermal jacket, then connector nut, metal ferrule, and O-ring to the degassed and sealed sample tube with solid sample. Screw tightly into port on right side for analysis. Place the foam insulator lid around the sample tube and the saturation pressure probe (attached to instrument).
20. Remove miniature dewar from platform below analysis port and add liquid to just below the open hole on the metal T-bar dipstick (which should be near right side of instrument). Then place liquid N_2 dewar onto the elevator shelf below the analysis port being sure that it will align with the sample tube above when it rises.
21. The elevator will automatically rise ~40 to 60 minutes into analysis. Be sure that the elevator return path remains clear of obstacles during analysis.
22. Click Unit 1 Tab → Sample Analysis → Browse to find sample of interest →Ok → Start

23. Ads/Des process may take 4 to 6 hrs in 3 stages: Preliminary (~45 min), Analysis (~2.5-3.5hrs), Termination (~30min). You can watch as the adsorption and desorption isotherms are plotted. A line of text at the bottom of the screen labeled "Details" will report out what is being done throughout the analysis. See screen shot below**. It is worthwhile to sit through the first few hours of analysis to read the steps as they happen or at least print out a summary of it later.**

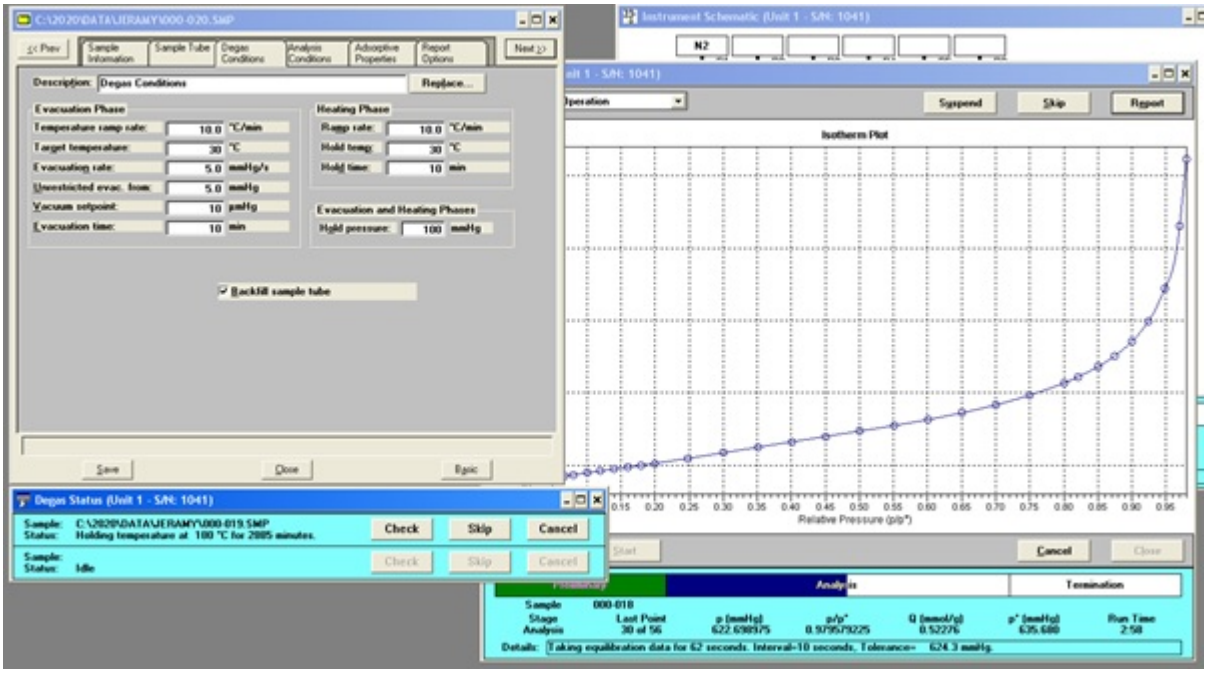


Figure 8 *Screen shot of details and plots reported in real time as analysis occurs*

24. After analysis is complete: cover Liq N_2 dewar with styrofoam lid to conserve until next analysis.
25. Click Reports Drop down menu on top of the screen → Start Report → Find sample # or name in C drive, 2020, data, Jeramy (You may need to dbl click the […] to find your data folder. Then dbl click on all reports of interest e.g. Summary, Isotherm, BET sfc area, t-plot, BJH Ads/Des, Langmuir isotherm are typical ones chosen. Is it usually easiest to print report as a pdf file but you can save as .rep (only able to be read by ASAP software), .xls spreadsheet, or .txt as well.

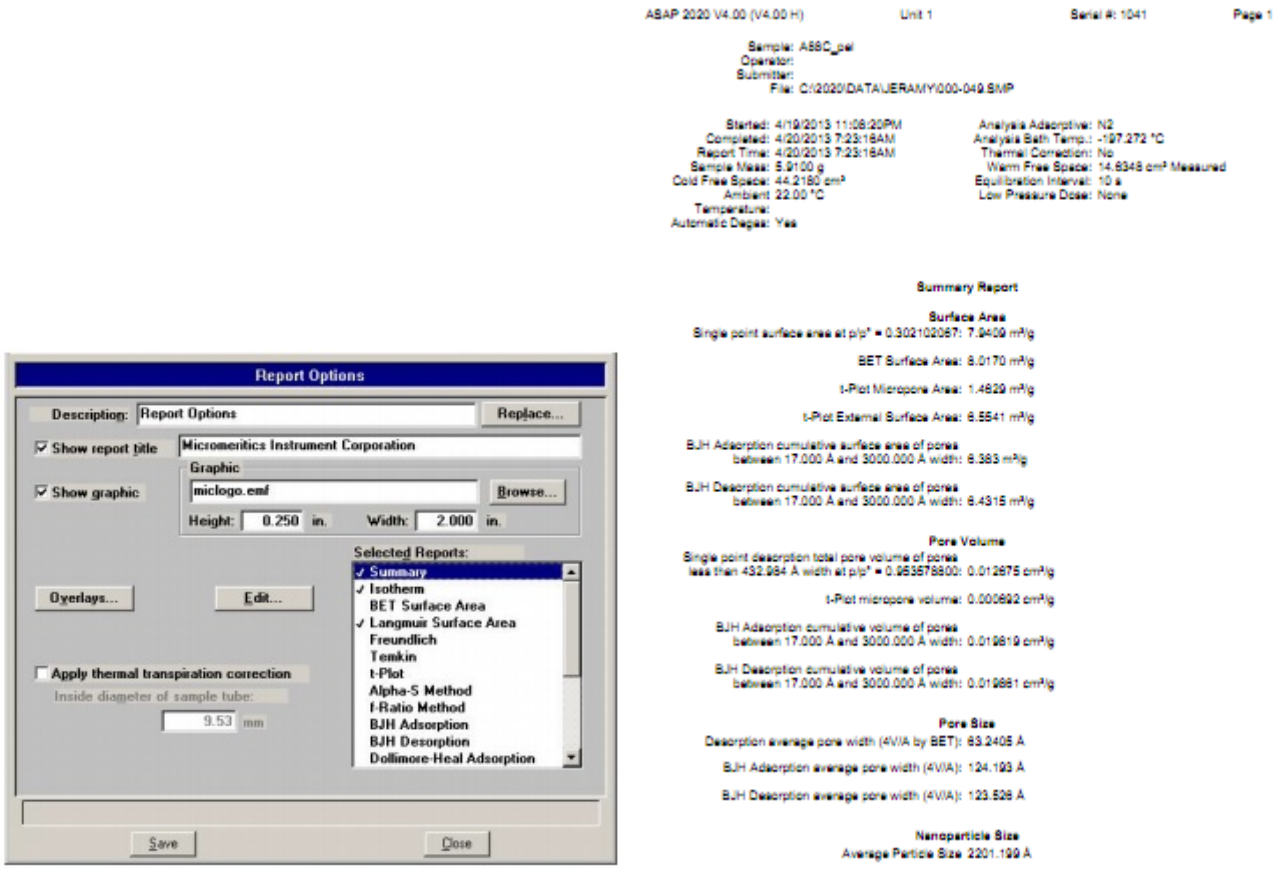


Figure 8 *Screen shot of (a) Reports drop-down menu and (b) Summary Report Page generated*

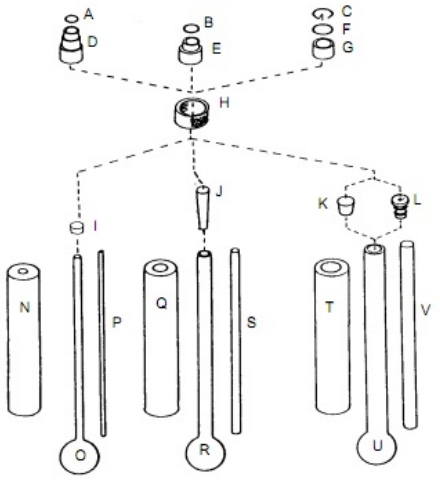
26. You may want to check the BET transform plot page and the BET Surface Area Report on reports to see if the 5 – 10 points chosen for BET calculation are linear with good correlational value (R^2 value). If not, you can go back to File → Sample info → Analysis Conditions and choose only those points that make a good linear fit by clicking or unclicking the X's in the column. Then redo the report. This will improve the accuracy of your BET specific surface area and other related calculations. You can see BET surface area calculations in Appendix B of this document or in ASAP 2020 Manual.
27. You may choose to view the Linear Langmuir Plot (see **Figure 3** above) to ascertain adsorption/desorption classification (type I, II, III, or IV) of your surface and seek out possible processes occurring during adsorption or desorption to you surface.
28. You may choose the BJH ads/des tabulated data if you would like to create a pore size distribution diagram.

IV. Instrument Shutdown and Cleaning Glass Sample Holder

29. It is ok to leave the instrument and vacuum pumps on during the days or weeks of active analysis. However, please turn of the instrument, the computer, and unplug the two preliminary vacuums during long periods without use. These two vacuums do not have an easily accessible on/off switch although and internal circuit breaker for them is located inside the platform panel of the instrument if needed.
30. Please remove all of your solid sample material from glass sample holder and clean properly for next user. This requires a) scrubbing with the small brush (chimney sweep style) with lightly soapy water (Alconox and water), b) 2-3 hot tap water rinses to remove soap, c) multiple deionized water rinses to prevent salt remnants and d) acetone wash (or whatever is best for cleaning any solid residue, isopropanol or methanol, etc). If sample tube is particularly dirty, adding the sample tube and filler rod to a bath of 5grams Alconox to 500mL water in a sonicator to remove all particulates on the glass surface.
31. Allow a short time for acetone to dry, then bake in a drying oven at 110°C for 2 hours.
32. After baking and cooling, use gloves to return the sample tube, filler rods and seal frits to their proper locations near the BET ASAP 2020 instrument to avoid transfer of oil on hands onto glass surfaces (causing negative effects on accuracy).

Appendix A Instrument and Sample Tube Equipment for Reordering

1/4-in., 3/8-in, and 1/2-in Sample Tubes



Part Number		Item and Description
004-25466-00 004-25465-00	A	O-ring, size 010, Buna-N, for 1/4-in. sample tube O-ring, size 010, Kalrez, for 1/4-in. sample tube
004-25022-00 004-25022-01	B	O-ring, size 012, Buna-N, for 3/8-in. sample tube O-ring, size 012, Kalrez, for 3/8-in. sample tube
260-25891-00	C	Opener, seal frit, for 1/2-in. sample tube
240-25803-00	D	Ferrule, 1/4 in.
240-25802-00	E	Ferrule, 3/8 in.
004-25044-00 004-25474-00	F	O-ring, size 013, Buna-N, for 1/2-in. sample tube O-ring, size 013, Kalrez, for 1/2-in. sample tube
260-25843-00	G	Ferrule, 1/2 in.
300-25824-00	H	Nut, sample tube

Part Number		Item and Description
004-32604-00	I	Cap (stopper) for 1/4-in. sample tube (not shown)
004-32004-00	J	Stopper, for 3/8-in. sample tubes
240-32000-00	K	Stopper, for 1/2-in. sample tube
260-25890-00	L	Seal Frit with built-in check valve for air-sensitive samples
202-25901-00	N	Isothermal jacket, 1/4 in.
240-61001-00	O	Sample tube, 1/4 in.
240-61014-00	P	Volume displacement insert, 1/4 in.
202-25902-00	Q	Isothermal jacket, 3/8 in.
240-61002-00	R	Sample tube, 3/8 in.
240-61015-00	S	Volume displacement insert, 3/8 in.
202-25903-00	T	Isothermal jacket, 1/2-in. sample tube
240-61003-00	U	Sample tube, 1/2 in.
240-61016-00	V	Volume displacement insert, 1/2 in.

Appendix B Important Equations used for analysis

See Appendix C of ASAP 2020 Operator's Manual for more theory and equations for all analysis possibilities of this instrument.

BET surface area is shown below

For each point designated for surface area calculations, the BET[1] transformation is calculated as follows:

$$B_1 = \frac{P_{rel_1}}{(1.0 - P_{rel_1}) \times N_{ads_1}}$$

where B_I is in units of g/cm^3 STP.

A least-squares fit is performed on the (P_{relI} ,B_I) designated pairs where P_{relI} is the independent variable and B_I is the dependent variable. The following are calculated:

a. Slope (S g/cm^3 STP)

b. Y-intercept (Y_{INT} g/cm^3 STP)

c. Error of the slope (S_{ERR} g/cm^3 STP)

d. Error of the y-intercept (YI_{ERR} g/cm^3 STP)

e. Correlation coefficient (Cc)

Using the results of the above calculations, the following can be calculated:

BET Surface Area (m^2/g):

$$SA_{BET} = \frac{CSA \times (6.023 \times 10^{23})}{(22414\ cm^3\ STP) \times (10^{18}\ nm^2/m^2) \times (S + Y_{INT})}$$

where

CSA = analysis gas molecular cross-sectional area (nm^2), user-entered on the Adsorptive Properties dialog box

Volume of the Monolayer (cm^3/g STP):

$$V_M = \frac{1}{C \times Y_{INT}} = \frac{1}{S + Y_{INT}}$$

BET C value:

$$C = \frac{S + Y_{INT}}{Y_{INT}}$$

Error of the BET Surface Area (m^2/g):

$$BET_{ERR} = \frac{SA_{BET} \times (S_{ERR}{}^2 + YI_{ERR}{}^2)^{0.5}}{Y_{INT} + S}$$

Appendix C Obligatory things to consider PRIOR to analysis

See section 0. above