

Micromeritics 3Flex Surface Characterization Analyzer



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1 Description

- (a) The SmartPrep is a flowing-gas degassing unit which removes adsorbed contaminants from the surface and pores of your sample in preparation for analysis. It contains six sample ports, each one independently temperature-controlled for greater flexibility. It contains two serial ports, one for connecting to the computer and the other available for connection of an additional SmartPrep. The temperature, ramp rates, and soak times of each sample are individually controlled by computer. Up to five ramps and soaks are allowed. All degas information is integrated into the sample data file for easy reference in the future.
- (b) The Smart VacPrep is an advanced six-port system that utilizes vacuum to prepare samples by heating and evacuation.
- (c) Each of the ports may be operated independently. Samples may be added or removed from degas ports without disturbing the treatment of other samples undergoing preparation. Degassing automatically terminates when the samples have completed all programmed steps.
- (d) The Smart VacPrep software includes a set of default ramp and temperature parameters. However, you also have the option of programming each port with a different set of parameters. A record of the sample preparation time and temperature can be recorded as part of the sample data file.
- (e) The degas program may be started or terminated by using software installed on the computer attached to the analysis instrument or with push-buttons on the front panel of the Smart VacPrep. This time-saving, Rapid-Start mode on each port gives you the ability to walk up to the instrument, attach the sample tube with heating mantle, and immediately start degassing a sample.
- (f) Micromeritics 3Flex Surface Characterization Analyzer is a fully automated, three-station instrument capable of high-performance physisorption, mesopore, micropore, and chemisorption analyses with superior accuracy, resolution, and data reduction. Each analysis station is upgradeable from mesopore to micropore with the option of designating one station for chemisorption analyses. All analysis stations can be configured for krypton analysis of low surface area materials. Vapor sorption capability is standard.

²http://www.micromeritics.com/Product-Showcase/Sample-Preparation-Systems/SmartPrep_DeGasser.aspx³<http://www.micromeritics.com/Product-Showcase/Sample-Preparation-Systems/Smart-VacPrep.aspx>

⁴<http://www.micromeritics.com/product-showcase/3Flex-Surface-Characterization-Analyzer.aspx>

2 Physisorption Analysis

Setting up Samples on Computer Software (3Flex): The software should be programmed before degassing or running a sample.

- (a) Open the 3Flex software on Desktop.
- (b) Go to File → New Sample → Physical Adsorption. A new folder will open containing four tabs: Sample Description, Degas Conditions, Analysis Conditions, and Report Options. Follow the steps below for each tab.
 - i. Sample Description:
 - A. *Method*: Select the method you would like to use to perform the experiment.
 - B. *Sample*: Give the sample a name. Note this name is for this folder only. The samplename is the name given when you save the sample file.
 - C. *Operator*: Provide your name.
 - D. *Submitter*: Provide your name.
 - E. *Sample tube*: Sample tube is the default selection.
 - F. *Mass*: You have an option to “Enter” or “Calculate” the mass. “Enter” refers to input a mass in the sample setup directly. “Calculate” refers to calculate the sample mass based on the mass values you enter for sample tube and sample tube + sample. It is not necessary to input a mass before the degas has been performed as your mass will change in that process. You will have the option to change your mass when transferring your sample from the smart vacprep system to the 3flex system and setting up to do the actual physisorption.
 - G. *Type of data*: Automatically collected
 - ii. Degas Conditions: **This tab is used to setup the smart vacprep degas system.**
 - A. *Degas Conditions*: Degas Conditions
 - B. *Copy quickstart degas conditions from smart vacprep unit*: 1
 - C. *Port*: None
 - D. *Backfill sample tube*: Check the box. You can opt to backfill “Automatically” or “Wait for Operator”. In general the sample tube is usually backfilled with nitrogen automatically but if you choose not to do so you can backfill it manually afterwards.
 - E. The next series of options deal with the pressure of your degas procedure and the initial heating process.
 - F. *Evacuation rate*: This specifies the rate at which you pull down initially from atmospheric pressure.
 - G. *Unrestricted evac from*: refers to the point at which the instrument should stop controlling the evacuation rate set above

and simply pull as strong of a vacuum as possible. This can be of concern if you have fluffy light powders since it is possible to suck the powder into the instrument if you pull vacuum too quickly. If this happens please contact one of the primary users as the filter for that port will likely need to be replaced or the instrument vacuum chamber cleaned out.

- H. *Vacuum level*: Specify the vacuum level you wish to obtain.
 - I. *Evacuation time*: Set the time you wish to hold the entered vacuum level.
 - J. *Temperature ramp time*: Specify the temperature ramp rate you intend to use for the initial degas evacuation phase.
 - K. *Target temperature*: Specify the temperature you intend to use for the initial degas evacuation phase.
 - L. *Hold pressure*: Specify the holding pressure for degas phase
 - M. *Insert*: Creates a line above the currently selected line in the box and append can bring in a procedure from file.
- iii. Analysis Conditions: This tab identifies the actual physisorption analysis procedure you will be using to collect your isotherms and hysteresis loops.
- A. *Analysis Conditions*: Run conditions
 - B. *Adsorptive*: Typically, nitrogen is used but other inert gases (Ar, He) can also be used.
 - C. The values in the box are used to set up the actual parameters for your analysis in terms of partial pressures of adsorbate and when to collect data points.
 - D. *Up to*: determines the range you are looking to specify data collection for.
 - E. *Relative Pressure*: refers to what the step should be in terms of partial pressure steps between taking data points.
 - F. *Dose Amount*: allows you to specify to take a point after certain doses of adsorbate are added but this is not typically used.
 - G. *Equilibration Interval*: refers to the amount of time allowed for the adsorbate gas to diffuse into or through your material and reach a pressure equilibrium within the sample holder after it has adsorbed the adsorbate.
 - H. The remaining tabs (see below) allow for control of the system outside the actual adsorbate analysis.
 - I. *Preparation*: allows you to prepare your sample. In the case of physisorption this is most likely degassing directly on the 3Flex instrument but typically the external smart vacprep setup is used for sample preparation.
 - J. *Free Space*: allows you to set when the instrument determines the free space measurement for your sample.
 - K. *Po and T*: allow you to control the permissible margin of error on measuring pressure and temperature.

- L. *Dosing*: allows you to control the permissible margin of error for the pressure of gasdosed in.
 - M. *Termination*: allows you to select whether your sample should be backfilled with nitrogen, remain under vacuum, etc. after the run. Typically, we will backfill with nitrogen.
- iv. Reporting Options:
- A. This tab allows for mostly cosmetic control of the report file you can generate from your sample data such as including the title or a picture of the micromeritics logo. The selected reports say what will be generated as standard when asked for a report following running your sample. This is not really necessary as you can specify to generate a new report from the sample data at any point and change which data will be included with that report. We will discuss this later in the manual.
 - B. After you have filled out all the tabs, save the sample.
- (c) Quick and Easy Options: There are some options in setting up a new sample which makes life quicker and easier. Both options are within the “Sample Description” section. One of these two options will be the process used by most users of the instrument.
- v. *Option 1*: In the method tab there is the option to browse and load a method that has been previously used. Several sample methods are provided as starting points for instrument users.
 - vi. *Option 2*: At the bottom of the method tab is a “Replace all” option. This allows you to load the entire file for a previously run sample. If you have run the sample before, or if it is one in a series of related samples, click this button, find the file, change the sample name and save it with the new name.

2. Preparing A Sample: Preparing samples involves four steps; weighing samples, mounting samples to the degas unit, degassing samples, and transferring samples to the 3Flex instrument.

(a) Weighing Samples

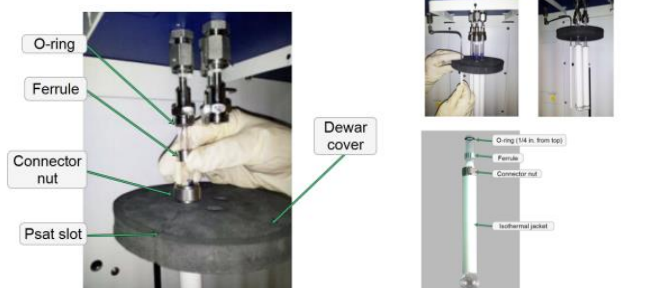
- i. Use flat bottom 12mm physisorption tubes for physisorption analysis. These should be washed and cleaned using a scrubber, being sure to remove all material from the corners of the tube.
- ii. Weight empty tube
- iii. $50m^2$ of 50 mg of material is suggested for use with this instrument. This is a general guideline to minimize errors in weighing. The instrument can run less sample but typically this is not advised.
- iv. The sample should be weighed out into the tube with an accuracy of up to four decimal scale. This is often easiest done by recording the mass of the tube, measuring a mass of sample on weigh paper, adding it to the tube, then measuring the combined mass of the tube and sample with TranSeal.

(b) Mounting Samples to Smart VacPrep

- i. If your sample does not have a large amount of volatile material, extremely high water content, templating agents, your material is a polymer that likely contains small oligomers, etc., use the Smart VacPrep setup for degassing.
- ii. If your sample has a large amount of volatile material, extremely high-water content, templating agents, your material is a polymer that likely contains small oligomers, etc., use the Smart Prep system which operates using flowing gas and heating rather than pulling vacuum on your sample and heating.
- iii. For using the Smart VacPrep, first unscrew the stainless steel attachment to the port you intend to use (1-6 from left to right as you face the instrument). On the port, you see a screwcap, a glass cap where the sample tube goes, white plastic ferrule, a stainless-steel liner inside the cap, and a black O-ring.
- iv. Place your sample tube in the stainless-steel screwcap with white plastic ferrule with smaller side up to the outlet of tube, and metal liner with taper section facing down to the bottom of tube.



- v. Put the black O-ring at the end of the sample tube.
- vi. Insert TranSeal to prevent backflow into reactor
- vii. Weight sample tube with sample and TranSeal
- viii. Place the assembly into the empty port and gently push the glass sample tube up until it stops against the metal of the port.
- ix. Tighten the screw cap around the sample tube sealing the O-ring.
- x. Beneath the ports there are six grey heating sleeves with built-in thermocouples. Be sure to use the thermocouple/heating sleeve that goes with the port your sample is in.
- xi. Slip the sleeve over the bottom of the sample tube and fasten with a stainless- steel clamp. Be sure to have the bottom of the sample tube in contact with the bottom of the sleeve as this is where the thermocouple is built within the sleeve.



(c) Degassing Samples

- i. On the 3Flex software, go to Smart VacPrep → Unit1 → Start Degas.
- ii. Browse to load the sample file you have created → Open → Start.
- iii. After completion of the degas procedure the instrument will backfill the sample tube if you chose to do so in the sample file or leave the tube under vacuum. If the latter is the case the tube can be manually backfilled from the Smart VacPrep tab.
- iv. After the sample tube is backfilled with nitrogen, remove it from the SmartVac Prep system.
- v. Recap the Smart VacPrep port with the glass cap after removing your sample.
- vi. Transfer your sample over to the 3Flex instrument.

(d) Transferring Sample to 3Flex

- i. The 3Flex instrument ports are typically kept under vacuum. To break the seal on the glass cap, backfill with nitrogen: From 3Flex software → Unit1 → Select Physical Adsorption → Backfill ports → Choose the port(s) you wish to use. The 3 ports are numbered 1-3 from back left to middle front to back right as you face the instrument. You can use up to three ports at a time when performing your measurements for physisorption; however, only port 2 (center front) can be used for microporous samples.
- ii. To prepare your sample tube, place a filler rod into the sample holder. Then slip a white sleeve down to the base of the sample tube. This is used to wick up nitrogen to the top of the sleeve throughout the run minimizing changes in temperature around the sample.
- iii. Place the grey foam circle around the sample tube aligned so that the reference rod (metal rod to the left of the 3 sample ports that can pivot and is covered with a white sleeve) can slide into the groove cut for it smoothly. Tighten the sample tube into the port as you did for the Smart VacPrep system taking note that now there should be a brown o-ring rather than a black one. A second or third sample can be placed into their ports by sliding through the bottom of the grey foam circle. Once all samples are attached to their appropriate ports the liquid nitrogen holder can be filled.
- iv. To verify that the liquid nitrogen dewar is filled to the proper level, there is a T-shaped rod located by the stand for sample tubes next to the 3Flex instrument. On the T-shaped rod is a circular hole. The liquid nitrogen level should come to the middle of the base of the rod and the hole when placed down into the liquid nitrogen dewar. Mount the dewar onto the mechanical lift stand.

3. Running A Sample: On 3Flex software, go to Unit 1 → Sample Analysis → Browse to desired sample(s) for desired port(s) → Start.
4. Generating a Report File: From the reports tab, select to create a report and select the sample you want to use. Specify the data you want to see and generate the report. You should now be given a report file containing the desired information and parameters.

3 Chemisorption Analysis

1. Setting up A Sample File:
 - (a) Open the 3Flex software on Desktop.
 - (b) Go to File → New Sample → Chemical Adsorption. A new folder will open containing three tabs: Sample Description, Analysis Conditions, Report Options. Follow the steps below for each tab.
 - i. Sample Description: You can select a method you have previously designed to auto-fill most of the parameters or follow the steps below.
 - A. *Method*: Select the method you would like to use to perform the experiment.
 - B. *Sample*: Give the sample a name. Note this name is for this folder only. The samplename is the name given when you save the sample file.
 - C. *Operator*: Provide your name.
 - D. *Submitter*: Provide your name.
 - E. *Sample tube*: Sample tube is the default selection.
 - F. *Mass*: You have an option to “Enter” or “Calculate” the mass. “Enter” refers to input a mass in the sample setup directly. “Calculate” refers to calculate the sample mass based on the mass values you enter for sample tube and sample tube + sample. Remember to weigh your sample AFTER you have calcined your sample since you cannot weigh your sample after any potential mass loss during your pretreatment on the 3flex instrument.
 - G. *Type of data*: Automatically collected
 - H. *Active Metals*: Enter in the % of sample weight of the metal you are interested in. You can also click on any metal and click on Adsorptive if you wish to modify the default stoichiometry factors for given gases. You can “Insert” any new metals if you do not see yours on the list.
 - ii. Analysis Conditions: This tab identifies the actual chemisorption analysis procedure you will be using to collect your isotherms and hysteresis loops.
 - A. *Analysis Conditions*: Run conditions
 - B. *Adsorptive*: Typically, nitrogen is used but other inert gases (Ar,

- He) can also be used.
- C. *Pre-analysis evacuation time:* 0.5 h
 - D. The values in the box are used to set up the actual parameters for your analysis in terms of partial pressures of adsorbate and when to collect data points. Enter in the Absolute Pressure data points you would like to take, including the interval and equilibration interval (20 s is recommended). For high surface area materials, you want to take dense data points near the beginning of your isotherm.
 - E. The remaining tabs (see below) allow for control of the system outside the actual adsorbate analysis.
 - F. *Preparation:* check "Backfill and match Transducer" in order to recalibrate the sample port pressure transducer to match the manifold pressure transducer. Check in-situ activation and enter in what tasks, gases, temperatures, and times you would like to pretreat your sample. We recommend you have a leak test at the end followed by evacuation again.
 - G. *Free Space:* Leave as default to measure after analysis
 - H. *Temperature:* Enter in the analysis temperature you wish to conduct chemisorption and the temperature rate.
 - I. *Dosing:* Enter in the pressure tolerance difference - 5 mmHg is the default.
 - J. *Termination:* set to backfill the port with the gas you wish to choose and if you want to cool the furnace.
- iii. Report Options: Select the cosmetic control of your report file and what specific reports you would like to be generated.
 - iv. Now that you have filled out the sample file, you can either save as a sample file or save as a method file so that you can pull up the method later for another sample and have the important parameters already filled.
 - v. Refer to manual for more detailed instructions on setting up the software.

2. Preparing A Sample

- (a) U-shaped chemisorption tubes should be washed and dried prior to loading the sample.
- (b) () of active metal surface area should be used with this instrument.
- (c) Be SURE to calcine your sample at an appropriate temperature BEFORE mounting running any chemisorption experiments. Your sample must be exposed to a temperature higher or equal to the highest temperature you plan to use for chemisorption.
- (d) The U-shaped tube should be weighed by itself with two small pieces of quartz wool. Be sure to use a 4 place weighing scale.
- (e) Add the first piece of quartz wool and push it down with the glass rod, load your sample, and cap it with the second piece of quartz wool. Make sure your quartz wool is not too tightly packed so gas can still flow through.

3. Mounting A Sample to 3Flex

- (a) If the glass cap is in place on the chemisorption port, go to 3Flex Software → Unit1 → BackFill ports → check 2, and begin to backfill the port with nitrogen so that you can take the cap off.
- (b) Screw the cap off and you are ready to mount your sample.
- (a) On the larger end of the U, slip on the large nut, followed by the smaller metal ring. Then roll the BROWN O-ring onto the tube. On the smaller end of the U, mount the smaller nut, followed by another metal ring with the thinner end facing down. Roll the second smaller brown O-ring onto the end. Place the filler rod into the sample tube. Remember the one for chemisorption has an opaque bottom. The filler rod is optional. It is used to minimize changes in temperature around the sample.
- (b) Carefully align the tube into the 2 ports for chemisorption.
- (c) Screw each end of the tube onto the instrument little by little. Do not screw one end all the way while leaving the other one unscrewed. Continue to screw in both ends gradually until both have a snug fit - do not over tighten or this will crack the glass.

4. Mounting Heater

- (a) Place the heater onto the mount. It should be a tight fit and not allow for any wiggle room.
- (b) Attach the cooling gas, thermocouple, and the mantle/furnace power. There is another thermocouple that needs to be mounted onto the sample tube. Plug in the second thermocouple and use the metal clip to fasten it onto the sample tube. The end of the thermocouple should be adjacent to the sample. Do this carefully and avoid cracking the glass.
- (c) Go to 3Flex → Unit1 → Enable Manual Control.
- (d) Right click the elevator in the instrument schematic port 2 and click on

Raise. Once the heater is raised all the way, go back and unclick Enable Manual Control. Place the two ceramic insulators onto the heater and your sample is ready to be run.

5. Running A Sample

- (a) Go to Unit 1 → Sample Analysis → Browse the sample file you want to run.
- (b) Click Start.
- (c) It may be worthwhile to check your run in a few hours to make sure it passes the leak check before doing the actual chemisorption.
- (d) Once your run is complete, the report file will automatically come up. You can hit Preview and then Save the report as an Excel or PDF.

4 Contacts

Instrument	Contacts
Micromeritics 3Flex Chemisorption Analyzer	Zack Partridge Customer Advocacy Specialist 770-662-3666 zack.partridge@micromeritics.com
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	Dereyone Moore Factory Service Representative dereyone.moore@micromeritics.com

