3FLEX

SURFACE CHARACTERIZATION ANALYZER

MICROMERITICS

OPERATOR MANUAL

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(Rev H)

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ABOUT THIS MANUAL

The following formats may be used in this manual.

NOTE - Notes contain important information pertinent to the subject matter.

CAUTION - Cautions contain information to help prevent actions that may damage the analyzer or components.

WARNING - Warnings contain information to help prevent actions that may cause personal injury.

Field Labels and Screen Titles

Labels and Buttons	Description
Buttons (in the application)	Buttons in the application are represented as bold font and blue letters — such as: **Save**, **Edit**, and **Replace All**.
Buttons (on the equipment)	Buttons on the equipment are represented as bold font and black letters — such as: **On**or **Off**.
Field Labels	Field Labels are represented as italicized words — such as: *Sample, Automatically Collected,* and *Analysis Conditions*.
Keyboard Commands	Keyboard commands are represented as bold font and black letters — such as: **F2** and **Alt+F4**.
Menu Instructions	Menu instructions are represented as bold and italicized words — such as: ***File > New Sample*** and ***Reports > Start Report***.
Screen Tabs	Screen Tabs are represented as italicized words — such as: *Sample Description*, *Analysis Conditions*, and *Report Options*.
Screen Titles	Screen Titles are represented as italicized words — such as: *Analysis Adsorptive Properties*, *Free Space*, and *Sample Tube*.

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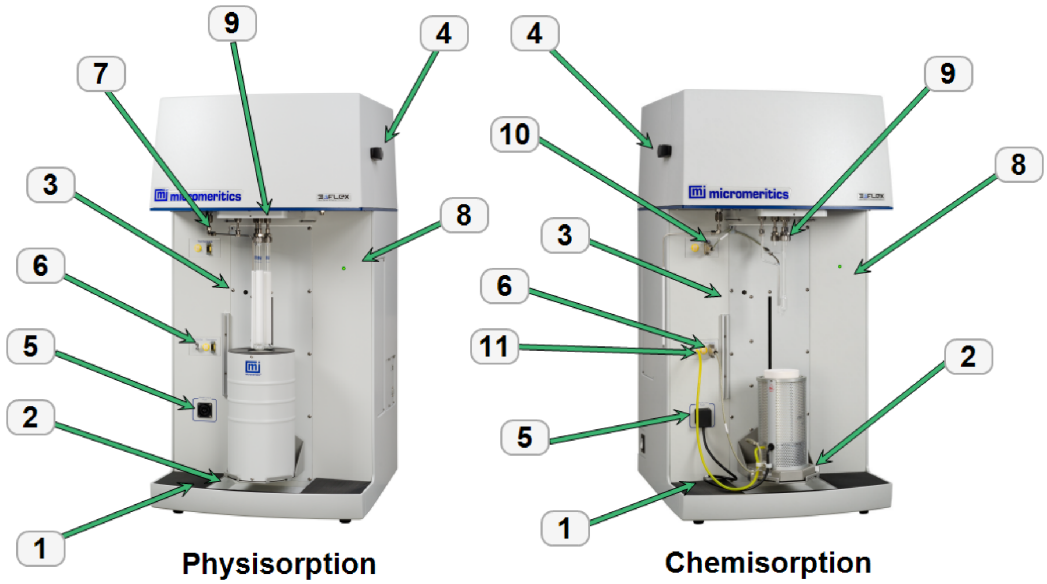
1 About the 3Flex Analyzer

Front Panel



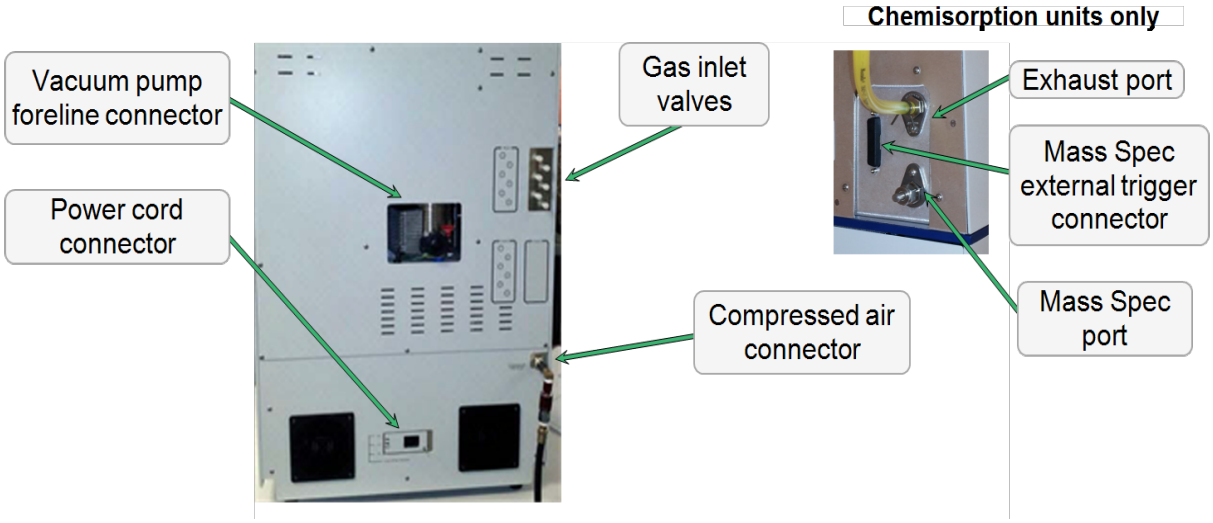
Front Panel Components

Component		Description
1	Access panels to reset buttons	Lift the pad and panel to access the reset buttons for the mantle, heater, and transformer.
2	Elevator	The elevator raises and lowers automatically when the analysis is started and completed. During analysis, the elevator *optionally* lowers after the free-space measurement to allow evacuation, then raises and continues the analysis.
3	Elevator reset button	Resets the elevator in case of failure. The message *Elevator Circuit Breaker Open* on the analyzer schematic indicates this reset button should be pushed.
4	Manifold compartment cover latch	Holds the removable manifold compartment cover.
5	Mantle / Furnace power connector	Power connector for the mantle or furnace.
6	Mantle / Furnace thermocouple connector	Connector for the thermocouple.
7	P_0 tube	For measuring the saturation pressure.
8	Power indicator light	Blinks when power is applied to the analyzer; illuminates when analysis program is initiated and ready for operation.

Front Panel Components (continued)

Component		Description
9	Sample ports	For installing up to three sample tubes.
10	Thermocouple	For chemisorption samples
11	Cooling gas port	Chemisorption exhaust port

BACK AND SIDE PANELS



Back and Side Panel Components

Component	Description
Compressed air connector	For compressed air supply for the pneumatically actuated, hard seal valves.
Ethernet port *(not shown)*	Located on the side panel. Port for an Ethernet cable allowing communication between the analyzer and the computer.
Exhaust port	The top port is to vent flowing chemisorption gas. The bottom port is used for the optional Mass Spec.
Gas inlet valves	Inlet valves 1-6 for analysis gases.
Mass Spec external trigger signal	For chemisorption: For connecting the external trigger signal for the optional Mass Spec.
Mass Spec exhaust port	For chemisorption: For connecting the exhaust line for the optional Mass Spec.
Power switch *(not shown)*	Located on the side panel. For turning the analyzer on and off.
Power cord connector	For setting the power voltage and connecting the analyzer to the power supply.
RS-232 connector *(not shown)*	Located on the side panel. Used to connect the Smart VacPrep.
Vacuum pump foreline connector	For attaching the dry diaphragm roughing vacuum pump hose.

EQUIPMENT OPTIONS

Option	Description
Vacuum Pump	The analyzer requires a dry roughing vacuum pump for sample analysis. Appropriate vacuum pumps are available from Micromeritics.
Vapor Adsorption Option	Vapor adsorption allows analyses with hydrocarbon vapors or water vapor. The analyzer allows for dosing from one dedicated sample port to the other two sample ports. A vapor adsorption option provides for dosing from a reservoir attached to the Psat port to all three sample ports. The vapor option includes a stainless steel chamber with a hard seal, manual cutoff valve to be attached in place of the Psat tube, and a heating mantle to control the temperature of the chamber at an operator-specified temperature between ambient and 43 °C.
Micropore Option	Each port on the standard analyzer can be upgraded individually for high quality micropore analyses with 10 torr and 0.1 torr transducers on each micropore port. Any remaining ports continue to operate in standard mode. The micropore option is required to run krypton.
Chemisorption Option	The Chemisorption option includes a Mass Flow Controller for precise flowing preparation of samples, a furnace to control sample temperature from ambient to 1100 °C, quartz flow-through sample tubes, an exhaust port for venting hazardous gases, a Mass Spec port and electronic trigger signal, and six additional gas inlets for a total of twelve. The single chemisorption port is port number 2. The Micropore Option is required for the chemisorption port.

Micropore Unit	Micropore port number
One micropore unit	2
Two micropore unit	1 and 2
Three micropore unit	1, 2, and 3

DEGASSER OPTIONS

Degasser Option	Description
Smart VacPrep	The Smart VacPrep prepares samples by heating and evacuation. It contains six sample ports in which up to five temperatures, ramp rates, and soak times per sample are individually controlled by the analyzer program so that all degas information is integrated into the sample data file for future reference. Samples can also be prepared, started, and completed independently. There is no need to wait for samples on other ports to finish. Convenient front panel buttons allow QuickStart operation with pre-programmed conditions. Up to three additional Smart VacPrep instruments can be connected to one computer permitting 24 preparation ports to be used.The Smart VacPrep is the recommended degassing unit.
FlowPrep 060	The FlowPrep applies both heat and a stream of inert gas to the sample to remove adsorbed contaminants from the surface and pores in preparation for analysis for up to six samples. Choose the temperature, gas, and flow rate best suited for your sample material. The FlowPrep is an independent unit and not controlled by the analyzer.
SmartPrep	The SmartPrep passes flowing-gas over the sample at elevated temperatures. It contains six sample ports in which temperature, ramp rates, and soak times are individually controlled by the analyzer program so that all degas information is integrated into the sample data file for future reference. It contains 2 serial ports, one for connecting to the computer and the other for connection of up to 3 additional SmartPrep instruments permitting 24 preparation ports to be used.
VacPrep 061	The VacPrep offers two methods for removing contaminants. In addition to flowing gas, it provides vacuum to prepare samples by heating and evacuation of up to six samples. This combination provides preparation method options best suited to your material or application. Needle valves are also provided for introducing the vacuum slowly to prevent fluidization of samples.The VacPrep is an independent unit and not controlled by the analyzer.

GAS REQUIREMENTS

Compressed gases are required for analyses. Gas cylinders or an outlet from a central source should be located near the analyzer.

Appropriate two-stage regulators which have been leak-checked and specially cleaned are required. Pressure relief valves should be set to no more than 30 psig (200 kPag). All gases should be of a purity of 99.999% or better.

POWER THE ANALYZER ON AND OFF

If a Smart VacPrep is used, it is recommended that the power to the Smart VacPrep remain ON when the analyzer is powered on. If it does become necessary to power off the Smart VacPrep, exit the analyzer program first. Restart the analyzer program, then power on the Smart VacPrep.

- Power on the equipment in the following order:
  1. Computer, monitor, and printer
  2. External vacuum pump
  3. Analyzer
  4. Degasser

Always exit the analysis program before powering off the computer. Failure to do so could result in loss of data.

- Power off the equipment in the following order:
  1. Computer, monitor, and printer
  2. Analyzer *
  3. External vacuum pump
  4. Degasser

  * If an analysis is in progress when closing the application, the following message is displayed:

  2459 - An Instrument is busy. A delay in restarting this application could result in loss of new data. Continue program exit? Yes / No

  Yes. Closes the program. The analysis continues and data continue to be collected. The data will be restored when the application is restarted. Reports queued in the print manager will print. If a power failure occurs and an uninterruptible power supply (UPS) is not attached to the analyzer, the data collected after exiting the analysis program are lost.

  No. The program remains open and the analysis continues to run.

THE BASIC WORKFLOW

This section outlines the basic steps for a quick start to using the analyzer. Refer to each link for additional details on performing the individual steps.

2. Set up the Sample and Parameter files
   a. Edit / create the default method (optional). See *Methods on page 2 - 14*.
   b. Create the sample file.
   c. Create the parameter files.
2. Degas the Sample

   Degassing can be performed either in situ or by using a separate degasser unit such as a Smart VacPrep or SmartPrep.

   - See .*Degas in Situ on page 5 - 4*.
   - See the Smart VacPrep Operator Manual part number 067-42800-01
   - See *Degas on the SmartPrep on page 5 - 9*.
3. Prepare for Analysis

   There are several steps required prior to running an analysis:

   a. See *Step 1 - Clean and Label Sample Tubes on page 6 - 2*
   b. See *Create Sample Files on page 3 - 1*.
4. Run the Analysis

   There are several types of analyses that can be performed.

   - See *Perform an Analysis Sequence on page 6 - 16*
   - See *Perform a Blank Analysis on page 6 - 18*
   - See *Perform a Reference Analysis on page 12 - 18*
   - See *Perform a Sample Analysis on page 6 - 22*
   - See *Perform a Vapor Analysis on page 6 - 26*
5. Get Reports

   There are numerous reports that can be run on an analysis with a *Completed* status.

   - See *About Reports on page 7 - 1*
   - See *Interactive Reports on page 7 - 8*
   - See *MicroActive Reports on page 7 - 9*

SPECIFICATIONS FOR THE 3FLEX

Specification	Description
Adsorptive Gas Inputs	6, expandable to 12
Control of cryogen level on sample tube	Isothermal jacket
Degas	3 in situ, optional Smart VacPrep provides 6 additional degas ports
Dewar	3.2 L capacity, > 80 hrs (single tube, no isothermal jacket)
Electrical	**Voltage.** 100 / 115 /230 VAC **Frequency.** 50 / 60 Hz
Gas Inputs	6
Heating Mantle	Temperature to 450 °C
Manifold outgas rate	< 0.1 µm/min
Minimum measurable surface area	0.1 m^2/g
Sample Analysis Ports	1, 2, or 3 micropore capable ports 1 chemisorption capable port
Sample Tubes	Metric, flat bottom, 9 and 12 mm
Transducers	10 mmHg, ± 0.12% of reading accuracy screen resolution 0.0001 mmHg 0.1 mmHg, ± 0.15% of reading accuracy screen resolution 0.000001 mmHg

Specification	Description
Vacuum System	Turbo molecular drag pump in series with four-stage diaphragm pump **Pumping Speed:** 53 L/sec (hydrogen) 61 L/sec (nitrogen) **Ultimate Vacuum:** 3.75×10^{-10} mmHg **Vacuum Gauge:** Dual Cold Cathode / microPirani gauge
Computer	
Windows 7 Professional or higher operating system is recommended for the best user experience. If the computer is to be connected to a network, a second Ethernet port on the computer must be used for that purpose. **All users of the application will need Read / Write permission to all directories and subdirectories where the application is installed.** *In keeping with a policy of ongoing product improvement, specifications are subject to change without notice.*	

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2 About the Software

The analyzer software allows other computer programs to run while an automatic operation is in progress. The *Help* menu provides access to this operator manual and tutorials on using the software.

The MicroActive feature offers a Windows interface with an easy way to collect, organize, archive, and reduce raw data and store sample information files for later use. Scalable and editable graphs, and cut-and-paste graphics, are easily generated. Customized reports can be generated to screen, paper, or exported for use in other programs. There are two report functions:

- Advanced reports (using the Python module)
- MicroActive reports

Report options can be specified when creating the sample information file. When running an analysis, data gathered during the analysis process are compiled into the predefined reports. Reports can also be defined and generated after an analysis has been run. Each selected report is displayed on its own tab and reflects data collected during the analysis.

Software Setup

If a software update is required for the analyzer to support the Smart VacPrep, the update can be downloaded from the internet at this URL:

http://www.micromeritics.com/Smart-VacPrep-Software.aspx

If the computer is to be connected to a network, a second Ethernet port on the computer must be used for that purpose.

The *Setup* program is located on the installation CD. It is used to:

- Reinstall the software version [*n*]
- Add an analyzer
- Move an analyzer
- Remove an analyzer
- Change analyzer setup
- Reinstall calibration files for an analyzer
- Import an analyzer from a previous installation on this PC
- Add or modify a Smart VacPrep

To access the *Setup* program:

1. Insert the *Setup* CD into the CD drive.
2. Locate and double click the *Setup.EXE* file.

If the IP address needs to be changed on the computer connected to the analyzer, refer to the computer's operating system manual or the internet for instructions. The IP address for the computer and the IP address specified in the setup program must match. The IP address must be 192.168.77.100.

SOFTWARE UPDATES

When performing a software update, existing data files are not overwritten. There are three types of subsequent installation:

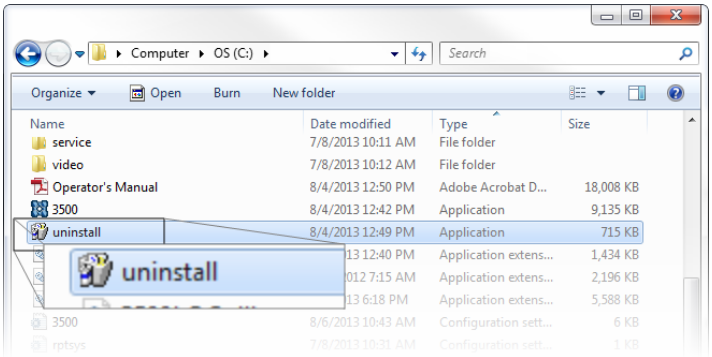
- a later version than the current installation
- the same version as the current installation
- an earlier version than the current installation

1. Insert the setup CD into the CD-ROM drive. The setup program starts automatically. If the program does not start automatically, navigate to the CD drive, locate and double click the *setup.exe* file.
2. Select one of the installation options, then follow the prompts on the screen.

UNINSTALL THE SOFTWARE

When the software is uninstalled, only the files required to run the application are removed. Parameter files, sample files, reports, calibration files, and data files are not removed.

To uninstall the software, locate and double click the *uninstall.EXE* file located in the software installation directory, then follow the prompts on the screen.



MENU STRUCTURE

All program functions use standard Windows menu functionality. The title bar contains a *Unit Number*. If multiple units (analyzers) are installed, ensure the appropriate unit is selected before continuing.

Main Menu Bar Options

Option	Description
File	Use to manage files.
Unit [*n*]	Use to perform analyses, calibrations, and other analyzer operations. A *Unit [n]* displays on the menu bar for each analyzer attached to the computer.
Smart VacPrep	Use to access the *Unit [n]* menu for each Smart VacPrep for starting degas operations, calibrations, and other operations. Also used to install or remove a Smart VacPrep degasser. The Setup program can also be used to install a Smart VacPrep. See ***Software Setup on page 2 - 1***.
Options	Use to edit the default method, specify system configuration, and change presentation options.
Window	Use to manage open windows and display a list of open windows. A checkmark appears to the left of the active window.
Help	Provides access to the operator manual, online instructional tutorials, the Micromeritics web page, the analyzer web page, and information about the analyzer.

COMMON FIELDS AND BUTTONS

The fields and buttons in the following table are located in multiple windows throughout the analyzer application and have the same description or function. Fields and button descriptions not listed in this table are found in tables in their respective sections.

Common Fields and Buttons Table

Field or Button	Description
Add Log Entry	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Autoscale checkbox	When enabled on report parameters windows, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y- ranges will be set so that all the data is shown. If *Autoscale* is not selected, the entered range is used.
Axis Range	On report parameters windows, the *From* / *To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and / or y-axes.
Bar Code	Enter bar code reader information if a bar code reader is connected to the computer's USB port. If a bar code reader is not used, this alphanumeric field can be used to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Browse	Searches for a file. Select a file from the *Name* column or from the library, then click **Open**. Alternatively, double click the file name to open (or import) the file.
Cancel	Discards any changes or cancels the current process.
Close	Closes the active window.
Close All	Closes all active windows. If changes were made and not yet saved, a prompt displays for each changed file providing the option to save the file.
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Delete	When working with report parameters, **Delete** removes the selected report. Deleted reports will have to be regenerated if deleted in error.
Destination group box	• **Preview.** Previews the predefined report on the screen. • **Print.** Sends the report to the default printer. • **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected. • **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report sys-

Common Fields and Buttons Table (continued)

Field or Button	Description
	tem (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
Edit	When working with report parameters, highlight the item in the *Selected Reports* list box and click **Edit** to modify the report details.
Exit	If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the application without saving the changes.
Export	Exports isotherm data in a sample information file as a .REP, .TXT or .XLS file. When saved to a file, the data can be imported into other applications.
File name text box	Select a file from either the *Name* column or from the library. The file name displays in the *File name* text box. Click **Open** or double click the file name to open the file. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
From / To text boxes	When working with report parameters windows, enter the *From* and *To* range for x- and / or y-axes.
List	Provides the option to create a list of sample or report options file information, for example, file name, date / time the file was created or last edited, file identification and file status.
Name column	A list of files in the selected directory or library.
Next	Click to move to the next window or next step.
OK	Saves and closes the active window.
Open	Opens the selected file. Alternatively, double click the file name in the *Name* column to open the file.
Prev	Click to move to the previous window.
Preview	Previews predefined reports. Click the tabs at the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled.
Print	Sends the report to the selected destination (screen, printer or file).
Remove	Click to remove an item from the list.
Replace	Click to select another file where the values will replace the current file's values.
Replace All	Click to select another .SMP file where the values will replace all values for the active Sample Information file. The original file will remain unchanged.
Report	Click to display a window to specify report output options. • **Start Date.** Displays a calendar to select the start date for the report.

Common Fields and Buttons Table (continued)

Field or Button	Description
	• **Preview.** Previews the predefined report on the screen. • **Print.** Sends the report to the default printer. • **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected. • **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
Save	Saves changes to the active window.
Save As	Saves a file in the active window under a different file name.
Start	Starts the report, test, analysis, or operation.
Table buttons	Use to modify the table contents. • **Insert.** Inserts one row above the selected row. • **Delete.** Deletes the selected row. • **Clear.** Clears all table entries and displays only one default value. • **Append.** Inserts one row at the end of the table.

FILE STATUS, DESCRIPTION, EXTENSION, AND LOCATION

In the *File Selector* window, the *Mic Description* column and the *Mic Status* column display file description and file status. The *File Selector* incorporates standard Windows features for resizing windows, reordering and repositioning columns, and right clicking an entry to display a menu of standard Windows functions.

File Status and Description Table

File Status	Description
Analyzing	Sample information files that are currently being used for analysis.
Complete	Sample information files used in an analysis that has been completed.
Entered	Sample information files containing manually entered data.
No Analysis	Sample information files which have not been used to perform an analysis.
Prepared	Sample information files that have been used in an automatic degas operation but have not been analyzed. This status is applicable only if using the Smart VacPrep or SmartPrep degasser.
Preparing	Currently being used in an automatic degas operation. This status is applicable only if using the Smart VacPrep or SmartPrep degasser.

File Type, Extension, and Location Table

File Type	File Name Extension	Default Location
Alpha-s curve	.ALS	Param Directory
Adsorptive properties	.ADP	Param Directory
Analysis conditions	.ANC	Param Directory
Degas conditions	.DEG	Param Directory
Heat of Adsorption Report	.HOA	Param Directory
Methods	.MTH	Methods Directory
Report options	.RPO	Param Directory
Sample information	.SMP	Data Directory
Sample tube properties	.STB	Param Directory
Thickness curve	.THK	Param Directory
The following file types are available when printing or exporting reports:		
Report	.REP	
Spreadsheet	.XLS	
Unicode	.TXT	
Portable document format	.PDF	

APPLICATION SHORTCUTS

MENU SHORTCUTS

Shortcut menus are available for:

- the analyzer schematic when manual control is enabled
- onscreen graphs and tabular reports.

KEYBOARD SHORTCUTS

Shortcut keys can be used to activate some menu commands. Shortcut keys or key combinations (when applicable) are listed to the right of the menu item.

Certain menus or functions can also be accessed using the **Alt** key plus the underlined letter in the menu command. For example, to access the File menu, press **Alt + F**, then press the underlined letter on the submenu. For example, **Alt + F** opens the File menu, then press **O** to access the *File Selector* for opening files.

If the underscore does not display beneath the letter on the menu or window, press the **Alt** key on the keyboard.

Keyboard Shortcut Table

Field or Button	Description
Alt + *[Unit n]*	Opens the *Unit [n]* menu.
Alt + F	Opens the *File* menu.
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Alt + H	Opens the *Help* menu.
Alt + I	Opens the *Options* menu.
Alt + R	Opens the *Reports* menu.
Alt + V	Opens the *Smart VacPrep* menu for adding or removing a Smart VacPrep.
Alt + W	Opens the *Window* menu.
Shift + F9	Opens the shortcut menu of (1) selected component on analyzer schematic when manual control is enabled or (2) onscreen reports.
Ctrl + N	Opens a new sample file.
Ctrl + O	Opens the *File Selector* window.
Ctrl + P	Opens the *File Selector* to start a report from a selected .SMP file.
Ctrl + S	Saves the open file.

Keyboard Shortcut Table (continued)

Field or Button	Description
F1	Opens the online help operator manual.
F2	Opens the *File Selector* window.
F3	When in the *File Selector* window, opens the file search box.
F4	When in the *File Selector* window, opens the address bar.
F6	Cascades open windows.
F7	Tiles open windows.
F8	Opens the *File Selector* to start a report from a selected .SMP file.
F10	Opens the *Heat of Adsorption* window.

If using the Smart VacPrep, reference the Smart VacPrep Operator Manual (part number 067-42801-01) for additional information. The Smart VacPrep Operator Manual is available in the Online Help and at the end of this operator manual.

OPTION PRESENTATION

Options > Option Presentation

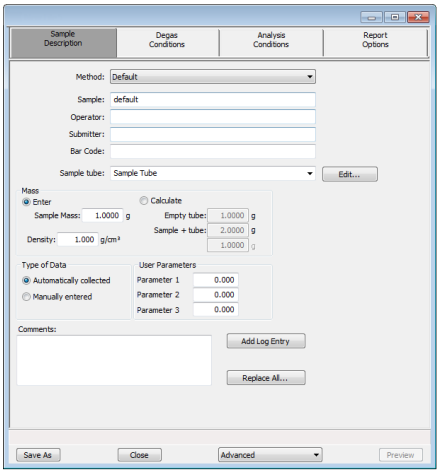
Use to change the way sample files and parameter files display: *Advanced*, *Basic*, or *Restricted*. Each display option shows sample information and options differently.

Presentation Display Table

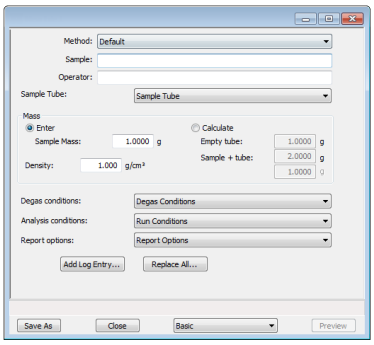
Presentation Display	Description
Advanced	Displays all parts of sample information and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Restricted	Displays the sample information file in a single window similar to the *Basic* display option with certain functions disabled. A password is set when the *Restricted* option is selected. That same password must be entered to change to the *Basic* or *Advanced* display option. This display type is typically used in laboratories where analysis conditions must remain constant — such as the pharmaceutical industry. The *Advanced* option is not available at the bottom of the window when using the *Restricted* display option.

- **Show Degas Conditions.** When enabled, displays the *Degas Conditions* tab when using *Advanced* option presentation and the Degas Conditions drop-down list when using *Basic* or *Restricted* option presentation.
- **Check Shield.** When enabled, checks to ensure the shield is in place around the dewar or furnace prior to starting an analysis. If this option is selected and the dewar or furnace shield is not in place prior to starting an analysis, a warning message displays on the analyzer schematic window. An entry is made in the analyzer log regardless of operator choice.

Specify or change the default option presentation by selecting ***Options > Option Presentation,*** or select *Basic* or *Advanced* from the drop-down list at the bottom of the window.



Advanced presentation option



Basic / Restricted presentation option

LIBRARIES

The library provides an easy way to locate and open specific analyzer files. The library is located within the *File Selector* window and can be viewed only within the application.

1. To locate and open a sample information file, go to ***File > Open***.
2. Click the *Sample Information* library folder on the left navigation bar.
3. Select the .SMP file on the pane on the right side of the window, then click **Open**.

See *Manage Libraries below*.

MANAGE LIBRARIES

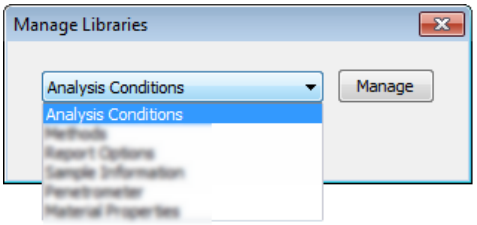
Options > Manage Libraries

This feature is available only for Windows 7 and higher operating systems.

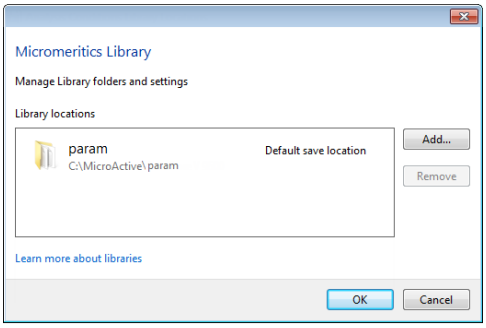
The library gathers sample and parameter files that are stored in multiple locations — such as folders on a C: drive, a network location, a connected external hard drive, or a connected USB flash drive — providing instant access at once to all of those files. Even though libraries do not store actual sample and parameter files, folders can be added or removed within each library.

One library can include up to 50 folders. Other items such as saved searches and search connectors cannot be included.

1. To manage folders in a library, go to ***Options > Manage Libraries***. Select the library to modify from the drop-down list, then click **Manage**.



- To add a folder to the library, click **Add** to browse and locate a folder.



- Select the folder, then click **Include folder**.
- To remove a folder, select the folder from the library locations box, then click **Remove.**

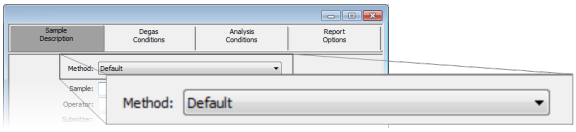
> When removing a folder from a library, the folder and its contents are not deleted from the original file storage location. However, when deleting files or folders from within a library, they are deleted from their original file storage location.

2. Click **OK** when done.

METHODS

A *Method* determines the default sample identification format and sequence number. A *Method* is a template of specifications that go into a newly created sample file. It allows for the definition of complete sets of parameters for each type of sample commonly analyzed, so that only a single selection is required for each new sample file created.

The *Method* drop-down list displays only those methods applicable to the open sample file type.



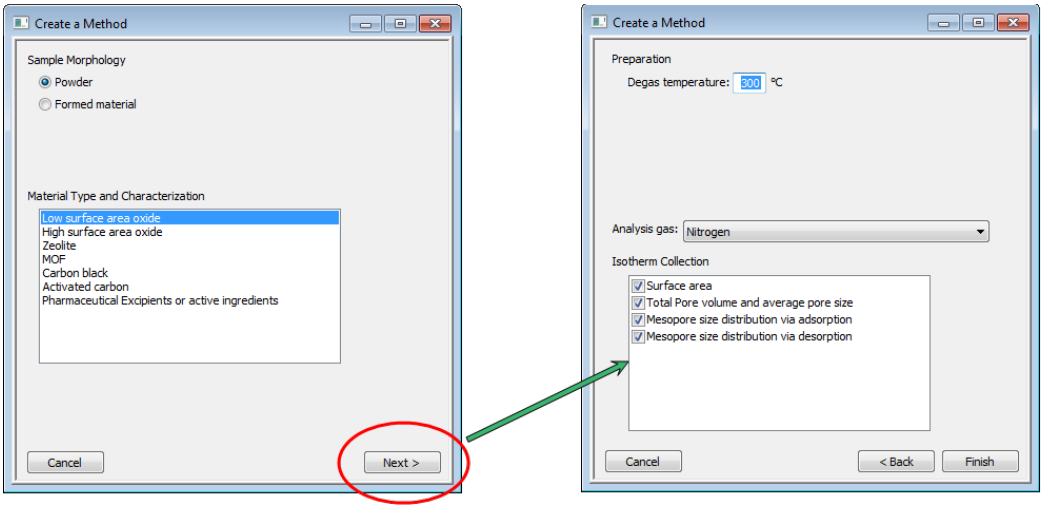
Default Method Files

Default Method Selected	Default File Modified
Physisorption	3500.SMP
Chemisorption	3500Chemi.SMP

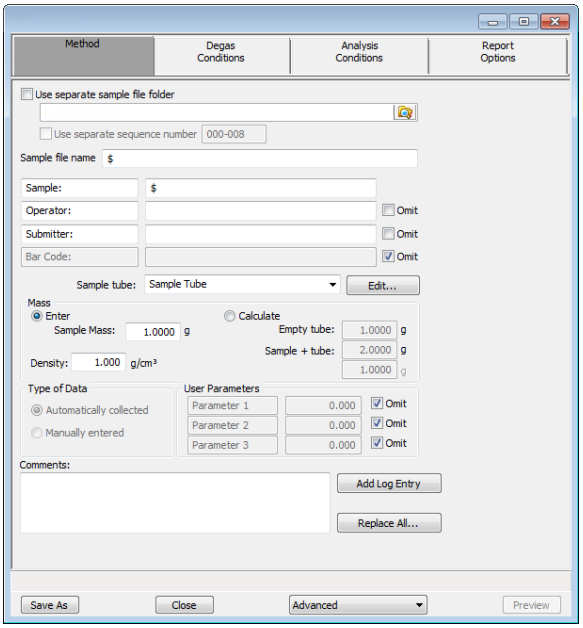
CREATE A NEW METHOD

File > New Method

1. On the *Create a Method* window, select the *Sample Morphology* to be used and the *Material Type and Characterization*, then click **Next**.



2. Enter a *Degas temperature*, then select an *Analysis gas* from the drop-down list.
3. Select the applicable isotherm collections. The *Isotherm Collection* options determine the pressures of the data points measured in the analysis. Click **Finish** to close the wizard and open an editor for the new method.
4. On the *Method* tab, if files created using this method are to be saved in a file folder other than the default, select *Use separate sample file folder*, then click the **Browse** icon to select a folder. The **Browse** icon is enabled only when *Use separate sample file folder* is selected. Select the new folder, then click **OK** on the *Browse for Folder* window.



5. If the file sequence numbers for this method will differ from other methods, select *Use separate sequence number*. This open is enabled only when the *Use separate sample file folder* is selected.
6. In the *Sequence Number* text box, specify an optional default alphanumeric file sequence string. This field must contain a minimum of three numbers. As files are created, this number is incrementally sequenced as a part of the file name.
7. In the *Sample file name* text box, enter an optional default file name. This information will be appended to the sequence number as a part of the file name. The **$** symbol must remain in this field.
8. In the *Sample* field text box, enter a format for the default sample identification. Include the **$** symbol to automatically include the contents of the *Sequence Number* field as part of the sample identification.
9. Enter default *Operator*, *Submitter*, and *Bar Code* identification information in the respective text boxes.

The labels for the *Sample*, *Operator*, *Submitter*, and *Bar Code* fields can be modified by overwriting the labels. These fields can also be omitted from a sample file by selecting the *Omit* checkbox.

10. In the *Sample Tube* drop-down list, select a sample tube. If the required sample tube does not appear in the list, click **Edit** and enter the description and other parameters for this tube. Then go to ***File > Save As > Sample Tube*** to save these values for the next time this sample tube is used.
11. In the *Mass* group box, indicate if mass is to be manually entered by the operator (*Enter*) or calculated by the system (*Calculate*).

12. In the *Type of Data* group box, indicate if the data is to be automatically collected by the system or manually entered by the operator.
13. Enter any pertinent information about the sample information file in the *Comments* text box.
14. Click **Add Log Entry** to enter notes for the analyzer log report. Create entries that cannot be recorded automatically through the software.
15. To auto-populate fields from another .SMP file, click **Replace All**, then select a .SMP file that contains the preferred parameters. Select the file, then click **Load**.
16. Click **Save As**. Select *Methods* in the library and enter a file name for the method in the *File name* text box.
17. Click **Save**.

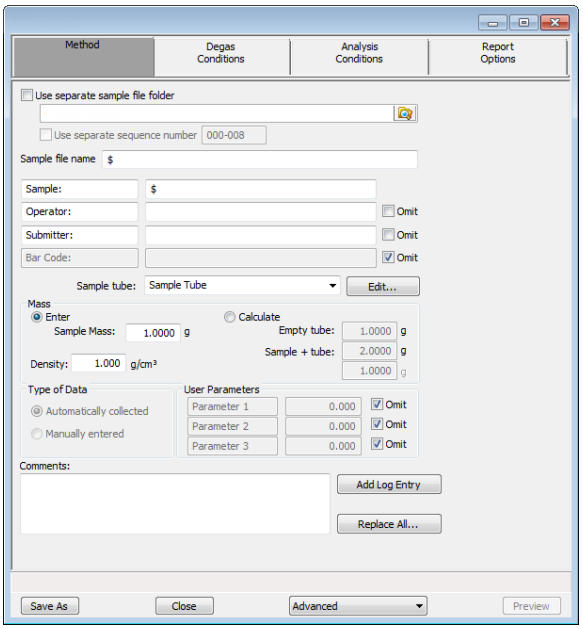
EDIT THE DEFAULT METHOD

Options > Default Method

See *Create a New Method on page 2 - 15*

EDIT A METHOD

File > Open > [Method]



1. In the *File Selector*, select a .MTH file and click **Open**.
2. On the *Method* tab, if files created using this method are to be saved in a file folder other than the default, select *Use separate sample file folder*, then click the **Browse** icon to select a folder. The

Browse icon is enabled only when *Use separate sample file folder* is selected. Select the new folder, then click **OK** on the *Browse for Folder* window.

3. If the file sequence numbers for this method will differ from other methods, select *Use separate sequence number*. This open is enabled only when the *Use separate sample file folder* is selected.
4. In the *Sequence Number* text box, specify an optional default alphanumeric file sequence string. This field must contain a minimum of three numbers. As files are created, this number is incrementally sequenced as a part of the file name.
5. In the *Sample file name* text box, enter an optional default file name. This information will be appended to the sequence number as a part of the file name. The **$** symbol must remain in this field.
6. In the *Sample* field text box, enter a format for the default sample identification. Include the **$** symbol to automatically include the contents of the *Sequence Number* field as part of the sample identification.
7. Enter default *Operator*, *Submitter*, and *Bar Code* identification information in the respective text boxes.

The labels for the *Sample*, *Operator*, *Submitter*, and *Bar Code* fields can be modified by overwriting the labels. These fields can also be omitted from a sample file by selecting the *Omit* checkbox.

8. In the *Sample Tube* drop-down list, select a sample tube. If the required sample tube does not appear in the list, click **Edit** and enter the description and other parameters for this tube. Then go to ***File > Save As > Sample Tube*** to save these values for the next time this sample tube is used.
9. In the *Mass* group box, indicate if mass is to be manually entered by the operator (*Enter*) or calculated by the system (*Calculate*).
10. In the *Type of Data* group box, indicate if the data is to be automatically collected by the system or manually entered by the operator.
11. The optional user-defined fields in the *User Parameters* group box may be used to enter and track information from another analyzer or source, along with other statistical process control (SPC) data.
12. Use the *Comments* text box to enter notes about the Method.
13. After completing the *Sample Description* tab select the parameter tabs across the top portion of the window to edit other sample information file parameters. The saved parameter settings become the defaults for new sample files when this method is selected.
14. Click **Save**, then click **Close**.

CONFIGURE THE ANALYZER

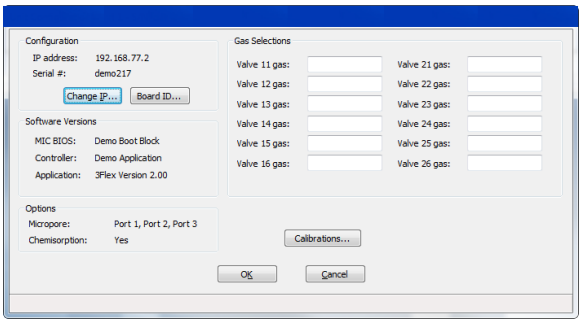
SPECIFY GAS PORTS

Unit [n] > Unit Configuration

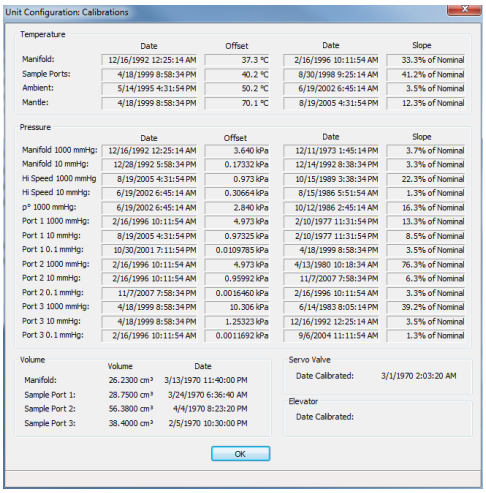
Use to display hardware/software configurations, calibrations, and gas selections of the connected analyzer.

The analyzer has gas inlets for up to six analysis gases (12 with the Chemisorption option). The gases connected to the inlets must be specified in the analysis program. If the gas is changed on one of the inlets, the same change must be made on the *Unit Configuration* window. The analysis software must be kept informed of any changes in gases.

Enter the gas mnemonic in the gas fields to indicate the type of gas connected to that valve.



Unit Configuration Fields and Buttons Table

Field or Button	Description
Calibrations	Displays calibration information for analyzer components.  Unit Configuration: Calibrations Temperature Date Offset Date Slope Manifold: 12/16/1992 12:25:14 AM 37.3 °C 2/16/1996 10:11:54 AM 33.3% of Nominal Sample Ports: 4/18/1999 8:58:34 PM 40.2 °C 8/30/1998 9:25:14 AM 41.2% of Nominal Ambient: 5/14/1995 4:31:54 PM 50.2 °C 6/19/2002 6:45:14 AM 3.5% of Nominal Mantle: 4/18/1999 8:58:34 PM 70.1 °C 8/19/2005 4:31:54 PM 12.3% of Nominal Pressure Date Offset Date Slope Manifold 1000 mmHg: 12/16/1992 12:25:14 AM 3.640 kPa 12/11/1973 1:45:14 PM 3.7% of Nominal Manifold 10 mmHg: 12/28/1992 5:58:34 PM 0.17332 kPa 12/14/1992 8:38:34 PM 3.3% of Nominal Hi Speed 1000 mmHg 8/19/2005 4:31:54 PM 0.973 kPa 10/15/1989 3:38:34 PM 22.3% of Nominal Hi Speed 10 mmHg: 6/19/2002 6:45:14 AM 0.30664 kPa 8/15/1986 5:51:54 AM 1.3% of Nominal p* 1000 mmHg: 6/19/2002 6:45:14 AM 2.840 kPa 10/12/1986 2:45:14 AM 16.3% of Nominal Port 1 1000 mmHg: 2/16/1996 10:11:54 AM 4.973 kPa 2/10/1977 11:31:54 PM 13.3% of Nominal Port 1 10 mmHg: 8/19/2005 4:31:54 PM 0.97325 kPa 2/10/1977 11:31:54 PM 8.5% of Nominal Port 1 0.1 mmHg: 10/30/2001 7:11:54 PM 0.0109785 kPa 4/18/1999 8:58:34 PM 3.5% of Nominal Port 2 1000 mmHg: 2/16/1996 10:11:54 AM 4.973 kPa 4/13/1980 10:18:34 AM 76.3% of Nominal Port 2 10 mmHg: 2/16/1996 10:11:54 AM 0.95992 kPa 11/7/2007 7:58:34 PM 6.3% of Nominal Port 2 0.1 mmHg: 11/7/2007 7:58:34 PM 0.0016460 kPa 2/16/1996 10:11:54 AM 3.3% of Nominal Port 3 1000 mmHg: 4/18/1999 8:58:34 PM 10.306 kPa 6/14/1983 8:05:14 PM 39.2% of Nominal Port 3 10 mmHg: 4/18/1999 8:58:34 PM 1.25323 kPa 12/16/1992 12:25:14 AM 3.5% of Nominal Port 3 0.1 mmHg: 2/16/1996 10:11:54 AM 0.0011692 kPa 9/6/2004 11:11:54 AM 1.3% of Nominal Volume Volume Date Manifold: 26.2300 cm³ 3/13/1970 11:40:00 PM Sample Port 1: 28.7500 cm³ 3/24/1970 6:36:40 AM Sample Port 2: 56.3800 cm³ 4/4/1970 8:23:20 PM Sample Port 3: 38.4000 cm³ 2/5/1970 10:30:00 PM Servo Valve Date Calibrated: 3/1/1970 2:03:20 AM Elevator Date Calibrated: OK
Configuration group box	Displays the IP address used by the analysis program and the serial number of the selected analyzer. • **Change IP**. Click to display the *Unit IP Setup* window. The IP address and Subnet mask assigned during installation display. Do not edit these fields unless instructed by a Micromeritics service representative. • **Board ID**. Click to read the board ID. These parameters cannot be edited.
Gas Selections group box	Enter the mnemonics for the analysis gases attached to inlet valves.
Options	Displays options installed on the analyzer.
Software Versions group box	Displays the software versions of the MIC BIOS, controller, and analysis program.

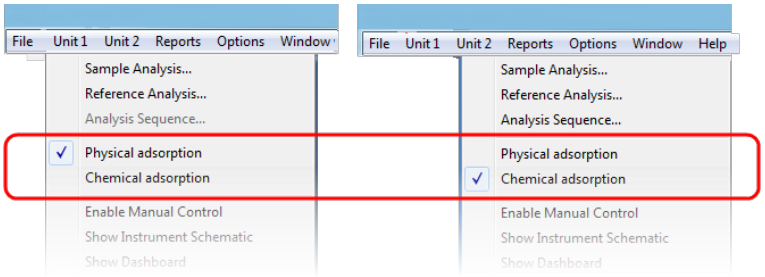
SPECIFY THE TYPE OF ANALYZER

Unit > Physical Adsorption

Unit > Chemical Adsorption

This topic does not apply if physisorption units only are installed.

Up to four analyzers can be connected, and each analyzer may be either physisorption or chemisorption. When multiple units are installed, the application's title bar reflects the number of analyzers installed and the type of analyzer.



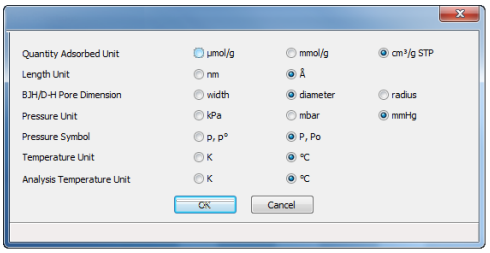
The analysis type affects the appearance of the analysis sample selection window, the analyzer schematic, and the status window. This menu selection is disabled when an analysis is being performed.

When application windows are opened, the title bar reflects both the serial number and the analysis type. The title bar changes when the analysis type changes.

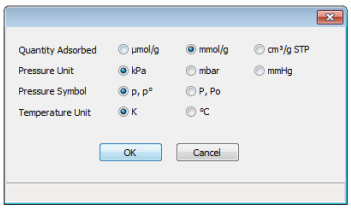
SPECIFY UNIT SELECTIONS

Options > Units

Use to specify how data should appear on the application windows and reports. This menu option is not available if using *Restricted* option presentation.



Physisorption



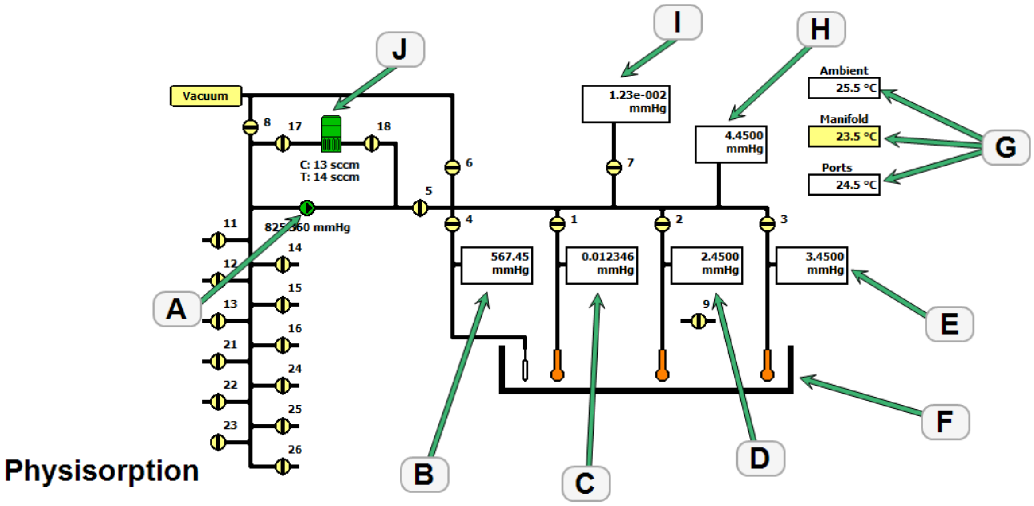
Chemisorption

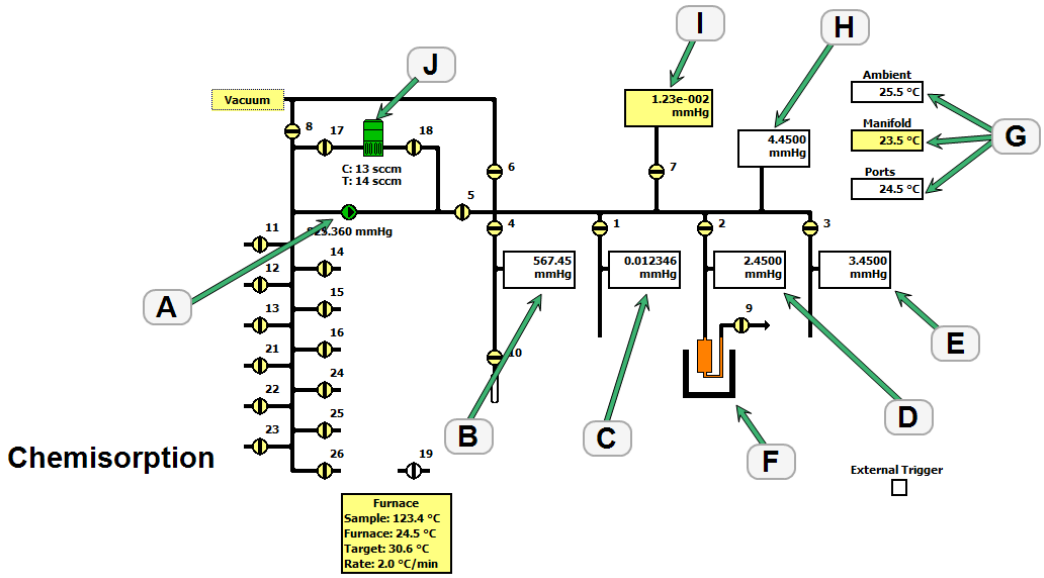
ANALYZER STATUS

SHOW INSTRUMENT SCHEMATIC

Unit [n] > Show Instrument Schematic

Use to display an analyzer schematic. To operate the valves and elevator from this window, manual control must be enabled (***Unit [n] > Enable Manual Control***).





Analyzer Schematic Icon Table

Icon or Symbol	Description
	**Open Valve**. Green indicates an open valve.
	**Closed Valve**. Yellow indicates a closed valve.
	**Servo Valve**. Closed.
	**Servo Valve**. Open.
	**Physisorption Elevator.**
	**Chemisorption Sample Tube** and **Furnace Elevator.** The sample tube icon is white when the sample and furnace temperatures are 50 °C or lower. If either the sample or furnace temperature exceeds 50 °C, the sample tube icon turns orange. Temperature readings and ramp rate are displayed below and to the left of the icon. The furnace icon resides on the elevator.

Analyzer Schematic Icon Table (continued)

Icon or Symbol	Description
	Chemisorption Mass Flow Controller (MFC). Controls the flow of gas into the sample port. The current (C) rate and the target (T) rate are shown to the right of the MFC icon. Applicable only for the gas used in the Flow prep tasks. The mass flow controller constant is preset for gases provided with the application. See *Chemisorption Tasks on page 4 - 14*.
	Physisorption Sample Tube. Cannot be manually controlled.
Shield Removed	A *Shield Removed* warning indicates the safety shield is not in place.

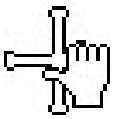
Analyzer Schematic Components Table

Schematic Components	Description
1-3	Sample ports
4	Po port
5	Servo isolation valve
6	Manifold vacuum
7	Vacuum gauge isolation valve
8	Inlet vacuum
9	Exhaust valve for chemisorption only
10	Reference volume (shown in Service Test mode only)
11-16 and 21-26	Inlet valves
A	Servo valve
B	Po pressure
C	Port 1 pressure
D	Port 2 pressure
E	Port 3 pressure
F	Elevator
G	Temperature sensors
H	Manifold pressure
I	Vacuum gauge
J	Mass flow control

Instrument Schematic Shortcut Menus

Each manually controlled schematic component has a shortcut menu displaying the operations available for that particular component. To access the shortcut menu, hover the mouse pointer over the component and right click.

Schematic Shortcuts Table

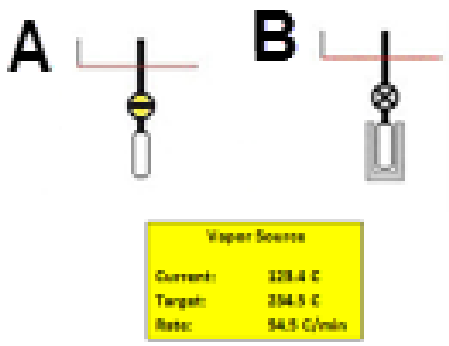
Schematic Shortcut Icon	Available Options:
Valve options 	• **Automatic.** Automatically operates the servo valve during dosing or evacuation. Enter the target pressure. • **Close.** Closes the selected valve. • **Direct.** Used in Service Test mode only under the direction of a Micromeritics service representative. • **Open.** Opens the selected valve. • **Pulse.** Use to quickly turn the valve on and off allowing the operation to proceed in small increments. • **Set.** Use to set the servo valve target pressure and to dose or evacuate.
Elevator options 	• **Set.** For use by Micromeritics Service Technician. • **Raise.** Select *Raise* to raise the elevator. When it is moving, press the keyboard space bar to stop the movement (or right click and select *Stop* from the menu). • **Lower.** Select *Lower* and press the keyboard space bar to lower the elevator. • **Stop.** Stops the elevator from moving.

Temperature control options 	• **On.** Enables the temperature control.
	• **Off.** Disables the temperature control.
	• **Set.** Select to set the following:
	  ○ Enable or disable temperature control
	  ○ Control sample temperature
	  ○ Control furnace temperature
	  ○ Cool the sample to less than 50 °C
	  ○ Set heater power percent

Alternate Schematic Icons

P_0 Port

The following icons will display instead of the P_0 port icon under the following conditions:



A - Reference volume icon. Service test mode only.

B - Vapor source with heating mantle icon. The current and target vapor source temperatures, as well as the rate of temperature increase, are also displayed.

Pressure Transducers

The schematic shows the pressure transducers present in the system.

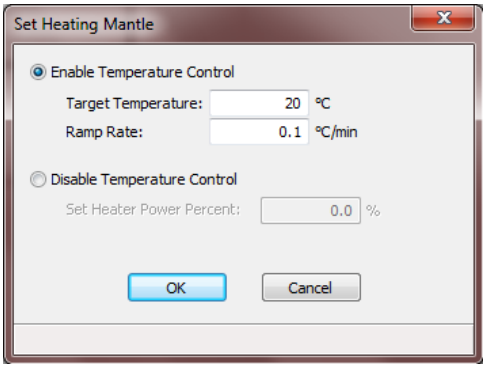
- All systems have 1000 mmHg pressure transducers on the manifold and the analysis ports.
- Optional 10 mmHg pressure transducers on the manifold and one or more analysis ports may be present.
- Optional 0.1 mmHg pressure transducers on one or more analysis ports may be present.

When multiple pressure transducers are present in a port or the manifold, the display automatically changes to show the pressure reading from the lowest range transducer currently on the scale.

Heating Mantle

When samples are being degassed on the analysis ports, the following icons appear beneath the elevator. When the mantle temperature is above 50 °C, the sample tube icons (not shown below) turn orange. If using a GlasCol heating mantle, the sample temperature shows in the yellow *Degas Mantle* box.

Right click the *Degas Mantle* box to set the target temperature or the rate of temperature increase.

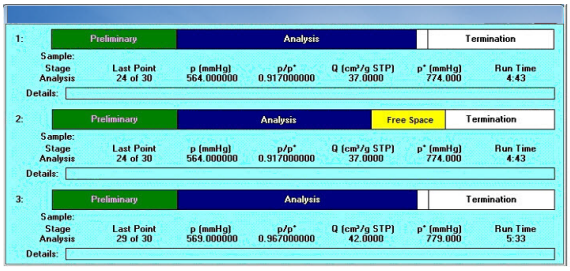


Select *Enable Temperature Control*, then enter both the *Target Temperature* and *Ramp Rate*. The *Set Heater Power Percent* field is enabled in Service Test Mode only.

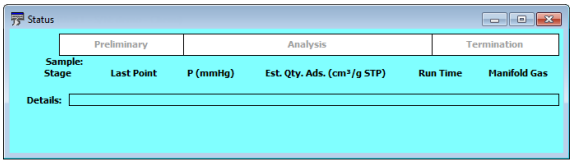
SHOW STATUS

Unit [n] > Show Status

Use to show the current status for each port.



Physisorption



Chemisorption

If multiple units are attached to the computer, select *Show Status* on each *Unit [n]* menu. The status for all units display.

SHOW DASHBOARD

Unit [n] > Show Dashboard

The dashboard displays the following:

- Number of analyses completed and started.
- Number of days until roughing pump maintenance is due
- Manifold outgas rate
- Manifold temperature statistics
- Nitrogen P_0 statistics

Data for the dashboard comes from the logged diagnostic data. The dashboard is automatically kept current as the relevant diagnostic data items are updated. The gauges will be updated even if the dashboard window is not open.

Red numbers on the dashboard require attention. To reset the dashboard numbers, right click on the dashboard setting, then click **Reset**.

Dashboard Gauges and Descriptions Table

Field or Button	Description
Analyses completed / started	Displays N/M where N is the number of analyses that have finished data collection and M is the number of analyses that have been started. Analyses canceled or terminated by errors before the termination stage starts are not counted as completed.
Days until roughing-pump service is due	Annual maintenance is recommended. The number of days until the anniversary of the last pump maintenance is shown. The displayed value is updated at least once per day and when the maintenance time is reset. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.
Manifold outgas rate	Provides the qualitative indication of the outgas rate in the dosing manifold. LED images constitute a bidirectional bar graph of the outgas

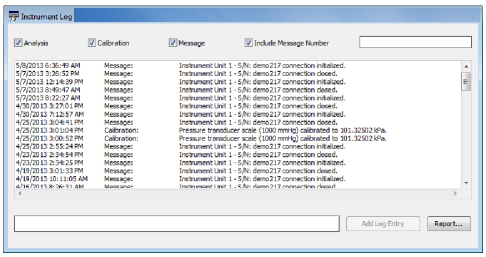
Dashboard Gauges and Descriptions Table (continued)

Field or Button	Description
	rate. The gauge is updated when the *Analysis Manifold Test* is run. See ***Schedule Diagnostic Tests on page 9 - 3*** and ***Start Diagnostic Test on page 9 - 1***. • Three green LEDs are lit if outgas rate is below 30% of outgas rate limit. • At 30%, the left LED turns off. • At 60%, the center LED turns off. • At 90%, three green LED lights turn off and the center yellow LED is turned on. • At 110% and above, only the red LED is lit and attention is required.
Manifold temperature	Displays the statistics of the manifold temperature reading. The mean, the value at two standard deviations, the minimum, and the maximum display.
Nitrogen Po	Displays statistics of the saturation pressures measured with nitrogen gas at liquid nitrogen temperatures. The mean, two-sigma, minimum, and maximum values display. The gauge is updated when a Po is logged with nitrogen as the adsorptive and a bath temperature of 77±2 K.

SHOW INSTRUMENT LOG

Unit [n] > Show Instrument Log

Use to display a log of recent analyses, calibrations, errors, or messages.



Instrument Log Fields and Buttons Table

Field	Description
Analysis / Calibration / Message	Select the logs to display.
Include Message Number	Enter any of the following information in the message text box to generate a log report that includes the entered text: • enter the message number in the text box to view all occurrences of the entered message, • enter an asterisk in the message box to see all numbered messages, or • enter several message numbers separated by commas to include only messages with those numbers. Numbered messages contain more detailed information about analyzer operation.

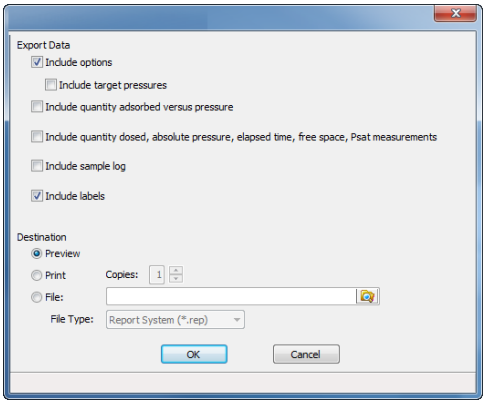
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

EXPORT FILES

File > Export

Provides the option to print the contents of one or more sample files to either the screen, a printer, or to a file. Isotherm data can be exported as a .PDF, .REP, .TXT, or .XLS file format. The type of data to include or exclude can be selected during the export process. When exported to a file, the data can be imported into other software that read .TXT or .XLS file formats.

1. Select one or more files from the library. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
2. Click **Export**.
3. In the *Export Options* window, select the type of data to include in the export file.



Types of data that can be included:

- Options
  - Target pressures
- Quantity adsorbed versus relative pressure
- Quantity dosed, absolute pressure, elapsed time, free space, Psat measurements
- Sample log
- Labels

4. Specify the export destination in the *Destination* section.

- **Preview.** Previews the predefined report on the screen.
- **Print.** Sends the report to the default printer.
- **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected.

- **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.

5. Click **OK**.

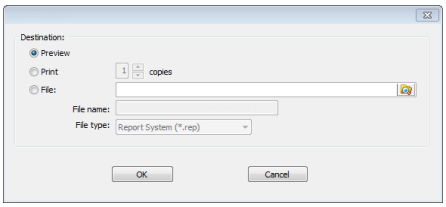
LIST FILES

File > List

Provides the option to create a list of sample file information, for example, file name, date, time the file was created or last edited, file identification, and file status.

The selections in your application may differ slightly from what is displayed on this page however the instructions are the same.

1. Select one or more files from the library. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
2. Click **List**.
3. In the *Report Settings* window, select one of the following:
   - **Preview.** Previews the predefined report on the screen.
   - **Print.** Sends the report to the default printer.
   - **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected.
   - **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.



4. Click **OK**.

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3 About Sample Files

Sample files include the information required by the analyzer to perform analyses and collect data. It identifies the sample, guides the analysis, and specifies report options and may be created in either *Advanced*, *Basic*, or *Restricted* presentation display option.

A sample information file can consist of parameter sets; however, parameter sets can also stand alone. A sample information file may be created either prior to or at the time of analysis.

Parameter sets allow repeated use of the file. For example, if the same analysis conditions exist for multiple analyses, an *Analysis Conditions* file containing the recurring conditions can be created. When the sample file is created, the *Analysis Conditions* file can be selected for the analysis conditions. Once it becomes part of the new sample file, the new file can be edited as needed without affecting the original *Analysis Conditions* file.

The analysis software contains a default method. A method is a template for sample files that contains the parameters to be used for an analysis. When a new sample information file is created, all the parameters are filled with the values in the default Method..

Specify or change the default option presentation by selecting ***Options > Option Presentation,*** or select *Basic* or *Advanced* from the drop-down list at the bottom of the window.

Create Sample Files

Create Sample Files in Advanced Option Presentation

Each analysis must be linked with a sample information file before the analysis can proceed. A sample information file can consist of parameter files; however, parameter files can also stand alone.

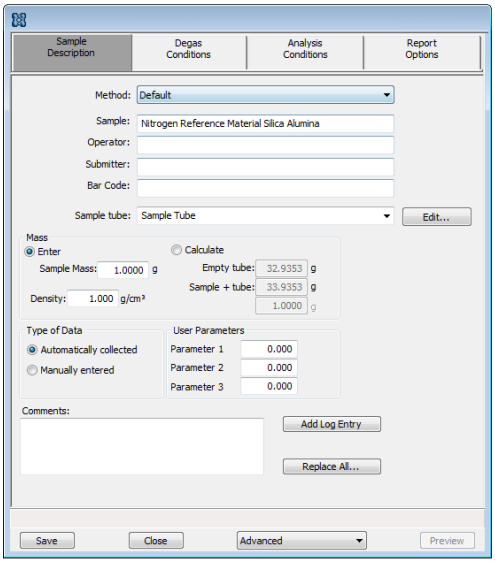
Specify or change the default display option by selecting ***Options > Option Presentation*** or select *Basic / Advanced* from the drop-down list at the bottom of the window.

The values specified in the parameter portions of the default sample file are saved as the defaults for new sample files. To navigate from one set of parameters to another, select the parameter tab across the top of the window.

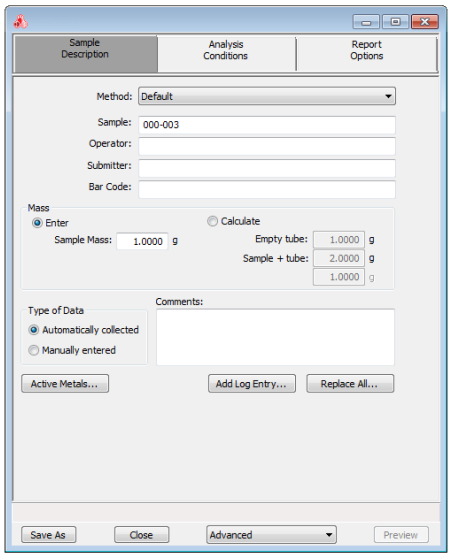
- *Sample Tube* files are edited on the *Sample Description* tab
- *Adsorptive Properties* files are edited on the *Analysis Conditions* tab

For physisorption, the *Degas Conditions* tab displays only if enabled in ***Options > Option Presentation > Show Degas Conditions***.

1. Go to ***Options > Option Presentation > Advanced*** and ensure *Advanced* has a checkmark.
2. To create a new sample file, go to ***File > New Sample***, or go to ***File > Open*** and select a sample file.
3. Select a method from the *Method* drop-down list.

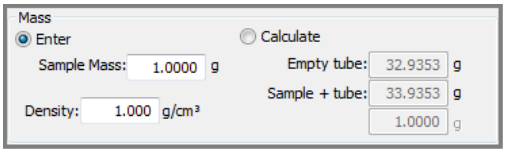


Physisorption

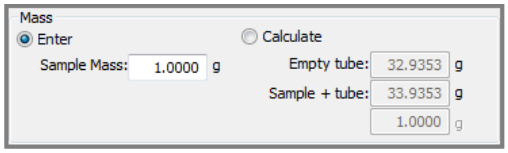


Chemisorption

4. Enter a sample description in the *Sample* text box.
5. Enter *Operator*, *Submitter*, and *Bar Code* identification information in the respective text boxes. This information will display on the *Sample Description* tab of new sample information files. This option may not display (or may have a different field label) if modified in the method from which the sample file was created, either through ***Options > Default Method*** or ***File > Open > Method***.
6. **For physisorption**. In the *Sample Tube* drop-down list, select a sample tube. If the required sample tube does not appear in the list, click **Edit** and enter the description and other parameters for this tube. Then go to ***File > Save As > Sample Tube*** to save these values for the next time this sample tube is used.
7. In the *Mass* group box, indicate if mass is to be manually entered by the operator (*Enter*) or calculated by the system (*Calculate*).



Physisorption



Chemisorption

8. In the *Type of Data* group box, indicate if the data is to be automatically collected by the system or manually entered by the operator.

9. **For physisorption.**The optional user-defined fields in the *User Parameters* group box may be used to enter and track information from another analyzer or source, along with other statistical process control (SPC) data.
10. **For chemisorption**. Click **Active Metals** to display a table of active metals. .
11. Enter any pertinent information about the sample information file in the *Comments* text box. Entered comments are displayed in the report header.
12. Click **Add Log Entry** to enter notes for the analyzer log report. Create entries that cannot be recorded automatically through the software.
13. To auto-populate fields from another .SMP file, click **Replace All**, then select a .SMP file that contains the preferred parameters. Select the file, then click **Load**.
14. After completing the *Sample Description* tab, click the other parameter tabs to edit more sample information file parameters.See *About Parameter Files on page 4 - 1*.
15. Click **Save**, then click **Close**. To save as a different file name, go to ***File > Save As*** and enter a new file name. The file can also be saved as a different file type such as Analysis Conditions, Report Options, etc.

Sample File Fields and Buttons Table

Field or Button	Description
Active Metals (for chemisorption)	Displays a list of active metals. See *Active Metals for Chemisorption on page 3 - 12*.
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Mass group box	If mass = 1, the reported surface area equals the total surface area but it is always shown as m^2/g. If the actual mass is entered, the surface area is reported as m^2/g. Choose whether to enter mass manually or have the system automatically calculate mass. Enter a value for sample mass. Mass can be changed any time before, during, or after analysis. • **Enter.** Enables the *Sample Mass* field. Enter a value for the sample mass. • **Calculate.** Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass: $Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$ • **Density**. (for physisorption.) Value is used for the calculated free space method only. Use 0.000 for a blank analysis.
Method	Select a method from the drop-down list. See*Methods on page 2 - 14*.
Operator	Enter operator identification information. This field label may have been renamed or may not display if modified in ***Options > Default Methods.***

Sample File Fields and Buttons Table (continued)

Field or Button	Description
Sample	Enter a sample description.
Sample Tube	Select a sample tube file from the drop-down list, or click **Edit** to modify or create a new Sample Tube file. See *Sample Tube on page 4 - 23*.
Submitter	Enter submitter identification information. This text box may have been renamed or may not display if modified in ***Options > Default Methods.***
Type of Data group box	• **Automatically collected.** Select if the type of data will be automatically collected by the system while an analysis is running. • **Manually entered.** Use to enter data manually that was collected from another source. If *Manually entered* is selected, the Isotherm Report becomes available in the *Basic/Advanced* drop-down list for pasting or importing data into the file. See *Manually Enter Data on page 3 - 8*.
User Parameters group box (for physisorption)	These fields are primarily used for the SPC (Statistical Process Control) reporting to specify sample characteristics or its manufacturing process but may be used for other data by entering specific analysis conditions or sample criteria. The entered parameters display on the *Summary Report*. This option may not display (or may have a different field label) if modified in the method from which the sample file was created, either through ***Options > Default Method*** or ***File > Open > Method***.

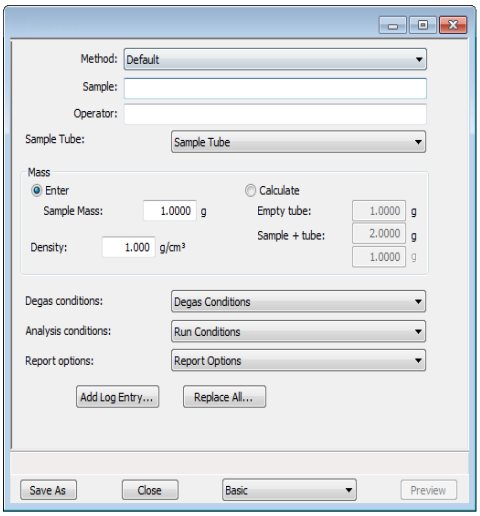
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

CREATE SAMPLE FILES IN BASIC OPTION PRESENTATION

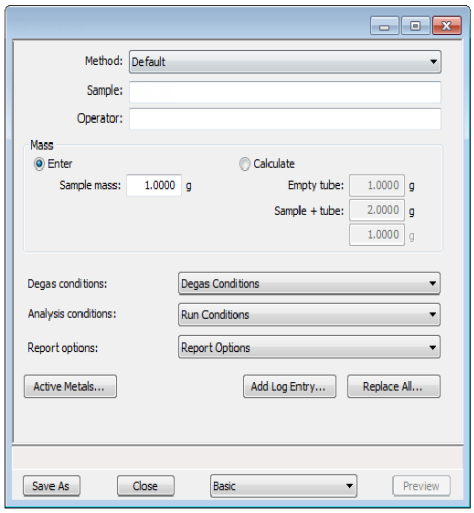
The *Basic* and *Restricted* formats use predefined parameter files to create a sample information file.

> When using the *Basic* option presentation, switch to *Advanced* to edit parameter file values. When using the *Restricted* option presentation, parameter files cannot be edited.

1. Go to ***Options > Option Presentation > Basic*** (or ***Restricted***).
2. To create a new sample file, go to ***File > New Sample***, or go to ***File > Open*** and select a sample file.
3. Select a method from the *Method* drop-down list. See *Methods on page 2 - 14*.

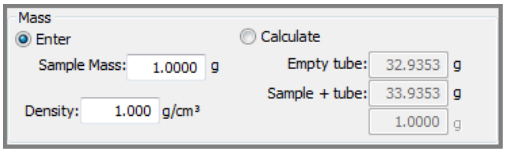


Physisorption

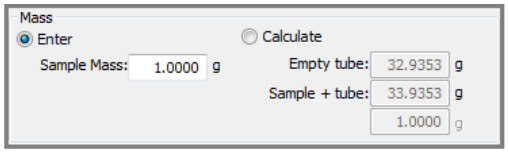


Chemisorption

4. In the *Sample* field, enter a sample description.
5. **For physisorption**. Select a sample tube from the *Sample Tube* drop-down list.
6. In the *Mass* group box, indicate if mass is to be manually entered by the operator (*Enter*) or calculated by the system (*Calculate*).



Physisorption



Chemisorption

7. **For chemisorption.** Click **Active Metals** to display and modify the characteristics of up to 15 elements used for analysis. Click **OK** when done. See *Active Metals for Chemisorption on page 3 - 12*.
8. Click the down arrows to select default parameter files for *Degas conditions*, *Analysis conditions*, and *Report options*. The *Degas Conditions* option will not display if it has not been enabled in ***Options > Option Presentation> Show Degas Conditions***.
9. To auto-populate fields from another .SMP file, click **Replace All** and select a .SMP file that contains the necessary parameters. Select the file and click **Replace**.
10. Click **Add Log Entry** to enter notes for the analyzer log report. Create entries that cannot be recorded automatically through the software, for example, when the port filter was changed.
11. Click **Save**, then click **Close**. The file can be retrieved later from the *Sample Information* folder in the library.

CREATE SAMPLE FILES IN RESTRICTED OPTION PRESENTATION

The instructions for creating a sample file using the *Restricted* option presentation are the same as the *Basic* option presentation.

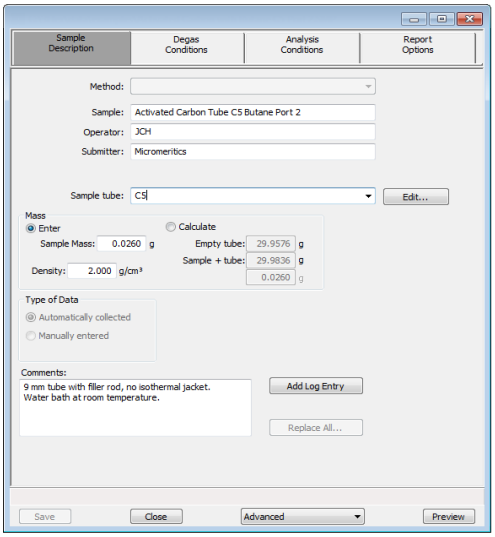
OPEN A SAMPLE FILE

File > Open > [.SMP File]

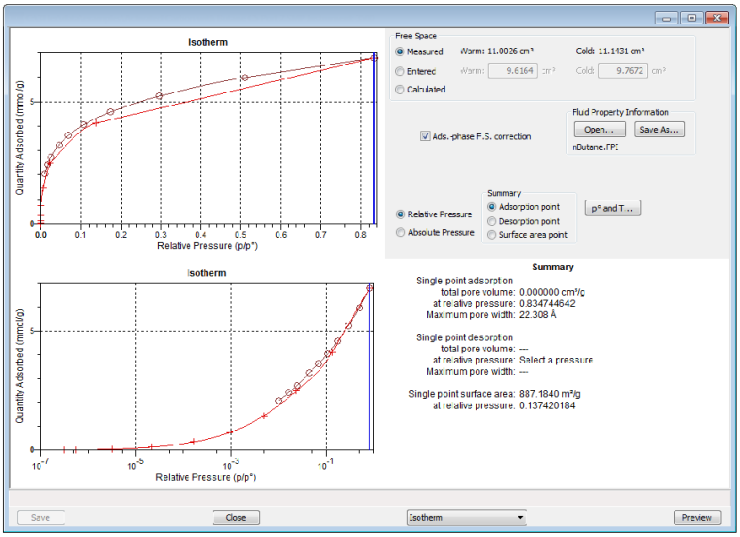
- When working with an existing file, it is recommended that a copy of the file be used rather than the original.
- Columns on the *File Selector* window can be sorted by clicking the column header. To sort the file list by status, click the *Mic Status* column header.

1. Go to ***File > Open.***
2. From the *Sample Information* library folder, select a .SMP file:

File Type	File Status	Displays
Physisorption or Chemisorption	Preparing Prepared No Analysis	Tabbed file editor
	Complete Analyzing Entered	MicroActive report window



Example of tabbed file editor



Example of MicroActive report

MANUALLY ENTER DATA

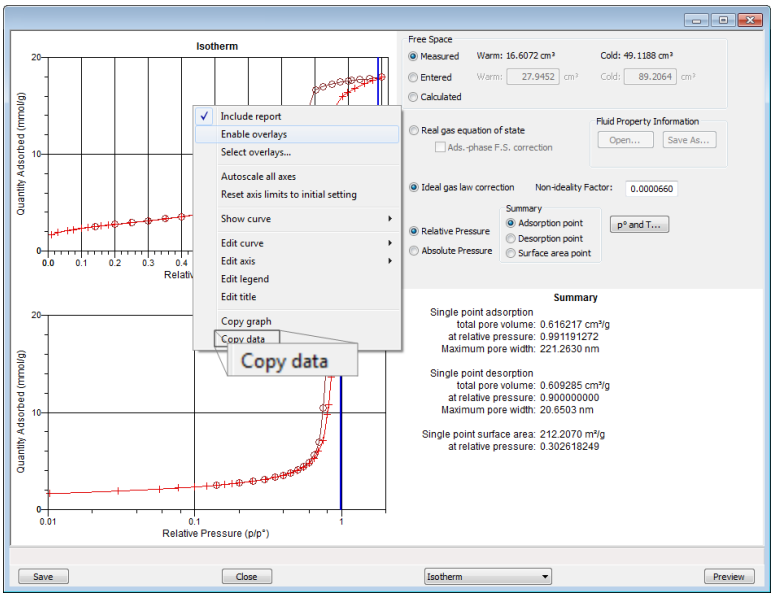
The images shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

This process allows the manual entry of pressure data from a sample file with a *Complete* status. There are two methods for manually entering data into a sample file:

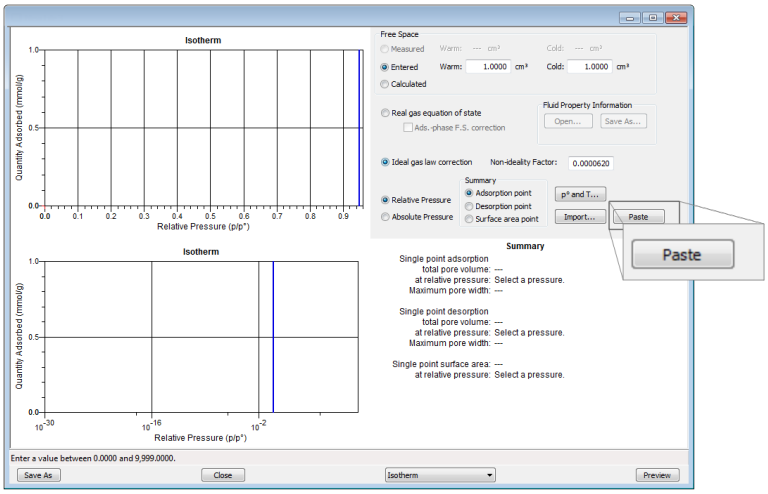
- Copy and paste onto the graph area of the interactive window
- Import data into the interactive window

COPY AND PASTE MANUALLY ENTERED DATA

1. Go to ***File > Open [.SMP file]***, then select the sample information file with a *Complete* status that contains the data to be copied and pasted.
2. Click **Open**. The file will open to the interactive reports window.
3. Right click in the graph area of the interactive reports window, then select *Copy Data*. This will copy the data from the active file to the clipbord.



4. Go to ***File > New Sample***, then open a new sample information file. To save the file as a new file name, go to ***File > Save As***, then enter a new file name in the *File name* text box.
5. On the *Sample Description* tab, select *Manually entered* in the *Type of Data* group box.
6. Click the *Advanced* down arrow at the bottom of the window, then select *Isotherm*.
7. Resize the interactive window to display the **Paste** button.



8. Ensure that all parameter fields are set appropriately, then click **Paste**. The data from the original sample file is pasted from the clipboard and displays in the new sample file.

IMPORT MANUALLY ENTERED DATA

When importing data from an external ASCII text file using the **Import** button on the interactive window, the ASCII text file must use the following rules:

ASCII text file format rules

- Data must be in two columns and separated by a comma or white-space.
- Acceptable column headings are:

For Physisorption or Chemisorption:

- Relative Pressure
- Absolute Pressure (mmHg)
- Absolute Pressure (kPa)
- Absolute Pressure (mBar)
- Quantity Adsorbed (mmol/g)
- Quantity Adsorbed (cm^3/g STP)
- Quantity Adsorbed (cm3/g STP)

Sample Physisorption ASCII Text File

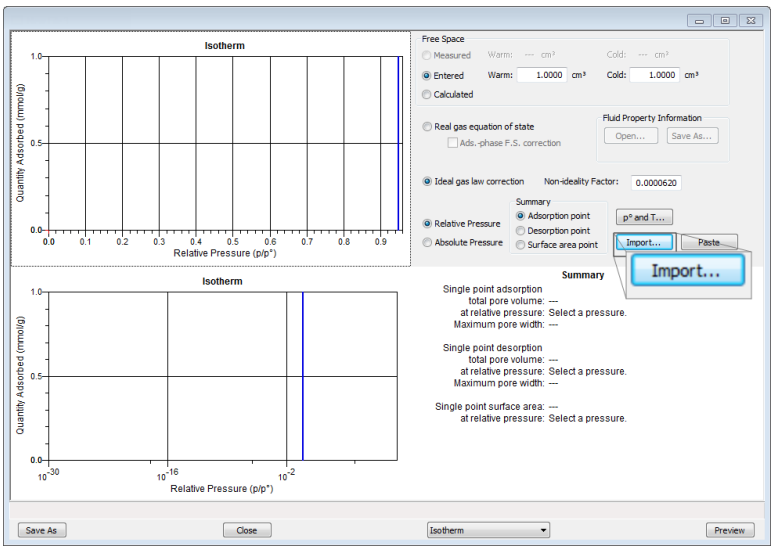
```
Silica Alumina : Adsorption
Relative Pressure        Quantity Adsorbed (cm3/g STP)
0.108629                 50.6657
0.22288                  60.7813
0.339909                 71.3095
0.459512                 84.4172
0.577447                 102.672
0.654583                 121.707
0.760074                 179.096
0.855713                 334.565
0.958511                 394.675
0.996251                 403.793


Silica Alumina : Desorption
Relative Pressure        Quantity Adsorbed (cm3/g STP)
0.996251                 403.793
0.86016                  389.626
0.753567                 256.264
0.664418                 133.099
0.542416                 96.7366
0.422295                 79.7351
```

```
0.346371                    71.5994
0.2519                      62.8256
0.152718                    54.2336
0.103389                    49.5803
```

To import the ASCII text file:

1. Go to ***File > New Sample***, then open a new sample information file. To save the file as a new file name, go to ***File > Save As***, then enter a new file name in the *File name* text box.
2. Click the down arrow at the bottom of the window and select *Advanced*.
3. On the *Sample Description* tab, select *Manually entered* in the *Type of Data* group box.
4. Click the *Advanced* down arrow at the bottom of the window, then select *Isotherm*.
5. Resize the interactive window until the **Import** button displays.

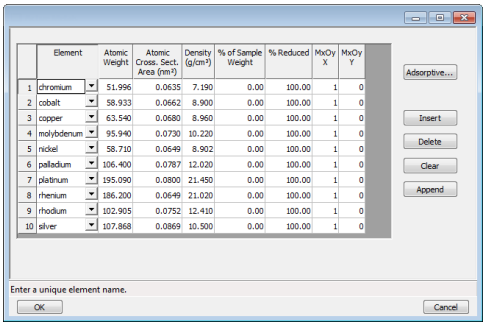


6. Ensure that all parameter fields are set appropriately, then click **Import**.
7. On the *File Selector* window, locate and select the .TXT file, then click **Open**. The data from the original sample file is imported and displays in the new sample file. If an error message appears instead, verify that the .TXT file format is correct.

ACTIVE METALS FOR CHEMISORPTION

See *Atomic Weights and Cross Sectional Areas on page A - 1*

Up to 20 elements can be specified. At least one element must have a non-zero % of sample weight. This window is available on the *Sample Description* tab or by going to ***Options > Active Metals Defaults***.



	Element	Atomic Weight	Atomic Cross. Sect. Area (nm²)	Density (g/cm³)	% of Sample Weight	% Reduced	MxOy X	MxOy Y
1	chromium	51.996	0.0635	7.190	0.00	100.00	1	0
2	cobalt	58.933	0.0662	8.900	0.00	100.00	1	0
3	copper	63.540	0.0680	8.960	0.00	100.00	1	0
4	molybdenum	95.940	0.0730	10.220	0.00	100.00	1	0
5	nickel	58.710	0.0649	8.902	0.00	100.00	1	0
6	palladium	106.400	0.0787	12.020	0.00	100.00	1	0
7	platinum	195.090	0.0800	21.450	0.00	100.00	1	0
8	rhenium	186.200	0.0649	21.020	0.00	100.00	1	0
9	rhodium	102.905	0.0752	12.410	0.00	100.00	1	0
10	silver	107.868	0.0869	10.500	0.00	100.00	1	0

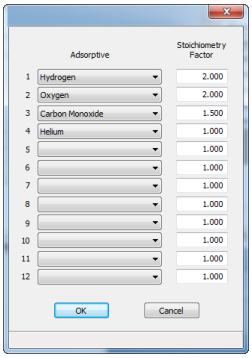
To enter a metal not shown in the default list:

1. Click inside the table. Click either **Insert** or **Append**, then enter the new metal.
2. Click the fields to the right of the element and make the necessary modifications.
3. Click **Save** when done.

Active Metals Fields and Buttons Table

Field or Button	Description
% Reduced *	The percent of metal reduced during preparation.
% of Sample Weight *	Percentage of element contained in the sample. If a composition is specified as a pure metal, X=1 and Y=0 for MxOy - the % of sample is for a pure metal. If a metal oxide composition is specified, Y > 0 - the % of sample is based upon the metal code.
Adsorptive	Click to display and modify both the adsorptive and Stoichiometry for the selected element.

Active Metals Fields and Buttons Table (continued)

Field or Button	Description
	 **Stoichiometry Factor.** A factor which expresses the ratio between the number of active metal molecules and the number of adsorbate molecules.
Atomic Cross Sect. Area (mn^2)	Atomic cross-sectional area of the element.
Atomic Weight	Atomic weight of the element.
Density g/cm^3	Density of the element.
Element	Select or enter the active metal.
MxOy, X *	Number of metal atoms in the oxide.
MxOy, Y *	Number of oxygen atoms in the oxide.
*** Options are shown only when using the Active Metals button on the *Sample Description* tab.**	
	For fields and buttons not listed in this table, see the *Common Fields and Buttons* section of this operator manual.

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4 About Parameter Files

Parameter sets allow repeated use of the file. For example, if the same analysis conditions exist for multiple analyses, an *Analysis Conditions* file containing the recurring conditions can be created. When the sample file is created, the *Analysis Conditions* file can be selected for the analysis conditions. Once it becomes part of the new sample file, the new file can be edited as needed without affecting the original *Analysis Conditions* file.

The following file types can exist as part of the sample information file as well as individual parameter files:

File Type	File Extension
Adsorptive Properties	.ADP
Analysis Conditions	.ANC
Degas Conditions	.DEG
Method	.MTH
Report Options	.RPO
Sample Tube	.STB

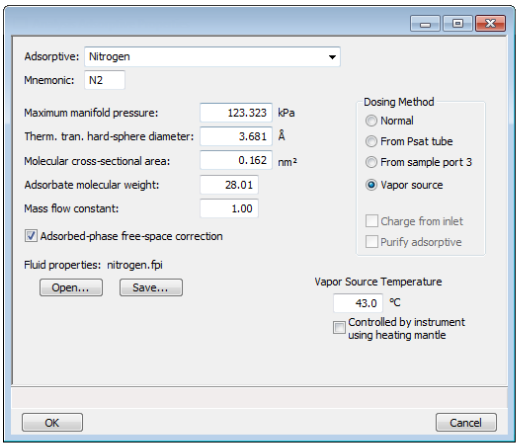
Predefined parameter files are included with the program and can be edited as needed or new parameter files can be created.

ADSORPTIVE PROPERTIES

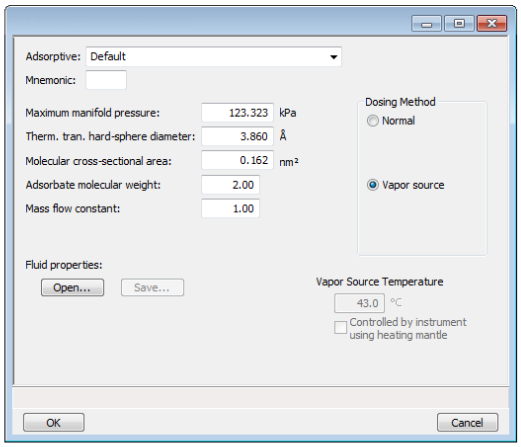
File > Open > [Adsorptive Properties file]

Adsorptive properties provide the adsorptive (analysis gas) characteristics for the analysis.

1. Go to ***File > Open***.
   - Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
   - Select the appropriate file type from the drop-down list on the lower right portion of the window, then select a file name in the list or enter it in the *File Name* field, then click **Open.**
2. Enter a description of the adsorptive in the *Adsorptive* text box (for example, the gas and the temperature). When saved, this description will display in the *Adsorptive* drop-down list of the *Analysis Conditions* window.



Physisorption



Chemisorption

3. Enter the mnemonic for the adsorptive gas (for example, N2) in the *Mnemonic* text box. If this gas is connected to a gas inlet port, this mnemonic must be entered in the *Unit Configuration Gas Selection* for the inlet port.
4. Enter pressure, mass and diameter information in the text boxes.
5. For physisorption: Adsorbed molecules occupy volume in the sample tube, reducing the cold free space. Select the *Adsorbed-phase free-space correction* checkbox to adjust the reported quantity adsorbed to correct for this effect. This option is appropriate for all sample analyses that use the real gas equation of state.
6. To import parameters from a *Fluid Properties* file, click **Open**, locate and select the .FPI file containing the new parameters, then click **Open**. Click **Save** to save the changes. Changing fluid properties should only be necessary if an adsorptive is to be used for which no adsorptive

properties are provided or at an analysis temperature not covered by the standard properties. Contact Micromeritics Scientific Services if new fluid properties are required (http://tech-support.micromeritics.com/portal).

7. In the *Dosing Method* group box, select the source to dose the adsorptive.
8. If *Vapor Source* is selected in the *Dosing Method* group box, the *Vapor Source Temperature* must be entered whether it is to be controlled by the analyzer or not. The analyzer will determine the maximum pressure that can be dosed based on this temperature and the saturation pressure information in the *Fluid Properties*.
9. Select the *Controlled by instrument using heating mantle* checkbox if the vapor source temperature is to be controlled by the instrument with the heating mantle.
10. Click **Save**, then click **Close**.

Adsorptive Properties Fields and Buttons Table

Field or Button	Description
Adsorbate molecular weight	The molecular mass is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot.
Adsorbed phase free-space correction	(For Physisorption only). Adsorbed molecules occupy volume in the sample tube, reducing the cold free space. Select the *Adsorbed-phase free-space correction* checkbox to adjust the reported quantity adsorbed to correct for this effect. This option is appropriate for all sample analyses that use the real gas equation of state.
Adsorptive	Name of the adsorptive gas whose properties are being defined.
Dosing Method	• **Normal.** Dose from a pressurized tank of gas attached to a gas inlet port. • **From Psat tube.** (for physisorption). Select if the Psat tube is to be filled with condensed adsorptive and dosed from the Psat tube. Select this option if using Krypton. • **From sample port 3** (for physisorption) Select if the tube attached to sample port 3 is to be filled with condensed adsorptive and dosed from port 3. • **Vapor source.** Select if a container of condensed vapor is to be attached to the Psat port in place of the Psat tube and is dosed from the Psat port. • **Charge from inlet.** (for physisorption) Use to have the tube automatically charged with condensate from a gas inlet port after the dewar is raised. • **Purify adsorptive.** (for physisorption) Use to have the condensate in the tube purified after charging by evacuating the gas over the condensate. If *Charge from inlet* is selected, select *Purify adsorptive* to have noncondensing contaminants automatically removed from the dos-

Adsorptive Properties Fields and Buttons Table (continued)

Field or Button	Description
	ing tube prior to analysis. After the adsorptive has condensed in the selected Psat tube or port 3, the remaining gas in the tube will be evacuated to remove noncondensing contaminants. A small amount of the purified adsorptive condensate will then return to gas phase to restore equilibrium pressure in the tube.
Fluid properties	Use to import parameters from a *Fluid Properties* file. Click **Open** to browse and select a .FPI file. Locate and select the file, then click **Open** on the file selector window. Click **Save** to save the changes made from the importing selected the .FPI file. Changing fluid properties should only be necessary if an adsorptive is to be used for which no adsorptive properties are provided.
Mass flow constant	Scaling factor for the Mass Flow Controller measured flow rate. Applicable only for the gas used in the flow prep tasks. The default is preset for gases provided with the application.
Maximum manifold pressure	The highest pressure to which the manifold will be dosed. To avoid damage to the analyzer, this number is limited to 925 mmHg. Low pressure sources, such as vapors, will require lower numbers. For gases to be used for dosing after changing a tube from the gas inlet, enter the maximum pressure for dosing from the inlet, not from the tube of condensate.
Mnemonic	Enter the mnemonic name for the adsorptive. If this gas is connected to a gas inlet port, this mnemonic must be entered in the *Unit Configuration Gas Selection* for the inlet port..
Molecular cross-sectional area	The area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations.
Therm. tran. hard-sphere diameter	An estimate of molecular size used in calculating the thermal transpiration correction.
Vapor Source Temperature	Select if the vapor source temperature is to be controlled by the analyzer. This field is enabled only if *Vapor Source* is selected.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

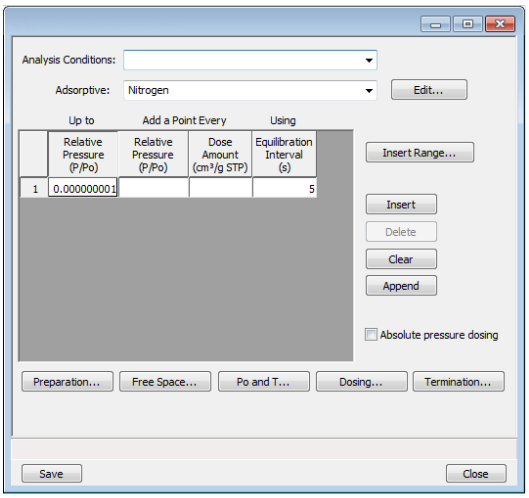
ANALYSIS CONDITIONS

File > Open > [.ANC File]

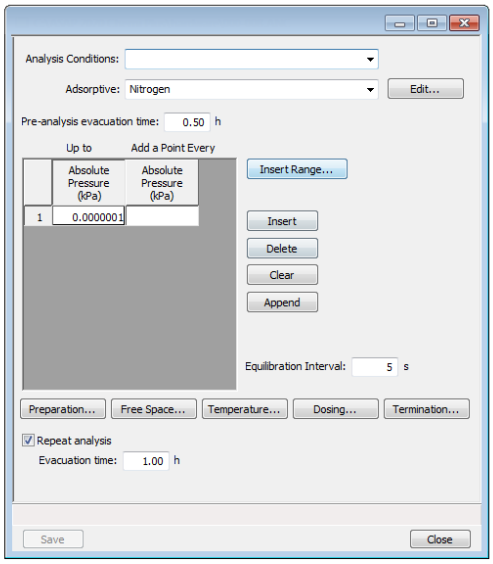
(or click the *Analysis Conditions* tab when in *Advanced* option presentation)

Analysis conditions specify the data used to guide an analysis.

1. Go to ***File > Open***.
   - Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
   - Select the appropriate file type from the drop-down list on the lower right portion of the window, then select a file name in the list or enter it in the *File Name* field, then click **Open.**
2. To overwrite analysis conditions with parameters from another *Analysis Conditions* file, on the *Analysis Conditions* tab, click the *Analysis Conditions* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file.
3. To overwrite adsorptive properties with parameters from another *Adsorptive Properties* file, click the *Adsorptive* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file. See *Adsorptive Properties on page 4 - 2*.
4. Click **Insert Range** to enter starting and ending relative pressure points.



Physisorption



Chemisorption

5. Enable *Absolute pressure dosing* to specify pressure targets in mmHg, mbar, or kPa instead of relative pressure. This option is typically selected when using adsorptives at analysis conditions

above the critical point of the gas; for example, H_2 adsorption on carbon at liquid nitrogen temperature.

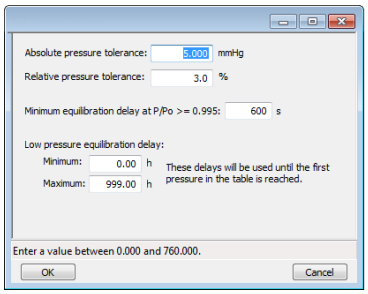
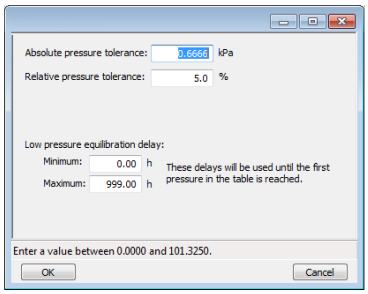
6. Use the following buttons to specify:

Analysis Conditions Buttons Table

Button	Use to Specify...
Dosing	Options for dosing tolerance, low pressure dosing, and dosing near saturation pressure.
Free Space	How the free space is to be measured.
P_0 and T (for physisorption)	How the saturation pressure (P_0) is to be measured or calculated and the analysis bath temperature.
Preparation	Evacuation rate / time / level, leak test and time values, elevator prompts, and in situ degassing or activation.
Temperature (for chemisorption)	Provides access to furnace and accessory temperature control.
Termination (for chemisorption)	Backfill options after analysis.

7. Click **Save**, then click **Close**.

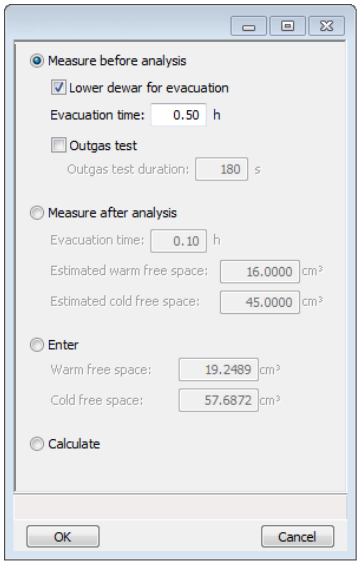
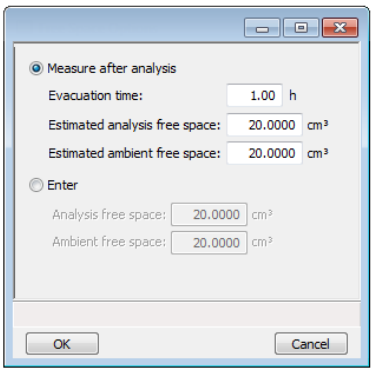
Analysis Conditions Fields and Buttons Table

Field or Button	Description
Absolute pressure dosing (for physisorption)	Specifies pressure targets in mmHg, mbar, or kPa instead of relative pressure. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, H_2 adsorption on carbon at liquid nitrogen temperature. If this option is selected, the *Relative Pressure* labels and entries change to *Absolute Pressure* in the selected pressure units.
Adsorptive	Select an *Adsorptive Properties* file from the drop-down list.
Analysis Conditions	Use to browse for a .ANC file that contains analysis condition parameters to be used in the analysis.
Dosing	 Physisorption  Chemisorption • **Absolute / Relative pressure tolerance.**Values used to determine how

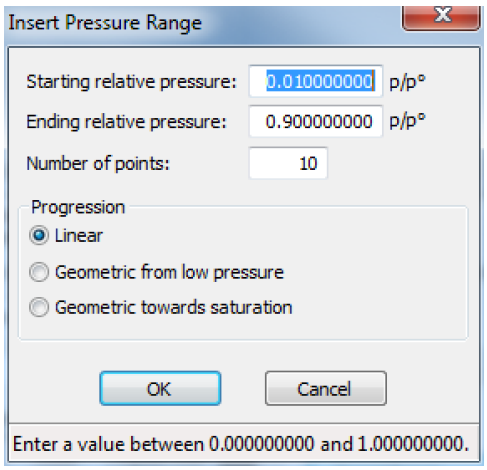
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	close the actual pressure must be to each target pressure from the pressure table. At lower pressures, the relative tolerance value is lower. At higher pressures, the absolute tolerance value is lower. For example: **Experiment 1:** There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 40 mmHg; 5% of 40 mmHg is 2 mmHg. Since 2 mmHg (relative tolerance) is lower than 5 mmHg (absolute tolerance), 2 mmHg is used. Therefore a minimum pressure of 38 mmHg (40 - 2) must be attained to collect data for a target pressure of 40 mmHg. **Experiment 2:** There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 200 mmHg; 5% of 200 mmHg is 10 mmHg. Since 5 mmHg (absolute tolerance) is lower than 10 mmHg (relative tolerance), 5 mmHg is used. Therefore a minimum pressure of 195 mmHg (200 - 5) must be attained to collect data for a target pressure of 200 mmHg. Normally, surface area measurement points are widely spaced and the resulting measurement is not very sensitive to the precise location of points so wider tolerances may be used. Unnecessarily tight tolerances lengthen the analysis. • **Minimum equilibration delay at p/po > = 0.995.** *(for physisorption)* The minimum number of seconds required before equilibration can occur for a relative pressure greater than or equal to 0.995. This field is not available if *Absolute pressure dosing* is selected on the *Analysis Conditions* tab. • **Low pressure equilibration delay**. These delays will be used until the first pressure in the table is reached.

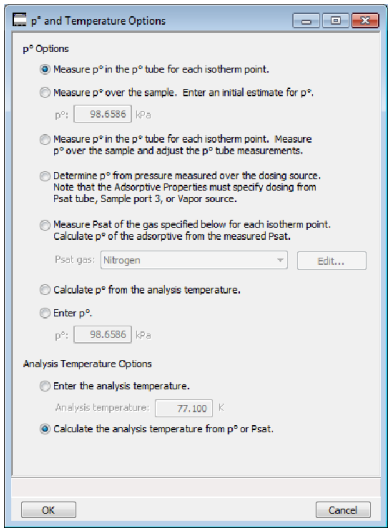
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
Free Space	 **Physisorption**  **Chemisorption** • **Measure before analysis** *(for physisorption)*. Select if the free space is to be measured before the analysis begins. **Lower dewar for evacuation.** If the dewar is to be lowered for evacuation, select this options and enter the length of time for evacuation after the free-space measurement in the *Evacuation time* field. **Outgas test.** Checks for system leaks or sample outgassing. After free space is measured, the dewar is lowered and the sample evacuated for the specified amount of time. The leak test is performed after evacuation. If a leak is found, the leak test repeats nine times, with 30 minutes evacuation between tests. If the 10th leak check fails, the analysis stops and the operator is notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak occurs. • **Measure after analysis**. Measures free space after analysis ends. Enter the evacuation time, the estimated warm free space measurement, and the estimated cold free space measurement. • **Enter**.Measures free space after analysis ends. Enter the evacuation time, the estimated warm free space measurement, and the estimated cold free space measurement. • **Calculate** *(for physisorption)*. Use to have the free space measurement

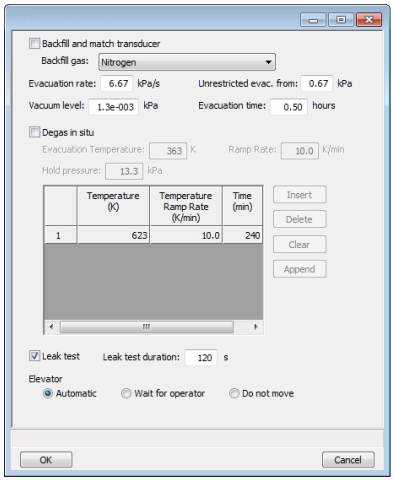
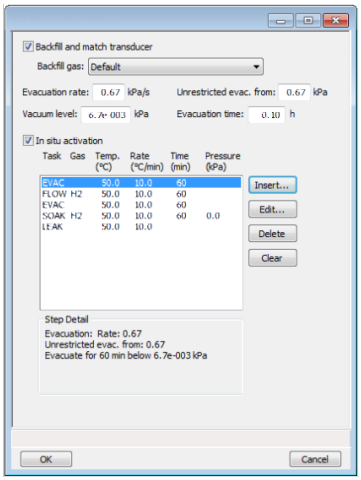
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	calculated using the sample and tube parameters.
Insert Range	To change the window options for physisorption analyses to Absolute pressure, select *Absolute pressure* dosing. Click to display the *Insert Pressure Range* window for entering parameters for the system to autofill the *Up to* column with starting pressure, ending pressure, the number of points to insert within the specified range and whether to have linear or geometric progression.  • **Ending relative pressure.** Relative pressure at which data points will no longer be taken. • **Geometric from low pressure.** Added pressure points will be spaced farther apart as the pressures get higher. • **Geometric towards saturation.** Added pressure points will be spaced closer together as the pressures get higher (closer to saturation). • **Linear.** Inserts evenly spaced points into the table. • **Number of points.** Number of points to be taken between the specified starting and ending relative pressures. • **Starting relative pressure.** Relative pressure at which data points will start to be taken.
p° and Temperature Options (for physisorption)	Use to select options for obtaining the saturation pressure (P_0) and analysis bath temperature.

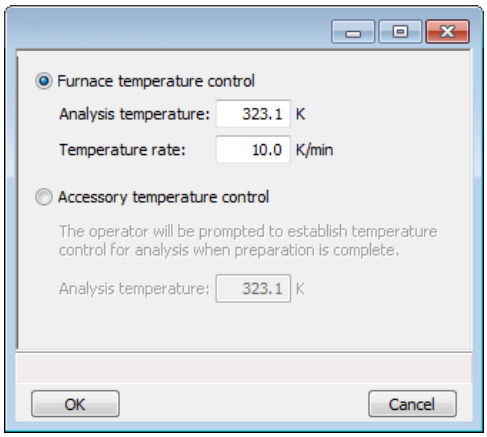
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	 • **Analysis Temperature Options.** Select an option to enter analysis temperature manually, or choose to have it automatically calculated from p° or P_0. • **p° Options.** Select one option indicating how P_0 is to be measured or calculated. • **Psat Gas.** If choosing to measure the P_0 for each isotherm point using a gas other than the adsorptive, select the P_0 gas from the drop-down list, then click **Edit** to modify the P_0 adsorptive properties. Refer to the *Adsorptive* drop-down list earlier in this table for details on editing this window.
Pre-analysis evacuation time (for chemisorption)	Evacuation is required prior to analysis. The default setting is 30 minutes. The evacuation rate, unrestricted pressure, and setpoint are set by clicking the **Preparation** button. Use the **Temperature** button to set the temperature and ramp rate.
Preparation	Use to enter analysis preparation details.

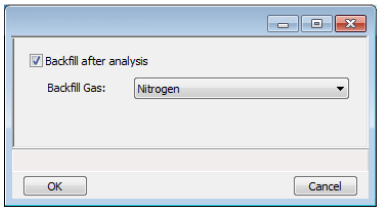
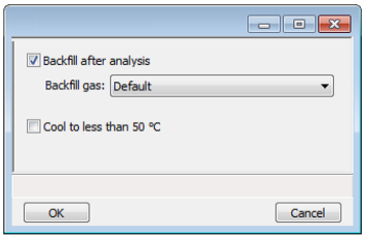
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	 **Physisorption**  **Chemisorption** • **Backfill and match transducer.** Backfills the sample tube to 760 mmHg at the beginning of the analysis and to recalibrate the sample port pressure transducer scale to match the manifold pressure transducer. Select the backfill gas to be used. • **Degas in situ.** *(for physisorption)* Degases the sample on the analysis port prior to analysis. **Evacuation Temperature.** Temperature of the sample during evacuation. **Hold pressure.** Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold pressure*. This feature prevents damage to the sample structure due to 'steaming,' as well as sample elutriation due to excessive escaping gas velocity. **Ramp Rate.** Rate at which the temperature is to change when advancing to the target temperature. • **Elevator.** *(for physisorption)* Select the appropriate elevator control option. **Automatic.** The elevator is raised and lowered automatically. **Wait for operator.** Prompts the operator to set the elevator or analysis bath to the preferred height. When the prompt is acknowledged, the analysis will continue. This option should be used if the analysis bath must be placed manually in the preferred position, or the elevator must

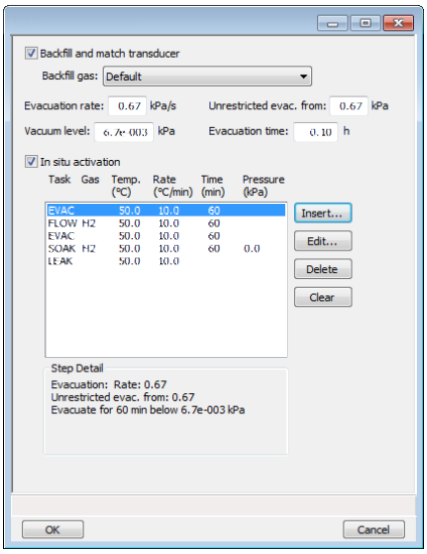
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	be raised to a height other than the standard analysis height. **Do not move.** Use to have the analysis proceed without pausing or moving the elevator. This option should be used when the analysis bath is already in position and should not be moved during analysis. • **Evacuation rate.** The rate for restricted evacuation. • **Evacuation time.** The length of time for preliminary evacuation. If this field is blank, the time entered in the *Pre-analysis evacuation time* field on the *Analysis Conditions* window is used. • **In situ activation.** *(for chemisorption)* When selected, preparation steps will be done. If not selected, the task table is disabled and analysis starts after the preliminary evacuation. • **Leak Test.** Enables the system to check for leaks or sample outgassing before the analysis. The leak test allows sample pressure to rise during the test. If the pressure rises more than 0.15 mmHg, the analysis does not proceed and the operator is notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak exists. **Leak test duration.** Enter the duration of the leak test. • **Vacuum level.** The pressure for unrestricted evacuation. • **Unrestricted evac. from.** The pressure at which unrestricted evacuation is to begin.
Repeat analysis (for chemisorption)	Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold pressure*. This feature prevents damage to the sample structure due to 'steaming,' as well as sample elutriation due to excessive escaping gas velocity.
Temperature (for chemisorption)	

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	• **Furnace temperature control.** Enter the analysis temperature rate. • **Accessory temperature control.** *For user supplied temperature control only*. Enter the intended analysis temperature. The operator will be prompted to establish the analysis temperature before analysis begins.
Termination	Select if backfill is to be done after the analysis. Click the drop-down list to select the backfill gas.  **Physisorption**  **Chemisorption** **Cool to less than 50 °C** *(for chemisorption)*. Select to enable the cool down option.
	For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

CHEMISORPTION TASKS



To ensure safe operation and reliable results, an evacuation task should be included:

- between tasks using different gases
- preceding a leak test

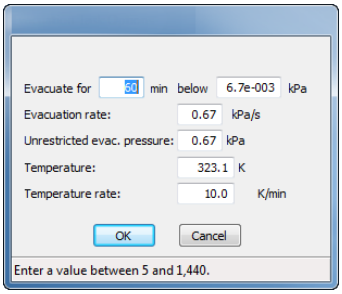
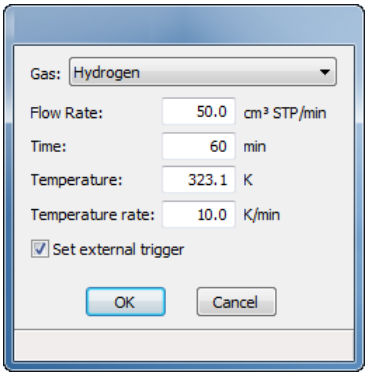
An evacuation will be performed at the analysis temperature for the Pre-analysis Evacuation Time after the last task and before analysis.

If an evacuation is not inserted, a warning message displays.

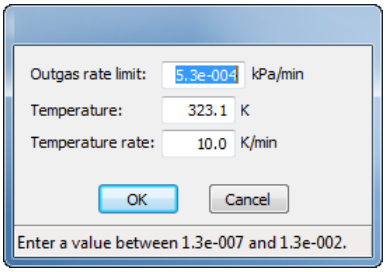
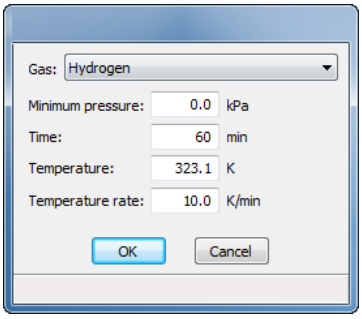
1. On the *Analysis Conditions* tab, click **Preparation**.
2. On the *Preparation Options* window, click **Insert**.
3. On the *Select a task* window, select a task to run and complete the fields using the following table as a guide. Click **OK** to insert the parameters into the *Preparation Options* table.

   View task details by selecting the task within the table. Task details display below the table.

Chemisorption Tasks Table

Task	Description
Evacuation	 • **Evacuate for ___ below ____.** The minutes and pressure for preliminary evacuation. • **Evacuation rate**. The rate for restricted evacuation. • **Unrestricted evac. pressure**. The pressure at which unrestricted evacuation is to begin. • **Temperature**. The temperature to reach during evacuation. • **Temperature rate**. Rate at which the temperature is to change when advancing to the target temperature.
Flow	 • **Gas**. Gas used for the flow task. • **Flow Rate**. The rate at which gas is to flow. • **Time**. The duration of time the sample should remain at the specified temperature. • **Temperature**. The temperature at which the gas will flow for the specified time. • **Temperature rate**. Rate at which the temperature is to change when advancing to the target temperature. • **Set external trigger**. If selected, the contact closure used to trigger an external mass spectrometer will be activated during the temperature ramp.

Chemisorption Tasks Table (continued)

Task	Description
Leak Test	 • **Outgas rate limit**. If a measured leak or outgas rate exceeds the entered value, the test will be reported as failed. Analysis will not be canceled. • **Temperature**. The target temperature for the leak test. • **Temperature rate**. Rate at which the temperature is to change when advancing to the target temperature.
Soak	 • **Gas**. Gas used during the soak task. • **Minimum pressure**. The minimum pressure to be maintained over the sample during the soak. • **Time**. The duration of time the sample is to soak at the specified temperature. • **Temperature**. The temperature at which sample is to be soaked. • **Temperature rate**. Rate at which the temperature is to change when advancing to the target temperature.

EVACUATION RULES

Evacuation parameters apply to all four stages of evacuation with the exception of evacuation time. Evacuation time is set using the fields specified in the following Evacuation Rules.

When an analysis starts, evacuation begins:

1. at ambient temperature before the first preparation step. This step uses the *Evacuation time* field on the *Preparation Options* window.
2. at analysis temperature after preparation and prior to the start of the analysis stage. This step uses the *Pre-analysis evacuation time* field on the *Analysis Conditions* window.
3. at analysis temperature before the repeat analysis if *Repeat analysis* is selected on the *Analysis Conditions* window. This step uses the *Repeat Analysis / Evacuation time* field.
4. at analysis temperature before the free space measurement. This step uses the *Evacuation time* field on the *Free Space Options* window. Measure after analysis must be selected to enable the *Evacuation time* field.

DEGAS CONDITIONS

File > Open > [.DEG File]

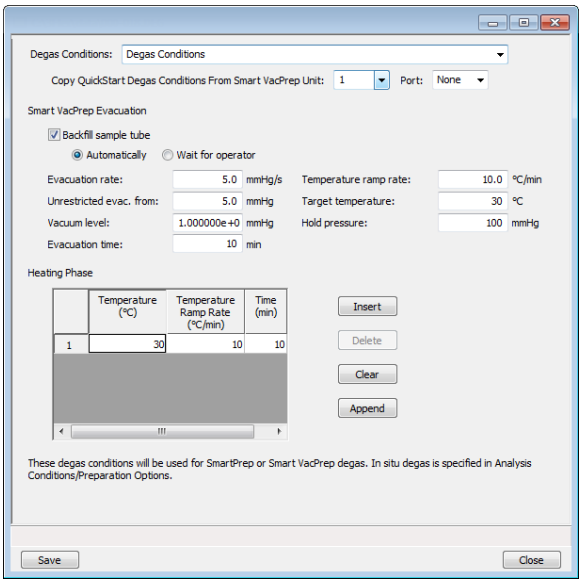
(or click the *Degas Conditions* tab when using *Advanced* presentation display)

Degassing is a required step in preparation for an analysis. The *Degas Conditions* tab provides settings that will be automatically applied during the degassing procedure when using the Smart VacPrep.

> Use this option only when the Smart VacPrep is installed.
>
> The *Degas Conditions* tab displays only if enabled in ***Options > Option Presentation > Show Degas Conditions***.

1. Go to ***File > Open***.
   - Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
   - Select the appropriate file type from the drop-down list on the lower right portion of the window, then select a file name in the list or enter it in the *File Name* field, then click **Open.**
2. To overwrite degas conditions with parameters from another *Degas Conditions* file, click the *Degas Conditions* down arrow, then select a file from the list. Alternatively, click **Browse** and locate the file.

   To overwrite degas conditions from a Smart VacPrep QuickStart setting, select the Smart VacPrep unit number and port number.



3. Click **Insert** to enter up to five stages of degassing (temperature, temperature ramp rate, and time). The maximum temperature when using a Smart VacPrep is 450 °C. The maximum temperature when using a SmartPrep is 400 °C.
4. Click **Save**, then click **Close**.

Degas Conditions Fields and Buttons Table

Field or Button	Description
Degas Conditions	Use to browse for a .DEG file that contains degas condition parameters to be used in the analysis.
Heating Phase	This option is applicable when degassing with either a Smart VacPrep or a SmartPrep. Enter up to five stages of degas conditions. • **Temperature.** Degas temperature. • **Temperature Ramp Rate.** Rate at which the temperature is to change. • **Time.** Amount of time to heat the sample. Use to modify the table contents. • **Insert.** Inserts one row above the selected row. • **Delete.** Deletes the selected row. • **Clear.** Clears all table entries and displays only one default value. • **Append.** Inserts one row at the end of the table.
Smart VacPrep Evacuation	This option is applicable only when degassing with a Smart VacPrep. • **Backfill sample tube.** Indicate if the sample tube should be backfilled automatically or wait for operator response. • **Evacuation Rate.** Rate used for evacuation. • **Unrestricted evac. from.** Pressure at which the unrestricted evacuation is to begin. • **Vacuum level.** Pressure for unrestricted evacuation. • **Evacuation time.** Length of time for preliminary evacuation before proceeding with the *Heating Phase* temperature schedule. The timer starts when the vacuum level is reached. • **Temperature ramp rate.** Rate at which the temperature is to change when advancing to the target pressure. • **Target temperature.** Targeted pressure for evacuation.

Degas Conditions Fields and Buttons Table (continued)

Field or Button	Description
	• **Hold pressure.** Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold* pressure. This prevents damage to the sample structure due to 'steaming' and /or elutriation due to excessive escaping gas velocity.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

REPORT OPTIONS

File > Open > [.RPO File]

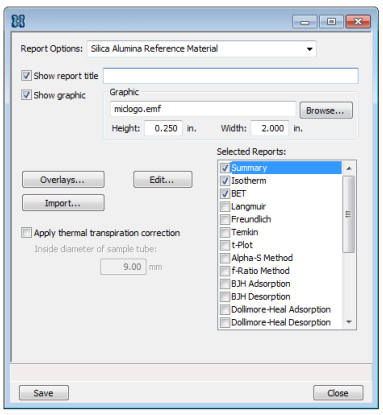
(or click the *Report Options* tab when in *Advanced* option presentation)

Use to specify report options for data collected from an analysis or manually entered data. *Report Options* files also help in customizing report details such as axis scale, axis range, column headings, and components of thickness curve equations. These files may contain tabular reports, plots, or both, as well as advanced report tables.

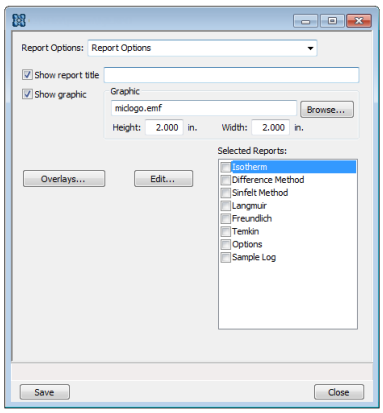
Customized report options files can be created then loaded into a sample file, allowing quick generation of reports.

Report Options files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis. See ***Graph and Sample Overlays on page 7 - 29***.

1. Go to ***File > Open***.
   - Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
   - Select the appropriate file type from the drop-down list on the lower right portion of the window, then select a file name in the list or enter it in the *File Name* field, then click **Open.**
2. To overwrite report options with parameters from another *Report Options* file, on the *Report Options* tab, click the *Report Options* down arrow, then select a file from the list. Alternatively, click **Browse** and locate the file.



Physisorption



Chemisorption

3. [*Optional*] To have a report title display on the report header, select *Show report title*, then enter a title to appear on the report header.

4. [*Optional*] To have a graphic display on the report header, select *Show graphic* to insert a graphic in the report header. Click **Browse** to locate a .BMP or a .EMF file. Specify the graphic size in the *Height* and *Width* text fields.
5. The *Selected Reports* list box displays the reports that may be generated.
   - Select checkboxes to the left of the reports to include in this file.
   - To specify report options, highlight the report in the *Selected Reports* list box, then click **Edit**. Make changes as necessary. Click **OK**.
8. Click **Save**, then click **Close**.

Report Options Fields and Buttons Table

Field or Button	Description
Apply thermal transpiration correction	Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Always use thermal transpiration when performing micropore analyses. See *Thermal Transpiration Correction on page B - 49*. • **Inside diameter of sample tube.** Enabled when *Apply thermal transpiration correction* is selected. Enter the inside diameter of the sample tube used in the analysis. If filler rods are used, enter the filler rod capillary diameter of 1 mm instead.
Import (for physisorption)	Import up to 25 pore distribution data files. These datasets are shown only in BJH and Dollimore-Heal reports.
Name column	Displays a list of files in the selected directory.
Overlays	See *Graph and Sample Overlays on page 7 - 29*.
Report Options drop-down list	Browse for a .RPO file that contains report options parameters to be used in the report.
Selected Reports list box	Select the report names to include in the report.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

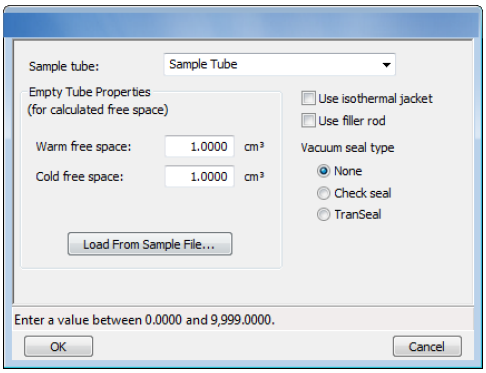
SAMPLE TUBE

File > Open > [.STB File]

(or click **Edit** next to the *Sample Tube* selection on the *Sample Description* tab when in *Advanced* option presentation)

Sample Tube files specify information about the sample tube.

1. Go to ***File > Open.***
   - Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
   - Select the appropriate file type from the drop-down list on the lower right portion of the window, then select a file name in the list or enter it in the *File Name* field, then click **Open.**
2. Enter a description of the sample tube in the *Sample Tube* field. This description will be available as a selection in the *Sample tube* drop-down list on the *Sample Description* tab.



3. In the *Empty Tube Properties* group box, enter the warm free space and the cold free space.
4. Indicate if an isothermal jacket and / or filler rod will be used by selecting *Use isothermal jacket* and / or *User filler rod.* An isothermal jacket should always be used with the analyzer. Filler rods help to ensure accuracy in samples with lower total surface areas by reducing the free-space volume. It is generally a good practice to use filler rods for samples having less than 100 square meters of total surface area. Filler rods are unnecessary for samples with total surface areas greater than 100 square meters.
5. Select the vacuum seal type to be used.
6. Click **Save**, then click **Close**.

Sample Tube Fields and Buttons Table

Field or Button	Description
Cold free space	Empty sample tube gas capacity measured with the dewar raised.
Load from Sample File	Loads parameters from the selected sample file.
Sample Tube	It is a good practice to label each sample tube with a unique identification. Enter that information here. This information will also appear in the *Sample Tube* drop-down list on the *Sample Description* tab.
Use filler rod	Select if a filler rod is to be used in the sample tube. A filler rod reduces the stem free space volume resulting in reduction of free space error.
Use isothermal jacket	Select if an isothermal jacket is to be used. An isothermal jacket maintains a constant temperature profile along the sample tube stem during an extended analysis of more than 1 or 2 hours.
Vacuum seal type	Select the seal type to be used.
Warm free space	Empty sample tube gas capacity measured at room temperature.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

5 About Degassing

Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The sample must be clean when an analysis is performed. The degas process heats the sample and places it under vacuum to remove the moisture and contaminants.

After the sample has been weighed, degas the sample on:

- the analysis port (see *Degas in Situ on page 5 - 4*)
- a Smart VacPrep (see the Smart VacPrep Operator Manual part number 067-42800-01)
- a SmartPrep (see *Degas on the SmartPrep on page 5 - 9*), or
- a user supplied degasser

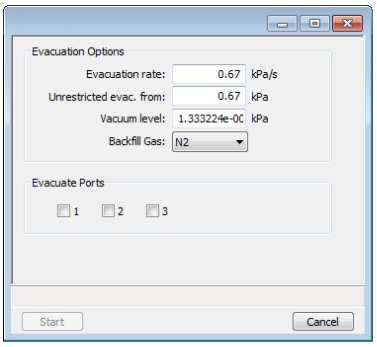
The Check Seal or TranSeal sample tube closures can be used with the Smart VacPrep to minimize sample contamination when transferring the sample tube from the Smart VacPrep to the analyzer port.

- If using the TranSeal, see the instructions included with the TranSeal (part number 350-42803-00).
- If using the Check Seal, see the instructions included with the Check Seal (part number 350-42802-00).

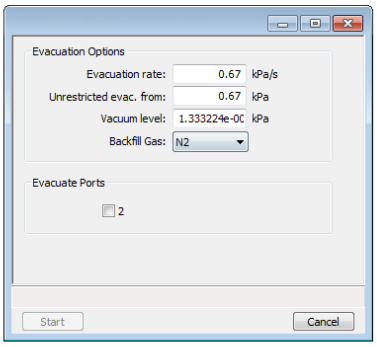
If using the Smart VacPrep, reference the Smart VacPrep Operator Manual (part number 067-42801-01) for additional information. The Smart VacPrep Operator Manual is available in the Online Help and at the end of this operator manual.

EVACUATE PORTS

Unit [n] > Evacuate Ports



Physisorption



Chemisorption

Allows manual evacuation of degas ports.

Evacuate Ports Fields and Buttons Table

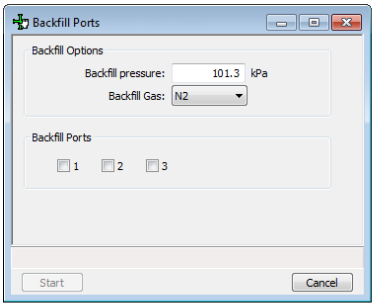
Field or Button	Description
Backfill Gas (for chemisorption)	Select the backfill gas from the drop-down list.
Evacuate Options	• **Evacuation rate.** .The rate for restricted evacuation. • **Unrestricted evac. pressure.** Pressure value at which unrestricted sample evacuation should begin • **Vacuum level.** Specify the vacuum level to be achieved before evacuation begins.
Evacuate Port[s]	Select the ports to evacuate.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

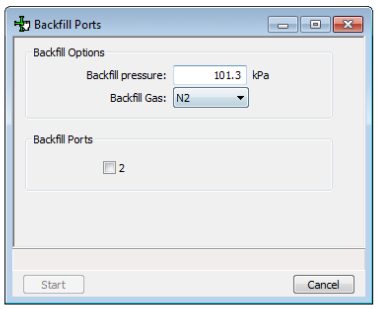
BACKFILL PORTS

Unit [n] > Backfill Ports

Use to backfill ports with gas.



Physisorption



Chemisorption

1. Enter the backfill pressure in the *Backfill pressure* field.
2. Select the ports to be backfilled.
3. Click **Start** to start the process or **Cancel** to stop the process.

DEGAS IN SITU

Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The sample must be clean when an analysis is performed. The degas process heats the sample and places it under vacuum to remove the moisture and contaminants.

Physisorption samples can be degassed on either the analyzer's analysis port (in situ) or on a separate device such as a Smart VacPrep. Degassing should be performed prior to starting analysis.

Samples containing excessive amounts of moisture or significant amounts of other contaminants must be degassed on a separate degas system before attaching to the analyzer to prevent contamination of the analyzer high vacuum system.

Microporous samples should receive a secondary in situ degas on the analyzer to remove any moisture readmitted during transfer from the separate degas system to the analyzer.

1. Install the sample tubes and dewar lid on the analysis port.
2. Go to ***Unit [n] > Sample Analysis***, then click **Browse** to locate an existing sample file or click **New** to create a new one. The sample file *Analysis Conditions tab* > **Preparation** section must have the *Degas in Situ* option enabled.
3. Click **Start**. The *Sample Analysis* window will display a prompt to raise the dewar lid and install the degas heating mantle.
4. Slide the dewar lid up against the sample port nuts. If isothermal jackets are installed, slide the jackets up to touch the bottom of the dewar lid.
5. Install the heating mantle.
6. Acknowledge the prompt on the *Sample Analysis* window; the degas will proceed.
7. Observe the temperatures on the analyzer schematic.
8. When the degas is completed and the mantle has cooled, the *Sample Analysis* window will display a prompt to remove the degas heating mantle, properly position the isothermal jackets and dewar lid, then install the dewar.
9. Remove the heating mantle (it is not necessary to unplug the mantle), support the bottom of the tubes, then remove the mantle cover.

To prevent potential burns, do not touch the sample tube or the heating mantle until they have cooled below 45 °C.

TRANSFER A DEGASSED SAMPLE TO AN ANALYSIS PORT

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

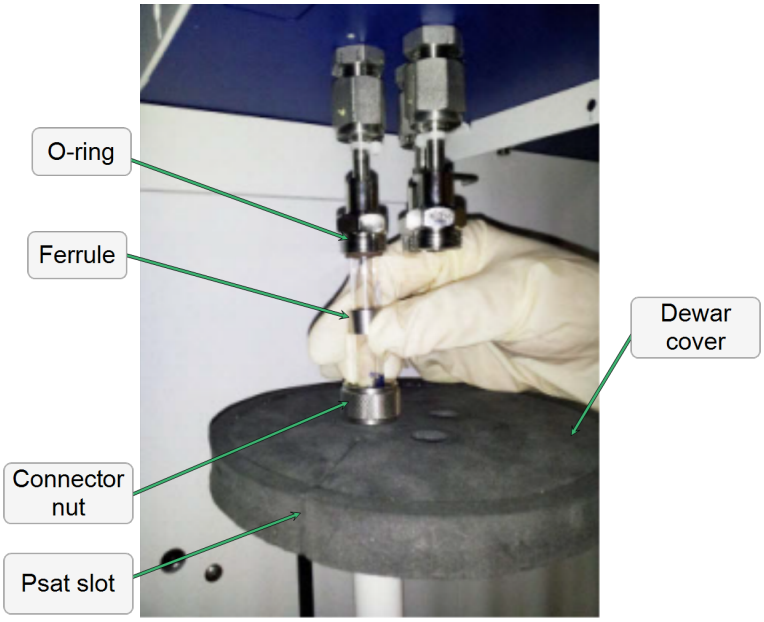
When degassing on a separate degasser such as a Smart VacPrep or SmartPrep, the sample tube must be removed from the degas port, weighed, and then installed onto the analysis port for analysis.

If the sample tube is not mounted on the analysis port immediately, leave it on the degas port. If it is necessary to remove the sample tube and a Check Seal or TranSeal was not used, insert a rubber stopper into the sample tube.

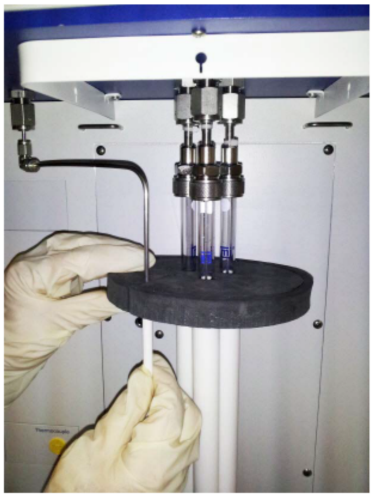
1. Allow the sample tube to cool.

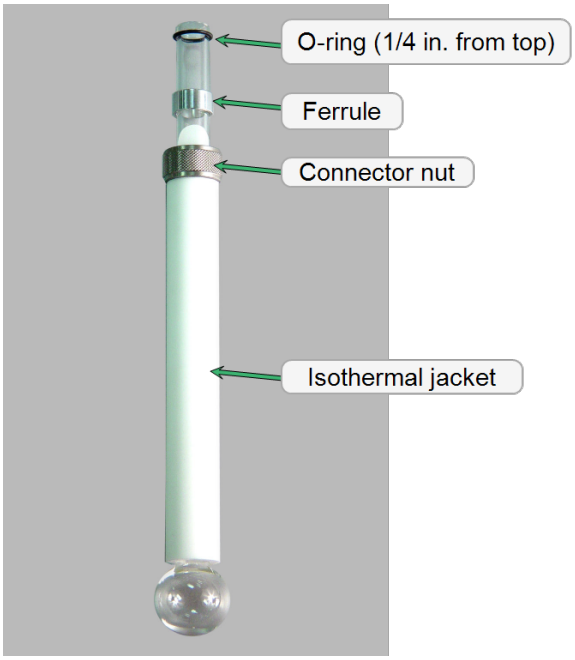
Do not touch the sample tube or the heating mantle until they have reached room temperature. Touching the sample tube, heating mantle, or heating mantle clip before they have cooled could result in burns.

2. Carefully remove the heating mantle clip and the heating mantle from the sample tube and allow the sample tube to cool to room temperature (approximately fifteen minutes).
3. While holding the sample tube, loosen the port connector nut and remove the sample tube from the degas port. If a Check Seal or TranSeal was not inserted prior to degassing, immediately insert a rubber stopper into the sample tube.
4. Weigh the sample tube set. Enter the mass on the Sample Data Worksheet as *Sample tube set plus sample mass* (After Degas).
5. Subtract the *Mass for empty sample tube set* (Before Degas) from the *Sample tube set plus sample mass* (After Degas) to determine the mass of the sample. Record this value as the *Sample mass* (After Degas).
6. If using a Check Seal, ensure that the Check Seal opener is installed in the analyzer sample port. If a rubber stopper was used, remove it from the sample tube.
7. Slide an isothermal jacket down over the sample tube stem until it touches the sample tube bulb.
8. Place the connector nut, ferrule, and O-ring onto the sample tube stem.
9. On the analyzer, loosen the connector nut on the Psat tube and rotate it out of the way.
10. Position the dewar lid so that the slot for the Psat tube is on the left between port 1 and port 2.

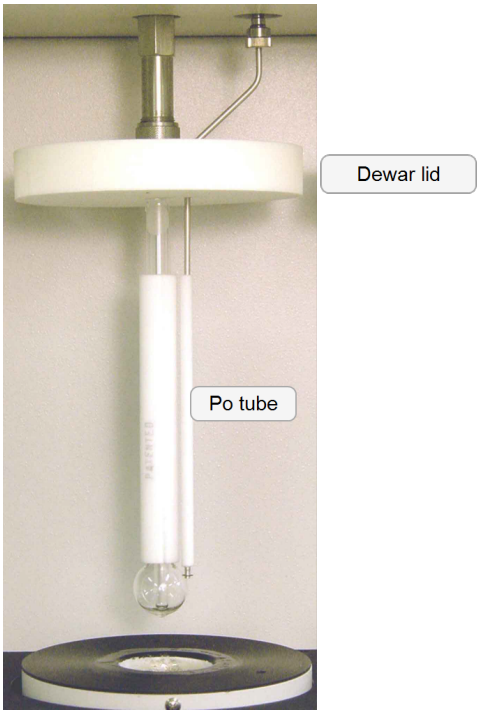


11. Insert the sample tube through one of the holes in the dewar lid.
12. Place the sample port nut, ferrule and O-ring onto the sample tube stem.
13. Insert the sample tube into the analysis port and ensure it is completely in the port. Securely hand tighten the sample port nut onto the analysis port.
14. Repeat all previous steps for each sample tube.
15. Position the dewar lid approximately 3/4 in (19 mm) below the sample port nut.
16. Slide the Psat tube into the Psat slot in the dewar lid and retighten the Psat tube connector nut.
17. Insert the jacket onto the Psat tube. Ensure that the Psat tube jacket is level with the sample tube isothermal jackets.





18. Attach the sample tube to the analysis port, pushing it fully up. Turn the connector nut clockwise and hand tighten.
19. If degassing a sample on the sample port, see *Degas in Situ on page 5 - 4*. Otherwise, place the dewar lid over the sample tube stem just above the isothermal jacket. Ensure the Po tube is next to the sample tube.



20. Install the dewar onto the elevator and place the safety shield over the sample tube and dewar.
21. Begin the analysis.

DEGAS ON THE SMARTPREP

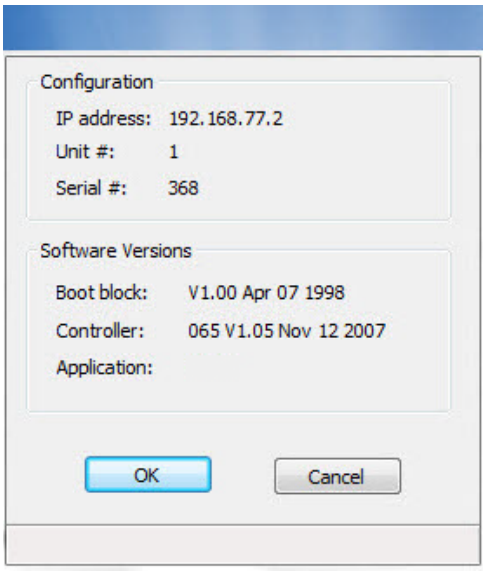
Unit [n] > Degas

If a SmartPrep is not connected to the analyzer, the ***Unit [n] > Degas*** menu options are disabled.

SMARTPREP CONFIGURATION

Unit [n] > Degas > SmartPrep Configuration

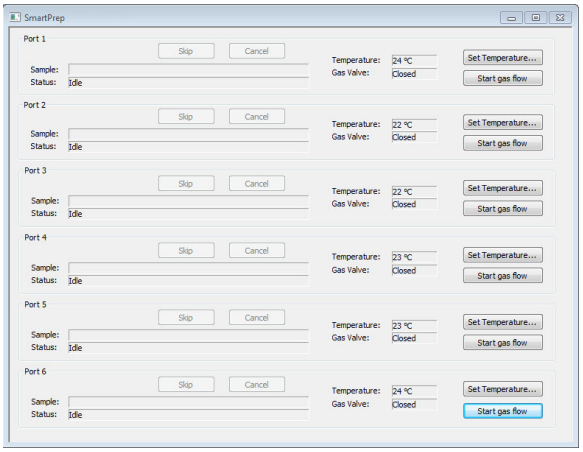
Displays the SmartPrep configuration and software versions.



SHOW SMARTPREP STATUS

Unit [n] > Degas > Start SmartPrep Degas

The SmartPrep Status window allows the monitoring of degas operations and to stop the gas flow after samples are degassed.



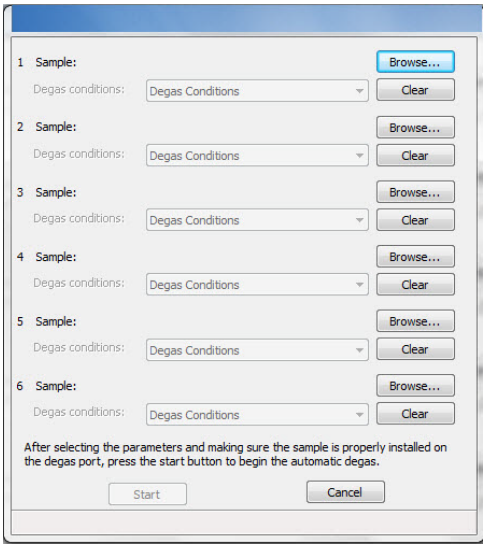
Show SmartPrep Status Fields and Buttons Table

Field or Button	Description
Cancel	Discards any changes or cancels the current process.
Set Temperature	Use to set the temperature of the selected port.
Skip	Use to bypass degassing of the selected sample.
Stop Gas flow	Stops the gas flow to the selected port.

Start SmartPrep Degas

Unit [n] > Degas > Show SmartPrep Degas

The six SmartPrep heating stations are represented by row numbers on the *Automatic Degas* window.



Start SmartPrep Degas Fields and Buttons Table

Field or Button	Description
Browse	Searches for a file. Select a file from the *Name* column or from the library, then click **Open**. Alternatively, double click the file name to open (or import) the file.
Cancel	Discards any changes or cancels the current process.
Clear	Clears all fields.
Start	Starts the operation or process.

DEGAS ON THE SMART VACPREP

Degassing on the Smart VacPrep requires updated analyzer software. If you have not already done so, update the analyzer software for Smart VacPrep support at this URL:

http://www.micromeritics.com/Smart-VacPrep-Software.aspx

If using the Smart VacPrep, reference the Smart VacPrep Operator Manual (part number 067-42801-01) for additional information. The Smart VacPrep Operator Manual is available in the Online Help and at the end of this operator manual.

See the Smart VacPrep Operator Manual part number 067-42800-01.

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6 Perform an Analysis

Prepare for Analysis

The following steps properly prepare the equipment and instrument for an analysis. It is recommended to perform the tasks in the following order:

Step 1 - Clean and Label Sample Tubes on the next page

Step 2 - Create the Sample File on page 6 - 4

Step 3 - Determine the Sample Mass on page 6 - 8

Step 4 - Degas the Sample on page 6 - 12

Step 5 - Install the Sample Tubes and Open the TranSeal on page 6 - 13

Step 6 - Fill and Install the Dewar on page 6 - 14

Dewar Precautions

Always handle glass dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution. If in doubt, contact your safety officer.

When handling dewars containing liquefied gases or cryogenic liquids:

- Wear protective equipment:
  - goggles or face shield
  - an insulated or rubber apron
  - insulated gloves
- When pouring liquefied gases from one container to another:
  - cool the receiving container gradually to minimize thermal shock
  - pour the liquified gas slowly to prevent splashing
  - vent the receiving container to the atmosphere

For glass dewars:

- Use a plastic stirring rod when stirring substances in a dewar containing liquefied gases (or other materials of extremely low temperature). Do not use a glass or metal stirring rod unless it has a protective coating.

- Do not handle heavy objects above the dewar. If unavoidable, place a protective cover over the dewar opening. If an object of sufficient weight is accidentally dropped into the dewar, shattering may occur.
- Do not remove the protective mesh covering. This cover minimizes the risk of flying particles should the dewar be knocked over, dropped, or broken.

STEP 1 - CLEAN AND LABEL SAMPLE TUBES

Sample tubes and filler rods must be clean and dry before samples are added and weighed. The following table indicates which materials are supplied by Micromeritics and which are supplied by the user. The procedures following the materials table are recommended.

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

Supplied by Micromeritics	Supplied by User
• Filler rod • Funnel • Sample Data Worksheet • Sample tube • Sample tube brush • Sample tube rack • Sample weighing support (required for chemisorption sample tubes) • Stopper for sample tube (Check Seal, rubber stopper, or TranSeal)	• Acetone or isopropyl alcohol • Analytical balance • Detergent • Drying oven • Pipe cleaners • Rubber gloves or lint-free cloth • Safety glasses • Ultrasonic cleaning unit • Waste container

1. Preheat drying oven to 110 ºC.
2. Verify that the ultrasonic cleaning unit is clean.
3. Use 5 grams of Alconox (or other suitable detergent) per 500 mL of warm water and fill the ultrasonic unit with enough water to cover the sample tubes and filler rods (if used). If too much detergent is used, it may be difficult to rinse from the sample tubes. Ensure the detergent is dissolved before placing the sample tubes and filler rods into the water.
4. Fill the sample tubes with warm water and place them in the ultrasonic cleaning unit, then place the filler rods in the unit. Turn on the ultrasonic cleaning unit for approximately fifteen minutes.

5. Use rubber gloves to ensure no oils or residue are transferred to the clean tubes and filler rods, then remove the sample tubes and filler rods from the unit.
6. Clean the interior of the sample tubes with the brush supplied with the analyzer.
7. Rinse the sample tubes and filler rods thoroughly with hot water. Rinse again with isopropyl alcohol or acetone. If isopropyl alcohol or acetone is not available, deionized water may be used.

8. Stand the sample tubes on the sample tube rack and place the filler rods in a basket or in the rack. Bake in a vacuum oven for two hours at 110 °C.

Samples tubes can also be cleaned with high purity acetone or isopropyl alcohol and dried for about 10 minutes under heat. If using this method, continue with step 10.

9. Remove the sample tubes and filler rods from the oven and allow to cool.

Do not insert the filler rods at this time. Filler rods are inserted before the sample tube is installed on the analysis port.

10. Blow out the sample tubes with oil free compressed air.
11. Rinse the sample tube closure with isopropyl alcohol, then wipe the sample tube closure dry with a clean, lint-free cloth.
12. Label the sample tube and stopper for identification.
13. Replace the rubber stopper, Check Seal, or TranSeal.

STEP 2 - CREATE THE SAMPLE FILE

- *Create Sample Files in Advanced Option Presentation on page 3 - 1*
- *Create Sample Files in Basic Option Presentation on page 3 - 5*
- *Create Sample Files in Restricted Option Presentation on page 3 - 6*

STEP 3 - DETERMINE THE SAMPLE MASS

Clean, dry sample tubes are essential for accurate results. How much sample to use can be determined best by experiment. In general, a sample providing 40 to 120 square meters of total surface area is recommended for nitrogen analysis. Less than 40 square meters may cause unreliable results. More than 120 square meters will extend analysis time.

Smaller quantities are required for samples having high surface areas. These samples require careful weighing after degassing because a small error may represent a considerable percent of total weight. Proper weighing techniques are most important in this case. Use no less than 100 mg to reduce the effect of weighing errors.

Care should be taken when loading powders; the accessory funnel is useful for this purpose. Large granules or chunks may be loaded with forceps.

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

Analysis results are expressed in units of surface area per gram of sample; therefore, it is important the true sample mass be known.

Follow the instructions on the *Sample Data Worksheet* and complete all fields to find the true sample mass.

Determine Sample Mass for Physisorption

1. Record the *Sample Tube Identification* on the *Sample Data Worksheet.*
2. Tare the balance and allow it to stabilize at zero.
3. Place the empty sample tube set on the balance.
4. Record the stabilized mass on the *Sample Data Worksheet* as *[A] Mass for empty sample tube set*. Remove the sample tube set from the balance.

Do not touch the sample with bare hands while performing the following steps. Doing so could affect the accuracy of results.

5. Place a sample container on the balance. Tare the balance and allow it to stabilize to zero (0).
6. Slowly pour the specified amount of sample into the sample container.
7. Remove the rubber stopper, Check Seal, or TranSeal from the sample tube.
8. Use the sample tube funnel (provided in the accessories kit) and pour the sample from the weighing container into the sample tube.

9. Replace the rubber stopper, Check Seal, or TranSeal.
10. Weigh the sample tube set containing the sample and record the value on the *Sample Data Worksheet* as *[B] Sample tube set plus sample mass (Before Degas)*.
11. Subtract the *[A] Mass for empty sample tube set* from the *[B] Mass of sample tube set plus sample* and record this value as the *[C] Sample mass (Before Degas).*

Determine Sample Mass for Chemisorption

CAUTION

Bulb sample tubes are for pellets and other samples without loose particles. Using powder samples in bulb tubes may cause the loose particles to go into the analyzer's exhaust.

1. Record the *Sample Tube Identification* on the *Sample Data Worksheet*.
2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero.
3. If analyzing a powder or sample made of fine particles, push a piece of quartz wool all the way down into the sample tube. See ***Use Quartz Filter Discs for Chemisorption on the facing page***.
4. If using quartz wool, put a second piece of quartz wool just inside the sample tube. If using filter discs, push a filter disc down into the tube until it sits on top of the quartz wool. Place a second filter disc just inside the sample tube.
5. Place the sample tube set (sample tube with quartz wool or filter discs and stoppers) on the sample support. Record the stabilized mass as the *[A] Mass for empty sample tube set* on the *Sample Data Worksheet*.

6. Remove the sample weighing support and sample tube set from the balance.
7. Place the sample container on the balance and allow the balance to stabilize at zero.

Do not touch the sample with bare hands. Oil from hands could affect the accuracy of results.

8. Slowly add approximately 0.5 to 1.0 gram of sample to the sample container.
9. If a second piece of quartz wool or filter disc was inserted, remove the top portion of the quartz wool or the filter disc from the sample tube.
10. Use a funnel to slowly pour sample from the container into the sample tube on top of the quartz wool in the tube.

Ensure all sample in the container is placed in the sample tube to avoid errors caused by incorrect sample mass.

11. If using quartz wool, insert the top portion of quartz wool into the tube and press it down. If using filter discs, insert the filter disc into the tube and press it down.

> Ensure the disc is flat on top of the sample. A seal must be created around the edge to prevent the sample from escaping.

12. Wipe the top of the sample tube with a lint-free cloth, such as a Kimwipe®, to remove any quartz wool that may have adhered to the surface.
13. Weigh the sample tube set containing the sample and the stoppers. Record this mass as the *Sample + tube*.

See ***Sample Data Worksheet for Gas Adsorption on page L - 2***

Use Quartz Filter Discs for Chemisorption

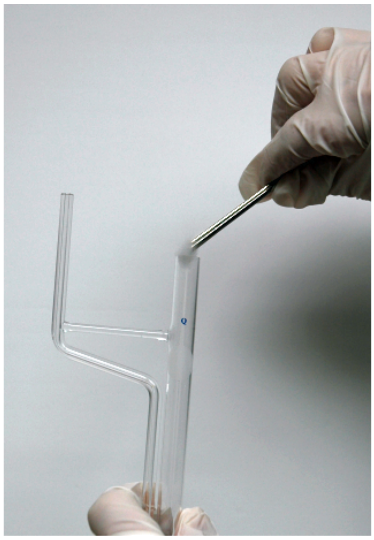
> The use of quartz wool is not mandatory, however it can provide extra protection for light powdered samples.

> Wear latex gloves when handling the sample tube. The natural oils in human skin can chemically damage and weaken the quartz tube. It is also important that the sample tube and its components, as well as the sample and exhaust ports, be clean and free of debris. Dust particles from quartz wool or the insulator disc of previous analyses may adhere to the port and / or components, preventing a proper seal of the sample tube.

> The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

Use quartz filter discs or quartz wool to aid in chemisorption sample preparation. Quartz filter discs (placed both below and above powdered samples) not only provide a more uniform sample surface but also keep the analyzer free of sample debris. The filters can be used up to 900 °C.

1. Insert a small portion of quartz wool into the sample tube to serve as a support for the powdered sample. Use a filler rod or smaller sample tube to push the quartz wool to the bottom of the sample tube.



Insert quartz wool and disc

Top disc, powdered sample, bottom disc, quartz wool

2. Insert a quartz disc into the sample tube and push it into the tube until it rests on top of the quartz wool. Inspect the disc to ensure that there is a good seal and that the sample will not go past the filter. An additional filter can be inserted if needed.
3. Insert a second filter disc on top of the quartz wool. Ensure that the filter is placed high enough into the sample tube for easy retrieval.
4. Take the initial tube weight (with both filters).
5. Remove the top filter disc. Place it on a clean surface, then use a funnel to add the powdered sample on the bottom filter disc.
6. Reinsert the top filter disc into the sample tube, then use a rod or smaller sample tube to push it down until it reaches the top of the sample.
7. To remove the quartz wool and disc after analysis, use the quartz wool extractor tool.

Step 3 - Determine the Sample Mass

Clean, dry sample tubes are essential for accurate results. How much sample to use can be determined best by experiment. In general, a sample providing 40 to 120 square meters of total surface area is recommended for nitrogen analysis. Less than 40 square meters may cause unreliable results. More than 120 square meters will extend analysis time.

Smaller quantities are required for samples having high surface areas. These samples require careful weighing after degassing because a small error may represent a considerable percent of total weight. Proper weighing techniques are most important in this case. Use no less than 100 mg to reduce the effect of weighing errors.

Care should be taken when loading powders; the accessory funnel is useful for this purpose. Large granules or chunks may be loaded with forceps.

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

Analysis results are expressed in units of surface area per gram of sample; therefore, it is important the true sample mass be known.

Follow the instructions on the *Sample Data Worksheet* and complete all fields to find the true sample mass.

Determine Sample Mass for Physisorption

1. Record the *Sample Tube Identification* on the *Sample Data Worksheet.*
2. Tare the balance and allow it to stabilize at zero.
3. Place the empty sample tube set on the balance.
4. Record the stabilized mass on the *Sample Data Worksheet* as *[A] Mass for empty sample tube set*. Remove the sample tube set from the balance.

Do not touch the sample with bare hands while performing the following steps. Doing so could affect the accuracy of results.

5. Place a sample container on the balance. Tare the balance and allow it to stabilize to zero (0).
6. Slowly pour the specified amount of sample into the sample container.
7. Remove the rubber stopper, Check Seal, or TranSeal from the sample tube.
8. Use the sample tube funnel (provided in the accessories kit) and pour the sample from the weighing container into the sample tube.

9. Replace the rubber stopper, Check Seal, or TranSeal.
10. Weigh the sample tube set containing the sample and record the value on the *Sample Data Worksheet* as *[B] Sample tube set plus sample mass (Before Degas)*.
11. Subtract the *[A] Mass for empty sample tube set* from the *[B] Mass of sample tube set plus sample* and record this value as the *[C] Sample mass (Before Degas)*.

Determine Sample Mass for Chemisorption

CAUTION

Bulb sample tubes are for pellets and other samples without loose particles. Using powder samples in bulb tubes may cause the loose particles to go into the analyzer's exhaust.

1. Record the *Sample Tube Identification* on the *Sample Data Worksheet*.
2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero.
3. If analyzing a powder or sample made of fine particles, push a piece of quartz wool all the way down into the sample tube. See ***Use Quartz Filter Discs for Chemisorption on page 6 - 7***.
4. If using quartz wool, put a second piece of quartz wool just inside the sample tube. If using filter discs, push a filter disc down into the tube until it sits on top of the quartz wool. Place a second filter disc just inside the sample tube.
5. Place the sample tube set (sample tube with quartz wool or filter discs and stoppers) on the sample support. Record the stabilized mass as the *[A] Mass for empty sample tube set* on the *Sample Data Worksheet*.

6. Remove the sample weighing support and sample tube set from the balance.
7. Place the sample container on the balance and allow the balance to stabilize at zero.

Do not touch the sample with bare hands. Oil from hands could affect the accuracy of results.

8. Slowly add approximately 0.5 to 1.0 gram of sample to the sample container.
9. If a second piece of quartz wool or filter disc was inserted, remove the top portion of the quartz wool or the filter disc from the sample tube.
10. Use a funnel to slowly pour sample from the container into the sample tube on top of the quartz wool in the tube.

Ensure all sample in the container is placed in the sample tube to avoid errors caused by incorrect sample mass.

11. If using quartz wool, insert the top portion of quartz wool into the tube and press it down. If using filter discs, insert the filter disc into the tube and press it down.

Ensure the disc is flat on top of the sample. A seal must be created around the edge to prevent the sample from escaping.

12. Wipe the top of the sample tube with a lint-free cloth, such as a Kimwipe®, to remove any quartz wool that may have adhered to the surface.
13. Weigh the sample tube set containing the sample and the stoppers. Record this mass as the *Sample + tube*.

See *Sample Data Worksheet for Gas Adsorption on page L - 2*

STEP 4 - DEGAS THE SAMPLE

After the sample has been weighed, use a degassing unit to remove any contaminants which may have adsorbed to the surface or pores. Appropriate degassing units are available from Micromeritics.

If using the Smart VacPrep degasser, go to ***Smart VacPrep > Unit [n] > Start Degas***, then degas the sample using menu commands and information entered on the *Degas Conditions* tab.

After degassing is complete, perform the following steps:

1. Weigh the sample tube set containing the sample, then record the mass on the Sample Data Worksheet as *[B] Sample tube set plus sample mass (After Degas)*.
2. Subtract the *[A] Mass for empty sample tube set (Before Degas)* from the *[B] Sample tube set plus sample mass (After Degas)* to obtain the sample's mass. Record this value as *[C] Sample mass (After Degas)*.

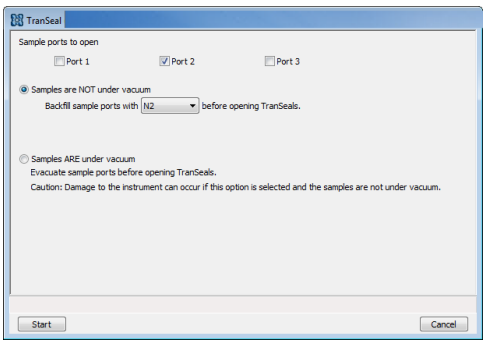
STEP 5 - INSTALL THE SAMPLE TUBES AND OPEN THE TRANSEAL

- See *Install the Sample Tubes for Physisorption on page 11-11*

Open the TranSeal

Unit > Open TranSeal

Outlines the process of safely opening one or more TranSeals on sample ports.



Damage may occur to the analyzer if the samples are not under vacuum and the *Samples ARE under vacuum* option is selected.

TranSeal Fields and Buttons Table

Field or Button	Description
Sample ports to open	Select the ports to open during analysis.
Samples are NOT under vacuum	Select if samples are NOT under vacuum and specify the amount of back-fill and adsorptive to be used prior to opening TranSeals.
Samples ARE under vacuum	Select if the samples ARE under vacuum. This option evacuates sample ports prior to opening TranSeals. Do not select this option if the samples are not under vacuum as analyzer damage may occur.
Start	Opens the TranSeals. The selected sample port will be either backfilled or evacuated as specified. The user will be prompted to open the TranSeals. For each selected port, an event is recorded in the *Instrument Log* file with the port pressure before and after opening the TranSeal.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* section of this operator manual.

STEP 6 - FILL AND INSTALL THE DEWAR

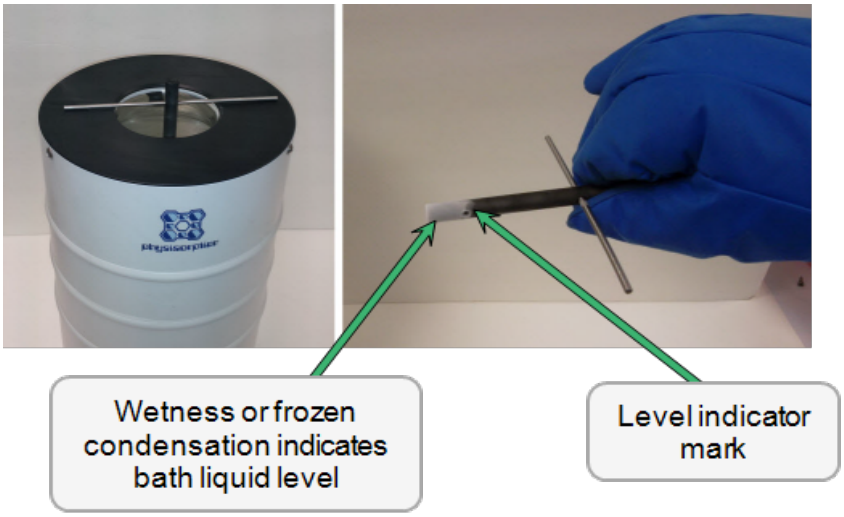
See *Dewar Precautions on page 6 - 1*.

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

1. Fill the dewar with the analysis bath liquid (liquid nitrogen) to no higher than 2 1/4 in. (5.7 cm) from the top. Filling the dewar higher than this will cause an error in the free space measurement.

Incorrect fluid levels can lead to measurement errors. Check the level of the bath liquid before each analysis.

2. Insert the dipstick and check the level of the analysis bath liquid. Condensation should not exceed the level indicator mark.



3. For best results, if the dewar has not been used for a while, allow approximately 30 minutes for the temperature of the dewar to stabilize with the bath liquid, then recheck the level of the bath liquid. Add additional liquid if necessary.
4. If using isothermal jackets, slide the jackets down the sample tube until the jackets touch the sample tube bulbs.
5. Slide the dewar lid to approximately 3/4 in. (19 mm) from the sample port nuts to ensure a proper seal on the top of the dewar.
6. Attach the safety shield to the brackets on the front of the analyzer.

PERFORM AN ANALYSIS SEQUENCE

Unit [n] > Analysis Sequence

Use to perform a sequence of analyses on port 2. If the *Analysis Sequence* option is selected and a sequence analysis is already in progress, the program continues to run the four steps for the analysis in progress. Files can be added and removed from the sequence while it is in progress.

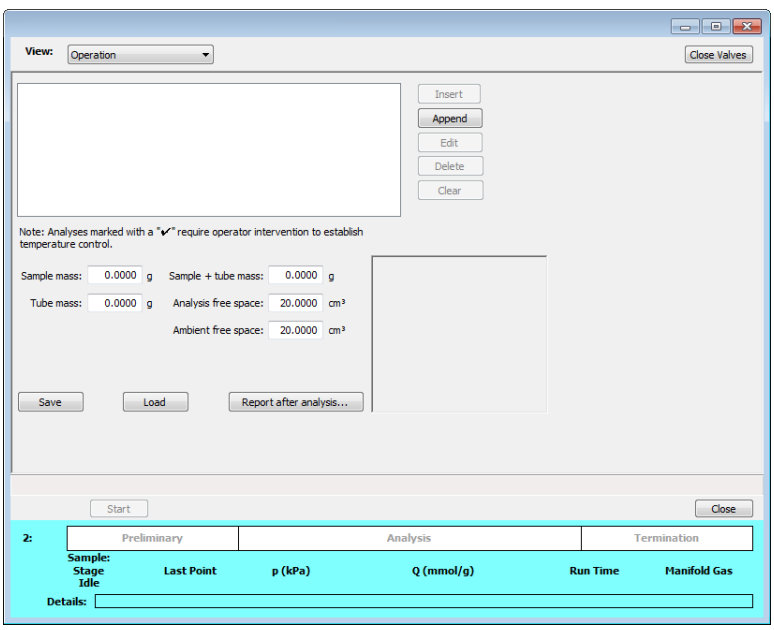
The analysis sequence can:

- run a sequence of only physisorption files
- run a physisorption analysis as a part of the chemisorption analysis sequence
- run a physisorption file in any position in the sequence

The physisorption live graph will be used for the physisorption analysis within a chemisorption sequence.

Operator intervention will be required between analyses in the sequence if the temperature control type changes. Intervention will be required if at the beginning of the analysis:

1. Go to ***Unit [n] > Analysis Sequence.***



2. To manually close all analyzer valves, click **Close Valves**.
3. Click **Insert** to select and insert a sample file above the selected sample file or click **Append** to select and append a sample file to the end of the list.

4. Edit the fields below the sample file selections.
5. Click **Save** to save the selected files as an analysis sequence file (.SEQ) for future analyses. Click **Load** to load the previously saved .SEQ file.
6. Click **Report after analysis** to generate reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK** to return to the previous window.
7. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.
8. When the analysis is complete, remove the sample tube and store (or dispose of) the sample material as applicable.

CAUTION

Use caution when removing the sample tube if using a hanging filler rod. The sample tube O-ring or dewar lid may snag the filler rod retaining ring. Loosen the snag gently; excessive force may break the tip of the filler rod.

Analysis Sequence Fields and Buttons Table

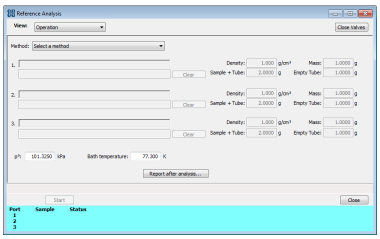
Field or Button	Description
Close Valves	Closes all valves on the unit.
Report after analysis	Generates reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK** to return to the previous window.
Sample mass / Sample + tube mass / Tube mass / Analysis free space / Ambient free space	Enter the values for the sample's mass and free space.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

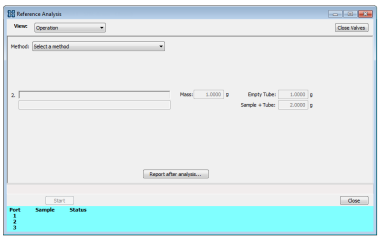
PERFORM A BLANK ANALYSIS

The *Blank Analysis* is run in the same manner as a *Reference Material* analysis except a blank method is selected and no sample material is placed in the sample tube.

1. Go to ***Unit [n] > Reference Analysis***.
2. Select *Operation* from the *View* drop-down list. The analysis will not start if another view is selected.
3. Click **Browse** from the *Method* drop-down list, select a method, then click **Open**.
4. To manually close all analyzer valves, click **Close Valves**.



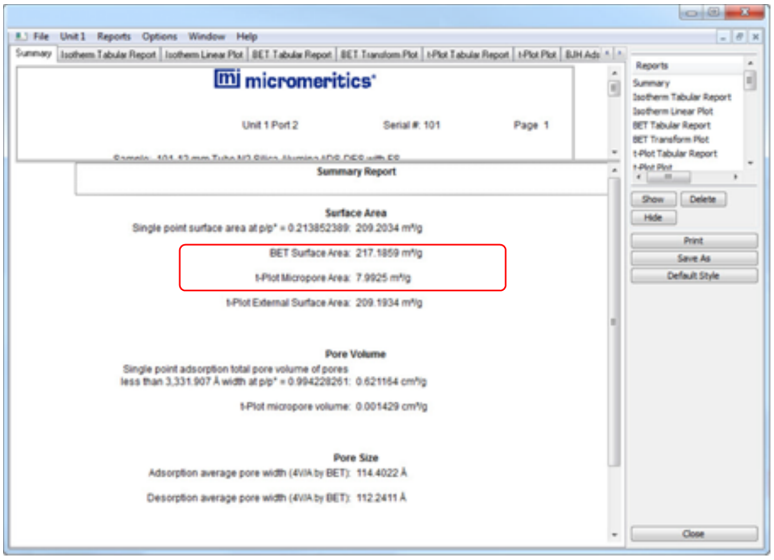
Physisorption



Chemisorption

5. Enter *Mass* for each sample. Verify the populated information is correct and modify if necessary. This information is pulled from the selected file. If a *Blank Analysis* is selected, enter a sample mass of 1 g.For physisorption, the *Density* value is applicable only if using the *Calculate* method for the free space determination.
6. For physisorption: Edit the *po* and *Bath temperature* fields, if necessary.
7. Click **Report after analysis** to generate reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK**.
8. Click **Start** to begin the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.
9. When the analyses are complete, click the *Report Port 1* icon and compare the *BET Surface Area* shown on the *Summary Report* with the *BET Surface Area* shown on the reference material bottle. The values should match within the tolerance level shown on the bottle. Repeat on ports 2 and 3 if running a physisorption analysis.

   If a *Blank Analysis* was selected, look at the *Blank Analysis Report*. The isotherm points should all fall between the minimum and maximum specification lines.



- If the results are within tolerance, the analyzer is operating properly. Click **Close**.
- If the results are not within tolerance, refer to the following *Cause and Action* table. After performing the action, perform the reference analysis again.

Cause and Action Table

Cause	Action
The sample was not degassed properly.	Degas the sample again.
The gas lines are not clean.	Perform the procedure for cleaning and verifying gas lines, then try again.
The measured free space is too high.	This indicates the helium is not pure enough. Use helium that is 99.999% pure, then try again.

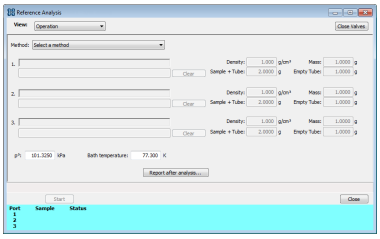
PERFORM A REFERENCE ANALYSIS

Unit [n] > Reference Analysis

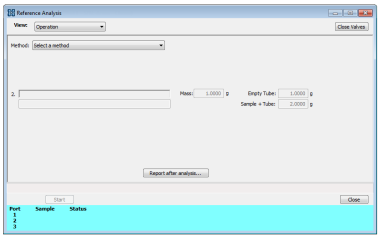
A reference analysis is used to verify the analyzer is operating properly and producing optimum results. These methods provide specifications for critical report quantities and reporting of whether quantities are in or out of specification.

When running a reference analysis, use the appropriate reference material provided in the accessories kit to perform this analysis. The results should match those shown on the label of the reference material bottle, within the tolerance level.

1. Go to ***Unit [n] > Reference Analysis***.
2. Select *Operation* from the *View* drop-down list. The analysis will not start if another view is selected.
3. Click **Browse** from the *Method* drop-down list, select a method, then click **Open**.
4. To manually close all analyzer valves, click **Close Valves**.



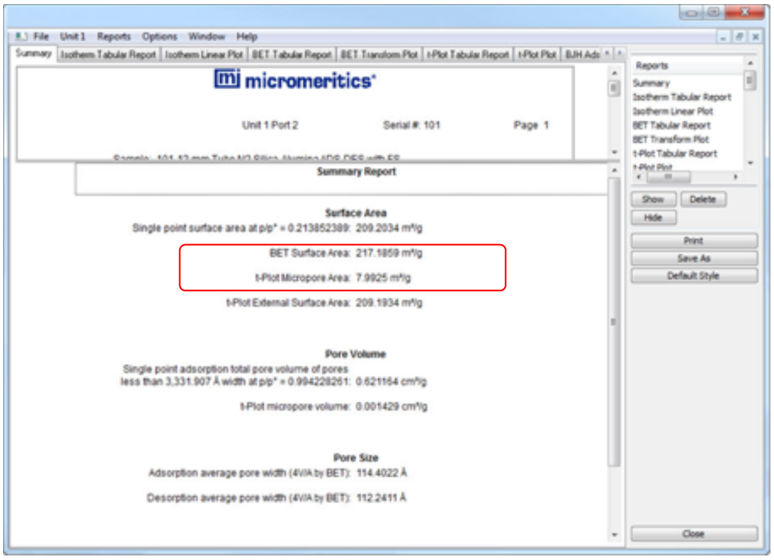
Physisorption



Chemisorption

5. Enter *Mass* for each sample. Verify the populated information is correct and modify if necessary. This information is pulled from the selected file. If a *Blank Analysis* is selected, enter a sample mass of 1 g.For physisorption, the *Density* value is applicable only if using the *Calculate* method for the free space determination.
6. For physisorption: Edit the *po* and *Bath temperature* fields, if necessary.
7. Click **Report after analysis** to generate reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK**.
8. Click **Start** to begin the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.
9. When the analyses are complete, click the *Report Port 1* icon and compare the *BET Surface Are*a shown on the *Summary Report* with the *BET Surface Area* shown on the reference material bottle. The values should match within the tolerance level shown on the bottle. Repeat on ports 2 and 3 if running a physisorption analysis.

   If a *Blank Analysis* was selected, look at the *Blank Analysis Report*. The isotherm points should all fall between the minimum and maximum specification lines.



- If the results are within tolerance, the analyzer is operating properly. Click **Close**.
- If the results are not within tolerance, refer to the following *Cause and Action* table. After performing the action, perform the reference analysis again.

Cause and Action Table

Cause	Action
The sample was not degassed properly.	Degas the sample again.
The gas lines are not clean.	Perform the procedure for cleaning and verifying gas lines, then try again.
The measured free space is too high.	This indicates the helium is not pure enough. Use helium that is 99.999% pure, then try again.

PERFORM A SAMPLE ANALYSIS

Unit [n] > Sample Analysis

- *Prepare for Analysis on page 6 - 1*

Use to perform up to three analyses with different analysis conditions and / or report options on a physisorption analyzer and one analysis on a chemisorption analyzer. Physisorption sample files can be loaded into ports 1, 2, and 3 allowing one analysis using different analysis conditions to run on each port.

For physisorption, use to perform up to three analyses with different analysis conditions and / or report options. Sample files can be loaded into ports 1, 2, and 3 allowing one analysis using different analysis conditions to run on each port.

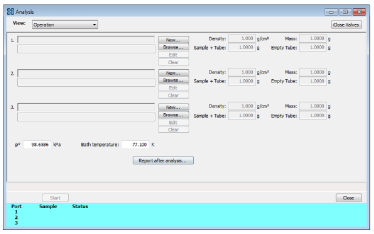
For chemisorption, use to perform an analysis on port 2.

When **Start** is selected, the selected sample file's analysis conditions will be compared with the port's hardware configuration to verify that the specified analysis is supported by the hardware:

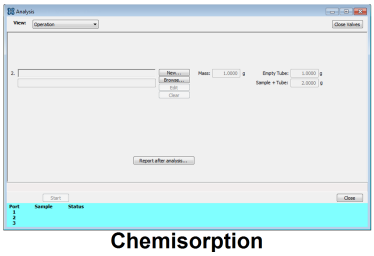
- If *Krypton* is selected as the adsorptive, there must be a 10 mmHg transducer present on the port.
- The minimum target pressure must be no less than the *Minimum Record Pressure* for the minimum range transducer present on the port.
- The selected sample files will be checked for matching adsorptive gases, matching Psat or P_0 gases if measured, and matching backfill gases. All selected gases, except those in a precharged vapor source, must be connected to gas inlets and identified on the *Unit [n] > Unit Configuration > Gas Selections* window. See *Specify Gas Ports on page 2 - 19*.
- If any selected sample file specifies an *Adsorptive Dose Method* from port 3 and a sample file is selected for port 3, an error message displays indicating the problem and the *Start* window will remain active.
- If *in-situ Degas* is selected for any samples, the operator is prompted to raise the isothermal jackets, then connect and install the degas heating mantle on the sample tubes. If this occurs, the operator will be prompted after degas to remove the heating mantle and properly position both the isothermal jackets and dewar lid.
- If *Vapor Source Temperature Control* is selected and the vapor heating mantle is not connected, the operator is prompted to install and connect the vapor heating mantle. If *Degas* is selected for any samples, this will occur after the prompt to remove the degas heating mantle. Otherwise, this will occur immediately at the start of analysis.

Click **Next** to schedule additional analyses after the completion of the first series of analyses. The **Next** button displays after the first set of analyses is complete. Samples cannot be removed from or added to ports until the full set of analyses has completed.

1. Go to ***Unit [n] > Sample Analysis***.



Physisorption



Chemisorption

2. To manually close all analyzer valves, click **Close Valves**.
3. For a selected port, either click **Browse** and select a sample information file, or click **New** to create a new sample information file. On Port 1, up to three sample files may be selected. The files will be loaded into ports 1, 2, and 3 in the order they appear in the file selector. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.

For physisorption: A sample cannot be attached to port 3 if port 3 is being used as the vapor source.

4. Verify the information populated into the sample identification, *Density* (on physisorption samples only), *Mass*, *Sample + Tube*, and *Empty Tube* fields. This information is pulled from the selected or newly created sample file. The *Density* value is applicable only if using the *Calculate* method for the free space determination.
5. For physisorption: Edit the p^0 and *Bath temperature* fields, if necessary.
6. Click **Report after analysis** to generate reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK** to return to the previous window.
7. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.
8. When the analysis is complete, remove the sample tube and store (or dispose of) the sample material as applicable.

CAUTION

Use caution when removing the sample tube if using a hanging filler rod. The sample tube O-ring or dewar lid may snag the filler rod retaining ring. Loosen the snag gently; excessive force may break the tip of the filler rod.

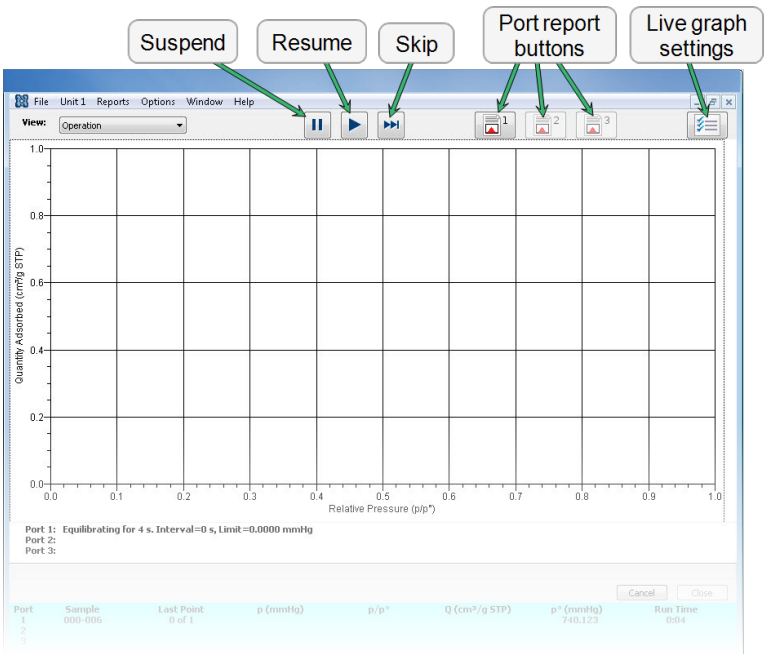
Analysis Fields and Buttons Table

Field or Button	Description
Bath temperature (for physisorption)	Enter the temperature for the analysis bath.
Close Valves	Closes all valves on the unit.
Density / Mass /	Enter values for the sample's mass and density. These values may be

Analysis Fields and Buttons Table (continued)

Field or Button	Description
Sample + Tube / **Empty Tube**	edited after analysis. • Density • Mass • Sample + Tube • Empty Tube
New	Creates a new sample information file.
p^0 (for physisorption)	Enabled if *Entered* is selected for the p_0 measurement for at least one file.
Report after analysis	Generates reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK** to return to the previous window.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**



Sample Analysis Graph Fields and Buttons Table

Field or Button	Description
Live Graph Settings	Select Thermal transpiration, X-axis Quantity (relative or absolute pressure) and the X-Axis Scale (linear or logarithmic).
Report after analysis	Generates reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK** to return to the previous window.
Report Port [n]	Generates a report on data being collected on the respective port. The reports are printed to the screen only.
Resume	Restarts the suspended analysis.
Skip	Skips to the next step. This button is visible only when an analysis is in progress. Select the ports to skip.
Status window	Displays the last point pressure and relative pressure for each port with varying numbers of digits after the decimal if 10 mmHg and 0.1 mmHg transducers are present on that port, as follows: P < 0.1: 6 digits, 0.1 <= P < 10: 4 digits, P >= 10: 2 digits. Relative pressure will show 3 more digits than absolute pressure.
Suspend	Suspends an analysis in progress. Select the ports containing the analysis to suspend.

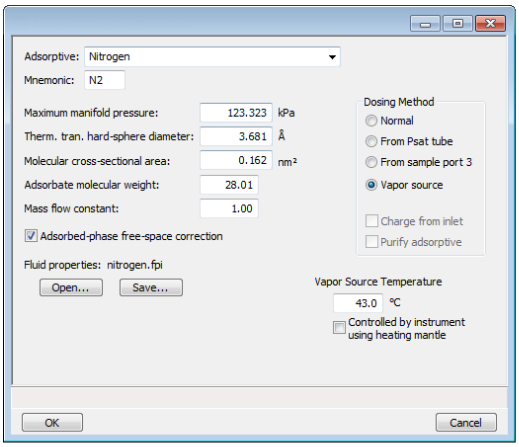
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

PERFORM A VAPOR ANALYSIS

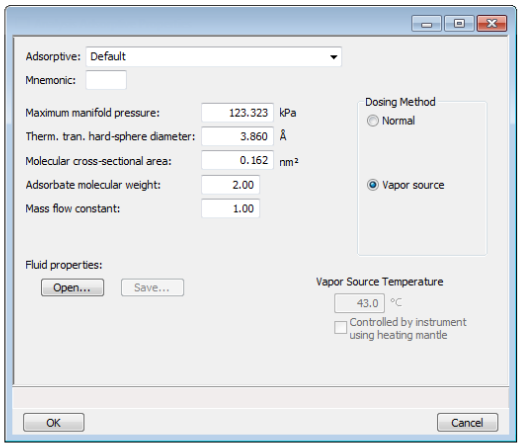
A vapor analysis requires that a vapor source container be installed. See *Install a Vapor Source Container on page 11-14*.

> Micromeritics offers two methods of installing a vapor source container - one method for analyzers with a shelf support and another method for analyzers without a shelf support.

1. Go to ***File > Open > [.SMP file]***, then open the sample file to be used for the analysis.
2. Go to the *Analysis Conditions* tab. Verify that the selected adsorptive is correct. If not, use the down arrow to select the correct adsorptive.
   a. Click **Edit** to the right of the *Adsorptive* down arrow to display the *Analysis Adsorptive Properties* window.
   b. Select *Vapor source* in the *Dosing Method* group box. If running the vapor analysis on Port 3, select *From sample port 3* and proceed to Step 3.
   c. Set the *Vapor Source Temperature* to the correct value if *Vapor Source* is selected. If sample port 3 is selected, the analysis bath temperature is assumed.
   d. Select *Controlled by instrument using heating mantle* if the vapor source is to be automatically heated to this temperature.



Physisorption



Chemisorption

3. Click **OK**, then click **Save** to save any changes.

7 About Reports

Reports can be generated for data collected on a sample that has completed analysis, collected on a sample currently being analyzed, or manually entered.

Open and Close Reports

Reports > Open Report... > [.REP File]

Opens saved reports.

Reports > Close Reports

Closes all open reports. This option is unavailable if reports are being generated.

Start Reports

Reports > Start Report

1. Select one or more .SMP files with a *Complete* status from the library. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. Click **Report**.
2. Select the report destination in the *Report Settings* window, then click **OK**.

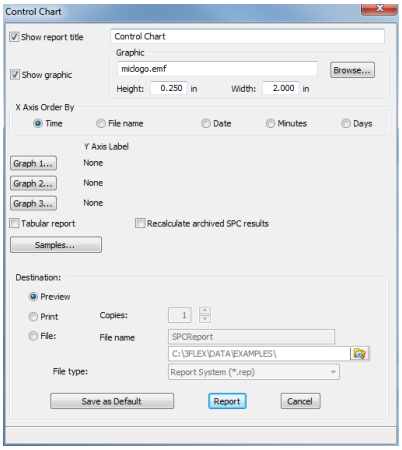
If only one report file was selected in Step 1, the *Selected Reports* window displays allowing the option to select additional reports. Select additional reports as needed, then click **OK**. If multiple files were selected, the reports are displayed in a tiled format.

3. Click a tab at the top of the window to review each report.

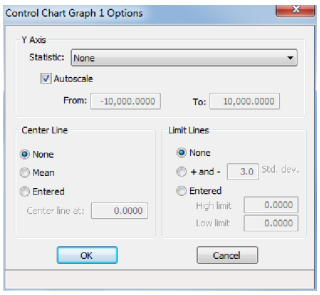
CONTROL CHART REPORT

Reports > Control Chart

Use to generate a Statistical Process Control *(SPC)* control chart report which plots the changes in a statistic.



Control Chart Fields and Buttons Table

Field or Button	Description
Graph [*n*]	Click to define the y-axis of each graph.  • **Statistic.** Displays the SPC variables selected on the ***Reports > SPC Report Options*** window. The selected variable will be plotted against time. This selection also becomes the y-axis label. • **Autoscale.** Allows the y-axis to be scaled automatically. To specify a range, deselect this option and enter a range in the *From* and *To* fields. • **Center Line.** Displays placement options for the center line in the graph. Choose *Entered* to specify placement of the line. • **Limit Lines group box.** Displays limiting lines options. Lines can be placed at some multiple of the standard deviation or at specified positions (*Entered*). When *Entered* is selected, enter the *High limit* and *Low limit* fields with appropriate values.
Recalculate archived SPC results	Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report.  If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application. When this option is selected, the following message displays: **Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.** **Do not show me this message again.**

Control Chart Fields and Buttons Table (continued)

Field or Button	Description
	If *Do not show me this message again* is selected, the message cannot be redisplayed without Micromeritics assistance. The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Report	Generates the report.
Samples	To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Available Files.** Contains files located in the directory specified in the *Look In* text box. • **Selected Files.** Files added from the *Available Files* list box. • **Add / Remove.** Select a file in the *Available Files* list box, then click **Add** to move the file to the *Selected Files* list box. Or select a file in the *Selected Files* list box, then click **Remove** to move the file back to the *Available Files* list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.

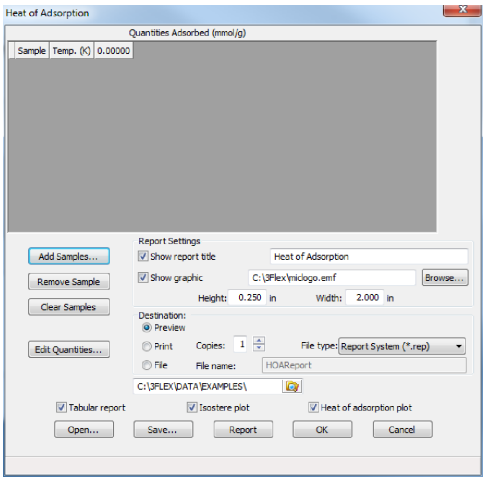
Control Chart Fields and Buttons Table (continued)

Field or Button	Description
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X Axis Order by	Select the order in which x-axis statistics are placed. Sort by: • **Time.** Time the files were analyzed. • **File name.** Alphanumeric order. • **Date.** Date the files were analyzed. • **Minutes.** Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. • **Days.** Number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

HEAT OF ADSORPTION REPORT

Reports > Heat of Adsorption

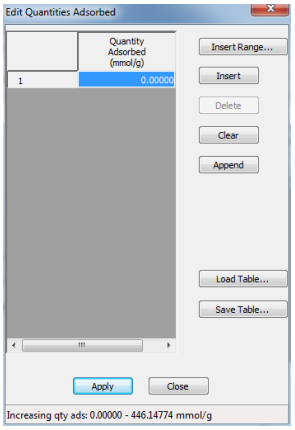


Use to select sample files, define quantities, and generate a *Heat of Adsorption* report. The isosteric heat of adsorption is an important parameter for characterizing the surface heterogeneity and for providing information about the adsorbent and the adsorption capacity. Multiple adsorption isotherms are obtained on the same sample using the same adsorptive but at different temperatures to obtain the heat of adsorption.

Heat of Adsorption Fields and Buttons Table

Field or Button	Description
Add Samples	Adds a sample file to the table. 1. Click **Add Samples**. 2. Double click the file in the *Name* column. Alternatively, select the file name, then click **Open**. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
Clear Samples	Removes all entries from the table.
Edit Quantities	Use to specify the range of surface coverage to include in the report.

Heat of Adsorption Fields and Buttons Table (continued)

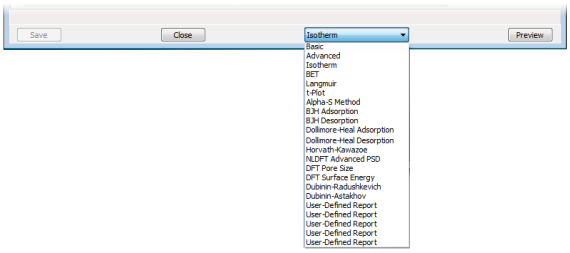
Field or Button	Description
	 **Insert Range.** Click to specify the starting and ending quantities adsorbed and number of points to insert. • **Load Table.** Imports values from another file. • **Save Table.** Saves the current table as a .QNT file. • **Apply.** Applies all table changes.
Heat of adsorption plot	Generates the *Heat of Adsorption* data in a graphical format.
Isostere plot	Generates a graph showing quantities of gas adsorbed versus the temperature.
Remove Sample	Removes the selected sample from the list.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

INTERACTIVE REPORTS

When opening a sample file that contains data from a complete or in-progress analysis, the interactive reporting feature is enabled.

1. When opening a sample file that contains analysis data, a window with the following information displays:
   - an isotherm linear plot and log plot of the data collected during analysis
   - a summary of the analysis giving a single total pore volume and surface area
2. To view the plots in either relative or absolute pressure, select either the *Relative Pressure* or *Absolute Pressure* option.
3. To view the reports selected for generation during the analysis, click **Preview**.
4. From the drop-down list at the bottom of the window:
   - change the option presentation of the sample information window to either *Basic* or *Advanced* to modify certain file parameters, or
   - select another plot from the list and edit the data contained in the plot.



5. When ranges are edited, the changes are reflected immediately in the plots and the summary data displayed in the window. Some editing options are:
   - Drag the blue bars to increase or decrease the range of data included in the plot.
   - Edit the Isotherm Linear Plot to include or omit the data point from the BET plot.
   - Right click to display a popup menu to include reports; enable or select overlays; edit curves, axes, legends, titles; and copy and paste the data in a graph or in tabular format.
6. After editing the report, click **Save** to save the changes in the sample information file.

MICROACTIVE REPORTS

MicroActive reports are generated automatically after an analysis is performed. This feature provides a quick and easy way to investigate and manipulate analysis data using a variety of reporting methods.

When a sample information file with a status of or *Entered* is opened, an isotherm linear plot and log plot of the data collected during analysis are displayed as well as a summary of the analysis giving the total pore volume. Numerous reports are accessible from a drop-down menu, including:

- Summary
- Isotherm
- BET
- Langmuir
- Freundlich (Chemisorption)
- Temkin (Chemisorption)
- *t*-Plot
- Alpha-S Method
- *f*-Ratio Method
- BJH Adsorption
- BJH Desorption
- Dollimore-Heal Adsorption
- Dollimore-Heal Desorption
- Horvath-Kawazoe
- NLDFT Advanced PSD
- DFT Pore Size
- DFT Surface Energy
- Dubinin - Radushkevich
- Dubinin - Astakhov
- MP-Method
- User-defined reports
- Validation
- Difference Method (Chemisorption)
- Sinfelt Method (Chemisorption)

When a report is opened, plots and summary data are displayed, and in some reports certain parameters (for example, thickness curve type, pore geometry, and interaction parameters) are also displayed. Plots may be edited by selecting the data points or data point range to be included in the plots and modifying the parameters. When a report is edited, the results are immediately reflected in the plots and summary data.

EVALUATE REPORT RESULTS

Analysis reports provide a record of test conditions, experimental data, and information extracted from the experimental data by application of various reduction methods. This topic discusses the elements of various reports presented by Micromeritics' static volumetric physical adsorption analyzers and suggest ways by which the merit of the reported information may be evaluated.

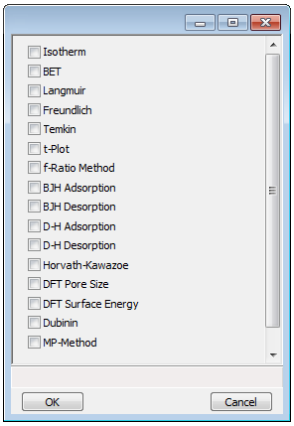
Regardless of the precautions exercised before the analysis, problems still may occur during the analysis, or as a result of using inappropriate parameters or even inappropriate methods. The analysis data should be inspected for evidence of experimental error. The traditional method of confirming the quality of the experiment is to repeat the analysis. Toward that end, Micromeritics' analyzers log and report the exact conditions of each analysis.

Analysis data can be evaluated by:

- Viewing the Validation Report
- Inspecting the Isotherm Plot
- Evaluating the Isotherm Tabular Data Set
- Reviewing Reduced Data

VIEW THE VALIDATION REPORT

The *Validation* report shows whether the data collected during an analysis are within typical ranges. Select the types of reports to include by selecting the report in the *Validation Report Options* window.



When a selected report is generated, if errors occur, a message is displayed across the top portion of the report and a unique symbol displays on the graph.

INSPECT THE ISOTHERM PLOT

Evaluation of data should begin with a visual inspection of the isotherm plot. The plot should be composed of data which have not been subjected to mathematical smoothing as far as possible. If the data describe a Type I isotherm, then the plot is best shown on a logarithmic pressure axis so that details of the low pressure region are revealed. Data in this region are important particularly for micropore studies. Examine the plot to determine if any points are outliers or if a region of the isotherm exhibits characteristics (spikes, steps, etc.) which are inconsistent with the physical process being monitored. The philosophical question of whether or not these suspected extraneous data points should be removed from the raw data is not considered here, but it may be appropriate to exclude an outlier from reduced data. Too many outliers can cause the integrity of the total data set to come under suspicion.

Examine specific reported values to confirm that the isotherm data were collected under reasonable conditions and using reasonable parameters. For example, confirm that the free-space values reported are typical for the sample holder and bath in use. A problem with either warm or cold free space values may indicate a free-space measurement error and affect all calculations of quantity adsorbed.

The raw data should be carefully examined before it is reduced. Errors that occur in raw data will only be exacerbated in reduced data.[1)]

EVALUATE THE ISOTHERM TABULAR DATA SET

Another place to look for reasonableness of the data is the adsorptive uptake by the sample in the BET range (P/P_0 = 0.05 to 0.30). Total uptake is the specific quantity adsorbed (cm^3/g STP) times the sample mass (g). As an example, the level of uncertainty in this range typically is less than 0.1 cm^3 STP for a high performance system. Total uptake quantities should be some multiple of this level of uncertainty. Otherwise, an unfavorable signal-to-noise ratio and unreliable data result. The solution is to use a greater quantity of sample to increase adsorptive uptake.

Another valuable bit of information resides in the tabulated saturation pressure. This pressure is expected to change somewhat over the duration of an analysis, but it is not expected to do so with large or abrupt transitions. Unreasonable saturation pressures or unusual changes may indicate that a gas different from the adsorptive was used in determining P_0, that the level of the cryogen fell too far, or that the cryogen is impure or inappropriate.

With experience, obvious signs of problems can be detected by a quick inspection of the tabular and graphical data. If the data appear satisfactory, the next step is to evaluate the reduced data.[2)]

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1) The information in this article is extracted from Analytical Methods in Fine Particle Technology, Webb, P. and Orr, C., (1997).

2) Most of the information in this article is extracted from Analytical Methods in Fine Particle Technology, Webb, P. and Orr, C., (1997).

REVIEW REDUCED DATA

Isotherm data may be analyzed by any one of several reduction methods depending on instrument model and pressure range employed. The quality of the results depends on the quality of the isotherm, the congruity of the data reduction parameters with experimental conditions, the agreement of the theoretical model with the physical gas-solid system, and compliance to the pressure range over which the method is valid. Typically, results can be appraised by examining a few salient areas of the report as described in the following topics.[1)]

PHYSICAL PARAMETERS

The value of physical parameters which are used only in data reduction routines should be reviewed to assure that they agree with experimental conditions. These parameters can be changed and the experimental data recalculated if an error is discovered or if exploring an alternate value is desired. Analysis condition values used in the calculation of quantity adsorbed can be changed also. These are typically the manually entered free space(s), nonideality correction factor, and bath temperature.

The area occupied by a single adsorbed molecule is a required parameter in the calculation of surface area by the BET and Langmuir methods. The software provides a default value, but other values are found in the literature. McClellan and Harnsberger[2)] provide a comprehensive review of such values.

The volume of pores of a specific size range is calculated from the gas quantity adsorbed in them by converting the quantity to its liquid equivalent volume. This is achieved through use of a density conversion factor calculated from the ratio of molar densities of the condensed adsorbate at bath temperature to the gaseous phase at STP. The necessary information is found in handbooks. The software contains default values for common adsorptives; values for other adsorptives must be calculated.

The terms for liquid surface tension g, contact angle between solid and liquid phase q, molar volume of the adsorbate n, gas constant R, and sample temperature T are treated as one constant, the adsorbate property factor A expressed by:

$$A = \frac{2\gamma\vartheta \quad \cos\theta}{RT}$$

using which, the Kelvin equation[3)] reduces to

1) Most of the information in this article is extracted from Analytical Methods in Fine Particle Technology, Webb, P. and Orr, C., (1997).
2) McClellan, A.L., and Harnsberger, H.F., Journal of Colloid and Interface Science, 23, 577 (1967).
3) Thomson, W., Phil. Mag. S., 42, 448 (1871).

$$In\frac{P^*}{Po} = \frac{A}{r_m}$$

Either surface tension, contact angle, or molar volume can be revised individually to give a new value for the factor A, or A can simply be altered arbitrarily for exploratory purposes.

The thermal transpiration correction requires two parameters which may be adjusted from those of the default values. The first is the inside stem (neck) diameter of the sample holder, and the second is the hard-sphere diameter of the adsorptive molecule. The sample holder inside diameter is available from the documentation provided with it or is measurable. Information on hard-sphere diameters of molecules may be obtained from handbooks.

For terms such as the interaction parameter found in the Horvath-Kawazoe calculation[1)] , the Dubinin affinity coefficient or Astakhov exponent[2)] , the default values as provided by the software generally are adequate. A search of the technical literature is required if the analysis involves a gas-solid system other than that covered by the default values.

The t-Plot method plots quantity adsorbed (Va) against thickness (t) derived from a thickness equation, and the Dubinin transform plots quantity adsorbed against log(P/P0)n. All of these data reduction methods were first proposed for specific applications. The user must make a judgment as to the applicability of the method to a gas-solid system.

If applied appropriately, all transform plots will exhibit a linear range and the regression analysis must be applied only over the linear range and within the range of application. Fitting a regression line to surface area transformation plots should yield a correlation coefficient of 0.9999 or better and for t-plots and Dubinin plots the correlation coefficient should be 0.99 or better.

If the data reduction model does not apply to the gas-solid system under examination, then it may be that either no linear range exists within the pressure range of validity, or that solutions derived from the regression line of the linear range are intuitively incorrect, that is, they have no relevance to the physical situation, such as a negative C-value from a BET transform.

BET C-VALUE

BET theory assumes uniform surface coverage with no favored adsorption sites and it also assumes that the gas is more strongly attracted to the surface than to other gas molecules. The typical range of BET C-values is from about 5 to well over 100. Values much less than 5 imply that the gas-to-gas affinity is competing with the gas-to-solid affinity which conflicts with the basic assumptions of BET theory. C-values much greater than 100 indicate very strong attraction for the surface or preferential adsorption

1) Everett, D.H. and Powl, J.C., J. Chem Soc., Faraday Trans. 1, 72, 619 (1976).
2) Dubinin, M.. and Radushkevich, L.V., Proc. Acad. Sci. USSR, 55, 331 (1947).

Provided the isotherm was determined with negligible error and the regression line to the BET transformation data was fit properly, then an out-of-range C-value probably indicates that the gas-solid interaction for the particular sample material does not conform to the BET model. An inappropriate adsorption model may be indicated also by the coefficient of correlation of the regression line, 0.999 being about the minimum value expected with five more or less equally spaced points. In the case of indications of poor conformance to the BET model, the Langmuir data reduction method should be examined.

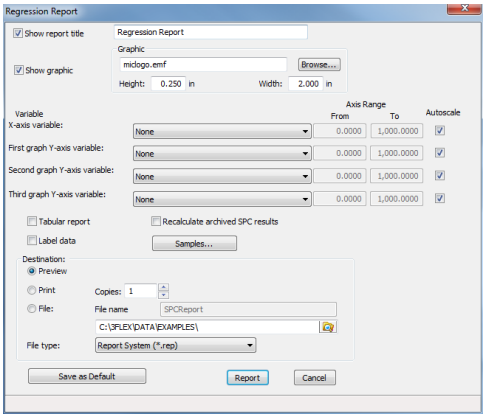
DATA ANALYSES BY THE BJH METHOD

In general, this method visualizes the incremental decomposition of an experimental isotherm, starting at the highest relative pressure or pore size. At each step the quantity of adsorptive involved is divided between pore-emptying and film-thinning processes and is accounted for totally. This computational algorithm frequently leads to inconsistencies when carried to small mesopore sizes. If the thickness curve used is too steep, ultimately it will predict a larger increment of adsorptive for a given pressure increment than is actually observed. The algorithm must stop since a negative pore volume is nonphysical. Accumulated error results in the calculation of a too large volume of (possibly nonexistent) small pores if the thickness curve used underestimates film thinning.

REGRESSION REPORT

Reports > Regression Report

Use to generate a Statistical Process Control (*SPC) Regression* report to determine the interdependency between two variables. Up to three dependent variables (y-axis) may be plotted against a single independent variable (x-axis). The degree of correlation between the variables is also reported.



Regression Report Fields and Buttons Table

Field or Button	Description
Autoscale	When enabled, allows the x- and y-axes to be scaled automatically.
Axis Range	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if *Autoscale* is selected.
Label data	Use to label the points on the plot to correspond with the values in the sample files.
Recalculate archived SPC results	Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report. If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application. When this option is selected, the following message displays: **Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC**

Regression Report Fields and Buttons Table (continued)

Field or Button	Description
	data speeds up future SPC reports. **Do not show me this message again.** If *Do not show me this message again* is selected, the message cannot be redisplayed without Micromeritics assistance. The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Samples	To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Available Files.** Contains files located in the directory specified in the *Look In* text box. • **Selected Files.** Files added from the *Available Files* list box. • **Add / Remove.** Select a file in the *Available Files* list box, then click **Add** to move the file to the *Selected Files* list box. Or select a file in the *Selected Files* list box, then click **Remove** to move the file back to the *Available Files* list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.

Regression Report Fields and Buttons Table (continued)

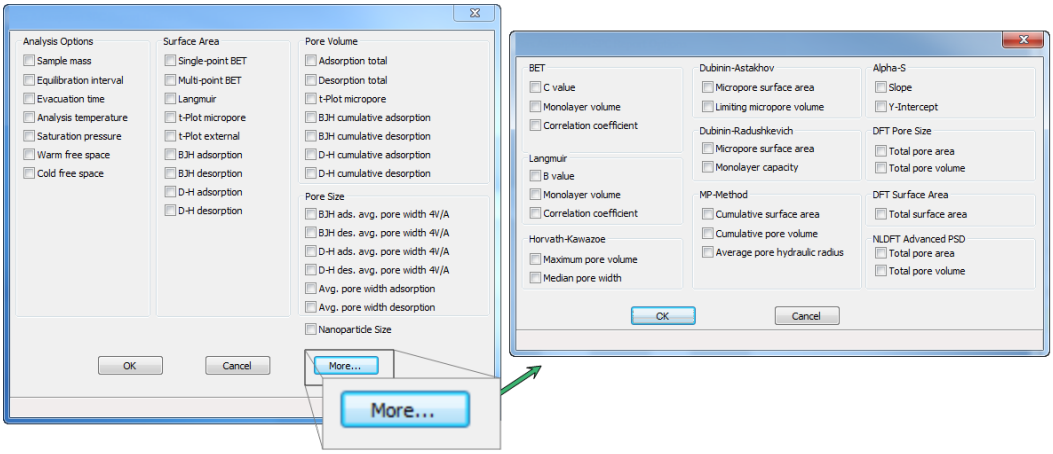
Field or Button	Description
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X- and Y-Axis Variable	Use to designate the x- and y-axes variables. The variables in the drop-down lists are those selected in the ***Reports > SPC Report Options*** window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

SPC REPORT

Reports > SPC Report Options

Use to generate reports with various *SPC* (Statistical Process Control) options. All selected variables must be computed for each sample file used in an SPC report; therefore, it is more efficient to select only the necessary variables.

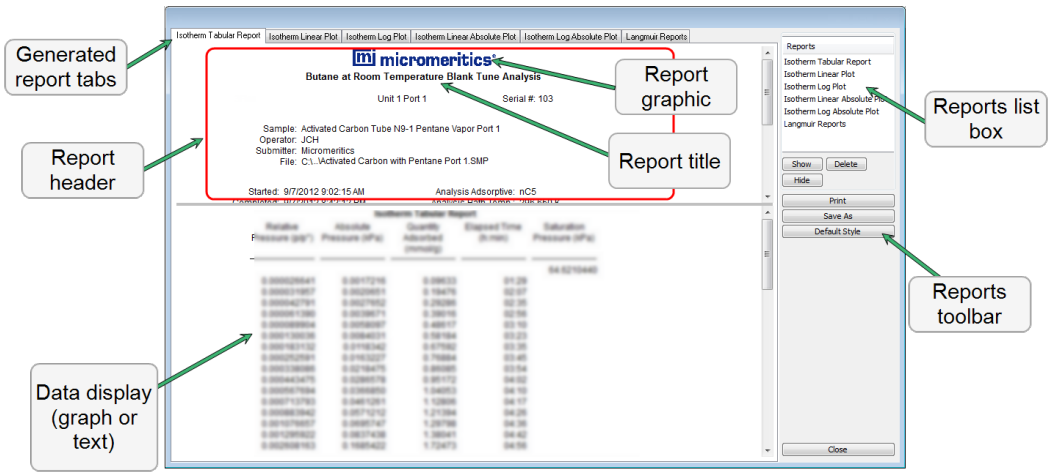


The selected items display as graph variable selections in ***Reports > Regression Report*** and graph selections in ***Reports > Control Chart***. If additional report options are required, click **More**.

REPORT FEATURES AND SHORTCUTS

Reports can be customized and manipulated using the toolbar, shortcut menus, the zoom feature, or axis cross-hairs.

- After analysis, reports can be viewed, printed, and / or copied and pasted into other documents.
- The report zoom feature provides the viewing of fine graph details and the ability to shift the axes.
- All reports contain a header displaying file statistics.

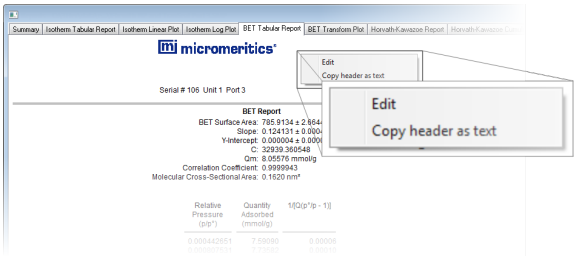


If configured, the report header can also contain a graphic and / or a title.

- Tabular and graphical reports contain sample and analyzer statistics such as analysis date / time, analysis conditions, etc.
- The headers contain notes of sample file changes occurring after analysis.
- Summary report headers contain the same information as tabular and graphical reports with the exception of notes.

REPORT HEADER SHORTCUTS

Display header shortcuts by right clicking in the report header.

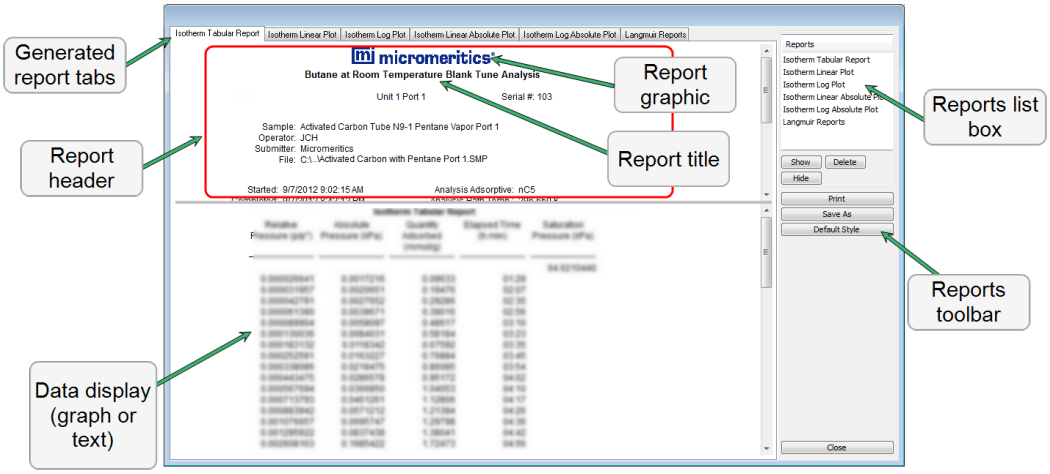


Report header Shortcut Field and Button Table

Field or Button	Description
Copy header as text	Use to copy the report header as text. Text is copied to the clipboard and then can be pasted into other documents.
Edit	Use to edit the report title and / or graphic in the report header.

REPORT TOOLBAR

The *Report* window has a toolbar on the right portion of the window and selectable tabs at the top of the report header. To view a specific report, either select the tab or the report in the *Reports* list box, then click **Show**.



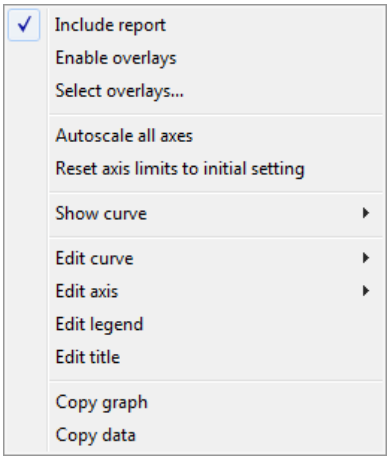
Report Toolbar Fields and Buttons Table

Field or Button	Description
Default Style	Click to specify default report parameters for fonts and curve properties. • **Font Type.** Use to edit the font type and attributes for the selected item. Select an item in the list, click **Edit**, and select from various font options. • **Thickness**. Enter a thickness number for the curve. • **Histogram Fill Style.** Select a histogram fill option. • **Graph border line thickness.** Enter a thickness number for the graph border.
Delete	Deletes the selected report in the *Reports* list box. Deleted reports will have to be regenerated if deleted in error.
Hide	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the *Reports* list box.
Print	Displays the *Print* window for report output. • **Name** drop-down list and **Properties**. Select the printer from the drop-down list and click **Properties** to change printer setup, etc. • **Copies.** Select the number of copies and collate option. • **Current.** Selects the active report (or selected tab). • **All.** Selects all reports in the *Reports* list box. • **Shown.** Selects only the reports not hidden. • **Clear.** Clears all selections.
Reports list box	Contains a list of all generated reports. The same reports display as tabs at the top of the report header unless the report has been hidden using the **Hide** button.
Show	Displays the selected or hidden report in the *Reports* list box.

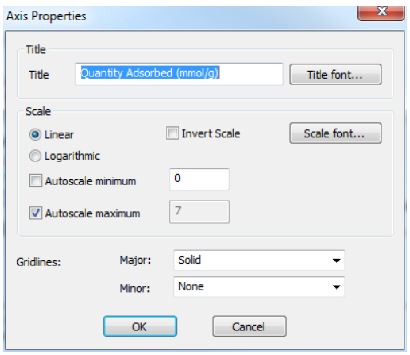
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

GRAPH FEATURES AND SHORTCUTS

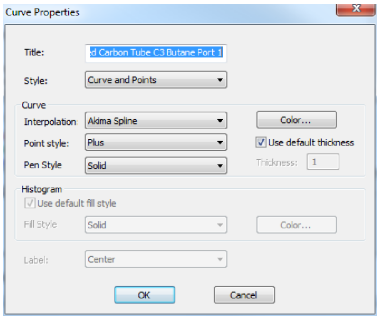
Display graph report shortcuts by right clicking in the body of the graph report.



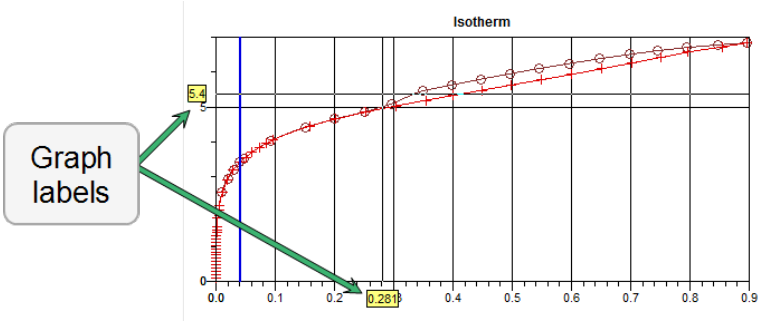
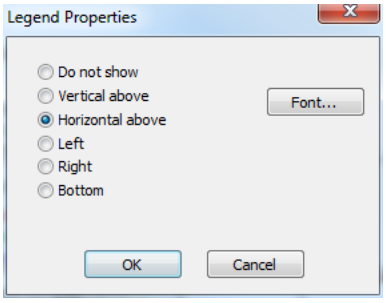
Graph Shortcuts Options and Description Table

Field or Button	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Copy Data	Copies the report data to the clipboard. It can then be pasted into other software programs as tab-delimited columns or copied as an overlay onto another graph.
Copy Graph	Copies the graph to the clipboard. It can then be pasted into other software programs.
Display imported data	Used with pore distribution data reports only. Use to hide or show imported or pasted ASCII text data on the active graph.
Edit axis	Use to edit the selected axis properties.  • **Title.** Use to edit the selected axis label.

Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
	• **Title font.** Use to modify the font for the selected axis label. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*. • **Linear / Logarithmic.** Select the option to scale the graph as linear or logarithmic. • **Autoscale minimum / maximum.** To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box. • **Invert scale.** Use to invert the scale. • **Scale font.** Use to modify the font for the scale label. Deselect *Use default font* to enable font options. • **Grid lines.** Use to change how to display major / minor grid lines.
Edit curve	Use to edit selected curve properties.  • **Title.** Use to change the title of the selected curve. • **Style.** Use to select another style for the collected data curve. • **Curve group box.** Use to change the interpolation, point style and pen style for the selected curve. These options are disabled if *Use default fill style* is selected in the *Histogram* group box. **Color.** Click to change the curve color. **Use default thickness.** Uses the default curve thickness. Deselect to enter a new thickness number in the *Thickness* text box. • **Histogram group box.** Enabled only if *Histogram* is selected in the *Style* drop-down list. Use to specify the type of fill, fill color

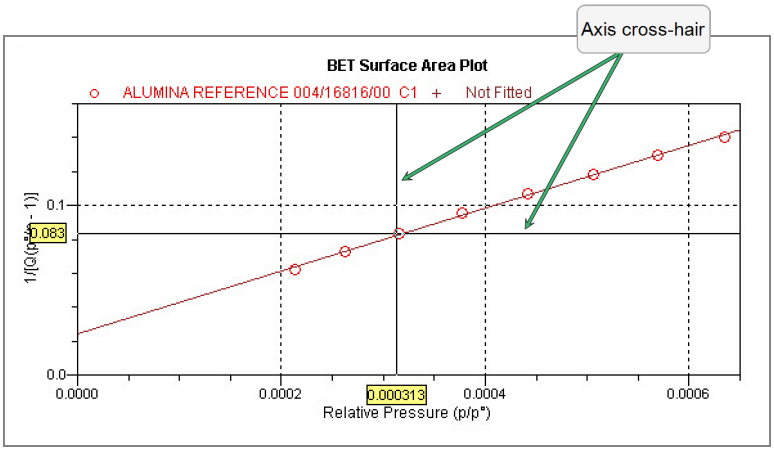
Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
	and label position for the selected curve. • **Label.** Select where the graph point labels will display (left, right, center, etc.) on the SPC report.
	
Edit imported data	Used with pore distribution data reports only. Use to select ASCII text files for import onto the active graph.
Edit legend	Use to change the legend location and font. Click **Font** to modify font attributes. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*. 
Edit title	Use to change the graph title and font. Click **Font** to font attributes. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.
Enable Overlays	If overlays have been selected, this option displays (or hides) the overlays.
Include report	When selected, places a checkmark to the left of the report in the *Select Reports* list box on the *Report Options* tab.
Paste Data	Used with pore distribution data reports only. Use to paste ASCII text data from the clipboard onto the the active graph.

Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
Select overlays...	Displays the option to select files to overlay onto the active graph. To view the overlays, ,click *Enable Overlays* on the shortcut menu.
Show curve	Displays a list of all curves. Select the curve(s) to display.

Axis Cross-hair

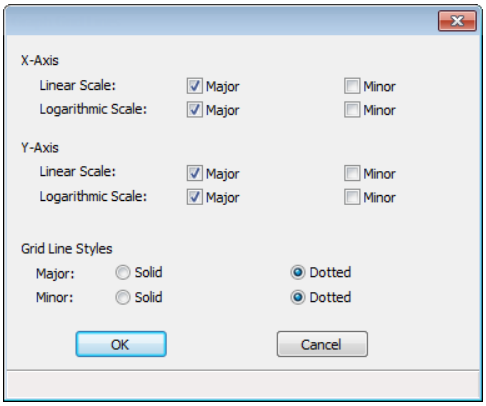


The cross-hair feature displays axis coordinates.

1. Left click on the graph to view the cross-hair coordinates.
2. To remove the cross-hair, right click in the graph area and select either *Autoscale all axes* or *Reset axis limits to initial setting.*

Graph Grid Lines

Options > Graph Grid Lines



Use to select how grid lines appear on reports. This menu option is not available if using *Restricted* option presentation.

Graph Grid Lines Fields and Buttons Table

Field or Button	Description
Grid Line Styles	Select if the major and / or minor grid lines should appear as solid or dotted lines.
X-Axis / Y-Axis	Select major and / or minor lines to display in reports for the logarithmic and linear scales. Deselect this option to remove the grid lines.

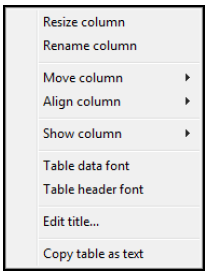
Zoom Feature

Use the zoom feature to examine graph details.

1. Open the graph.
2. Hold down the left mouse button, then drag the mouse pointer across the graphical area to be enlarged. A box will display in the area to be enlarged.
3. Release the mouse button. The enlarged area fills the graph area. To return to normal view, right click in the graph area, then select either *Autoscale all axes* or *Reset axis limits to initial setting* on the shortcut menu.

TABULAR REPORT FEATURES AND SHORTCUTS

Display tabular report shortcuts by right clicking in the body of the tabular report. Column shortcuts require right clicking on the column to be modified.



Tabular Reports Shortcut Options and Descriptions Table

Field or Button	Description
Align column	Select to change the column alignment to either left, right, or centered.
Copy table as text	Use to copy the report contents to the clipboard as tab-delimited text. It can then be pasted into another document.
Edit title	Use to edit the report title and / or title font attributes. Click **Font** to modify font attributes. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.
Move column	Right click the column to be moved. Select *Move column* on the shortcut menu and select *Left* or *Right* for the move.
Rename column	Right click the column to be renamed. Select *Rename column* on the short-cut menu and enter the new column name.
Resize column	Right click the column to be resized. Select *Resize column* on the shortcut menu and enter the new column width in inches.
Show column	Displays a list of all columns. Click a column to add a checkmark to show the column or remove the checkmark to hide the column.
Table data font	Right click in the report data. Select *Table data font* on the shortcut menu. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.
Table header font	Right click in the report data. Select *Table header font* on the shortcut menu. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.

GRAPH AND SAMPLE OVERLAYS

Use the graph overlay functions to compare multiple graph options. Graphical lines are differentiated by the use of varying colored symbols outlined on a legend. Overlays may be generated in two ways:

- **Multiple Graph Overlays.** Overlay two different types of graphs from one sample.
- **Multiple Sample Overlays.** Overlay graphs of the same type with that of the current plot.

This feature is available only when using *Advanced* option presentation.

GENERATE PORE-SIZE DISTRIBUTION GRAPH OVERLAYS

The overlay process allows the importing of pore-size distribution data from an ASCII text file. The ASCII text file must follow the format rules outlined below.

Multiple graph overlays can only be generated for:

- BJH Adsorption / Desorption
- Dollimore-Heal Adsorption / Desorption
- Horvath-Kawazoe
- DFT Pore Size

ASCII text file format rules:

- he header must consist of one line to include title, two unit specifications, and distribution type:
  - Accepted pore dimension units are: A, nm, um
  - Accepted pore volume units are: cm3/g, cm^3/g, ml/g
  - Accepted distribution types are: cumulative, incremental

  Two examples of a header format:

  My Title (A, cm3/g, incremental)

  My Title (A, cm3/g, cumulative)

- The data must be in two columns and should be separated by a comma or white-space.
- The data lines must be ordered so that pore dimensions are monotonically increasing or decreasing.

Sample ASCII text file

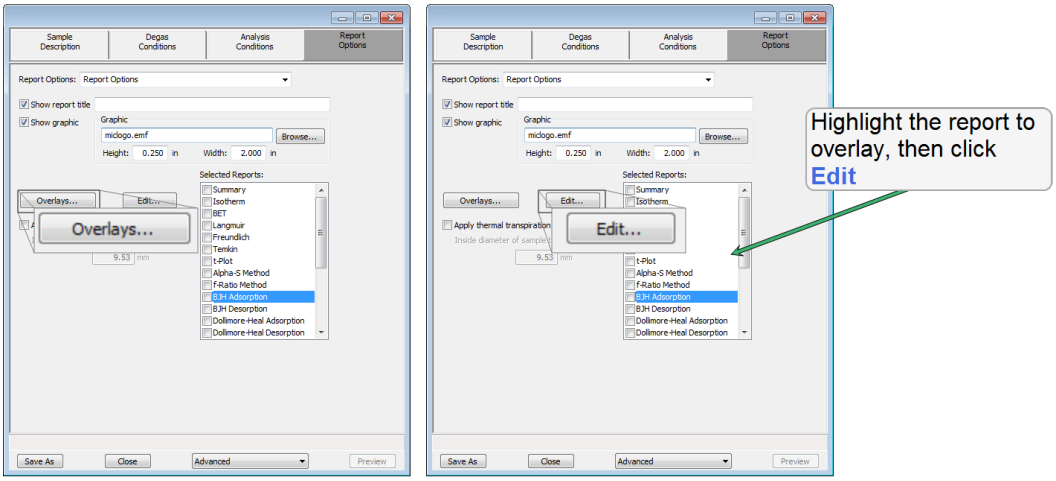
silica alumina bjh	(A, cm3/g, cumulative)
456.657	0.0133559
444.847	0.0546427
429.168	0.0869924
425.419	0.119721
419.629	0.132681
360.634	0.156611
340.859	0.197672
326.601	0.233092

To import ASCII text files to generate graph overlays:

The following steps use BJH Adsorption as an example. Window appearance will vary depending on the selected report. This function can be performed on samples files with a *Completed* status or during an analysis.

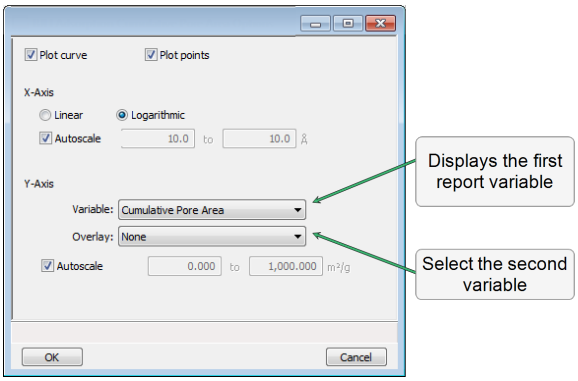
1. Go to ***File > Open***. Select a sample file to overlay graphs onto other samples. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. Click **Open**.
2. Select *Advanced* from the drop-down list at the bottom of the window.
3. Select the *Report Options* tab, then click **Overlays** to browse for the .TXT file.

   If the ASCII text file does not display on the *Plot Overlay Sample Selection* window, click **Import**. Locate the file, then click **Open**. Header information from the ASCII text file will then appear in the *Select Imported Overlays* window. Select the entry, then click **OK**. If an error message appears instead, verify that the .TXT file format is correct. Select the entry, then click **OK**.



4. On the *Report Options* tab, highlight the type of report in the *Selected Reports* list box to overlay with a graph, then click **Edit**.

5. On the *Report Options* window, highlight the type of report in the *Selected Reports* list box to overlay with a graph, then click **Edit**.
6. Click the down arrow at the *Variable* field and select a variable to overlay. Click the down arrow of the *Overlay* field, then select *Imported Data*. Click **OK** to return to the *Report Options* window.

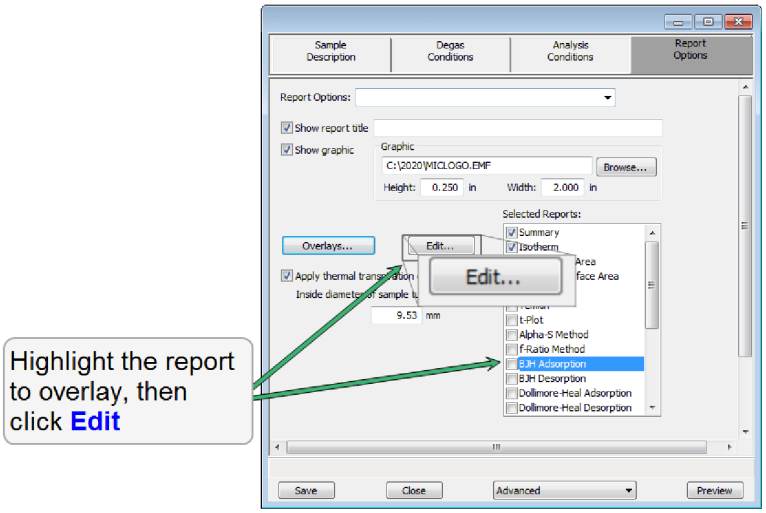


7. Click **OK** again to return to the *Report Options* tab.
8. Click **Save As** to save the selections.
9. To view the report, click **Preview.**

OVERLAY MULTIPLE SAMPLE FILES

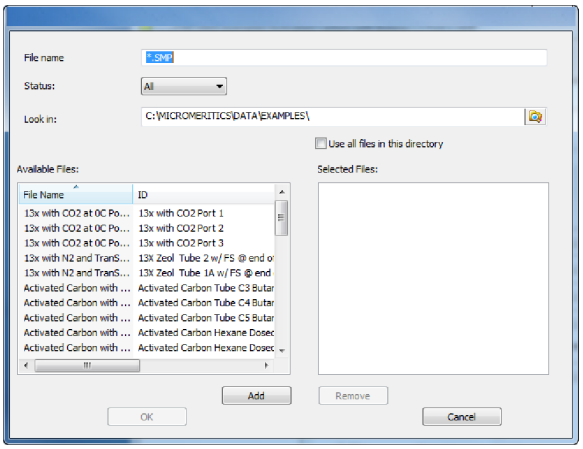
To overlay the same type of graph on multiple samples:

1. Go to ***File > Open***.
2. Select a .SMP file, then click **Open**. If the Isotherm plot displays, select *Advanced* from the drop-down list at the bottom of the window to display the tabbed window view.
3. Click the *Report Options* tab.
4. In the *Selected Reports* list box, highlight a report then click **Edit**. Use the following table to complete the process for the selected report.



If overlaying this type of report...	Then...
• **Isotherm** (for physisorption)	a. On the *Isotherm Report Options* window, select one or more plots in the *Selected Reports* group box, then click **Options** to the right of the selected plot. b. On the *Plot Options* window, select *Plot curve* and / or *Plot points* if they are to be included in the overlay. If the x- and / or y-axes are to be autoscaled, enable *Autoscale*; otherwise, enter the *From* and *To* points for the axes. Click **OK** to save and close the window. c. On the *Isotherm Report Options* window, in the *Plot Options* group box, select *Plot overlays*. Click **OK**. d. Continue to Step 5.
(for physisorption) • **Alpha-S Method** • **BET Surface Area** • ***f*-Ratio Method** • **Freundlich** • **Langmuir Surface Ara** • ***t*-plot** • **Temkin** (for chemisorption) • **Difference Method** • **Freundlich** • **Langmuir** • **Sinfelt Method** • **Temkin**	a. On the pop-up window, select *Overlay samples*. Verify other fields. Click **OK** to return to the *Report Options* tab. b. Continue to Step 5.
(for physisorption) • **BJH Adsorption** • **BJH Desorption** • **Dollimore-Heal Adsorption** • **Dollimore-Heal Desorption** • **MP-Method**	a. Select the report variable from the *Selected Reports* group box, then click **Edit**. b. Click the down arrow on the *Overlay* field, then select the *Samples* option. Verify other fields. Click **OK** to return to the *Report Options* window. c. Click **OK** again to return to the *Report Options* tab.

5. On the *Report Options* tab, click **Overlays**.
6. On the *Plot Overlay Sample Selection* window, use one of the following options to move up to 25 files from the *Available Files* box to the *Selected Files* box:



- Double click a file name in the *Available Files* box to move the file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, double click the file name in the *Selected Files* box, or
- Select a file name in the *Available Files* box. Click **Add** to move the selected file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, select a file name in the *Selected Files* box, then click **Remove**. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.

6. Click **OK**.
7. To view the report, click **Preview**.

REPORT EXAMPLES

T-PLOT REPORT EXAMPLE

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Unit 1 Port 2 Serial #: 101 Page 11

Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started: 8/17/2012 8:44:16 PM
Completed: 8/18/2012 6:33:42 PM
Report Time: 8/30/2012 6:34:58 AM
Sample Mass: 0.2555 g
Cold Free Space: 57.0915 cm³
Low Pressure Dose: None
Automatic Degas: Yes

Analysis Adsorptive: N2
Analysis Bath Temp.: -196.058 °C
Thermal Correction: No
Warm Free Space: 16.0907 cm³ Measured
Equilibration Interval: 10 s
Sample Density: 1.000 g/cm³

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240

t-Plot Report

Micropore Volume: 0.001429 cm³/g
Micropore Area: 7.9925 m²/g
External Surface Area: 209.1934 m²/g
Slope: 13.517568 ± 0.050021 cm³/g·Å STP
Y-Intercept: 0.923393 ± 0.218961 cm³/g STP
Correlation Coefficient: 0.999959
Surface Area Correction Factor: 1.000
Density Conversion Factor: 0.0015476
Total Surface Area (BET): 217.1859 m²/g
Thickness Range: 3.5000 Å to 5.0000 Å
Thickness Equation: Harkins and Jura

Thickness Curve

t = [13.99 / (0.034 - log(p/p°))] ^ 0.5

t-Plot Report - Data

Relative Pressure (p/p°)	Statistical Thickness (Å)	Quantity Adsorbed (cm³/g STP)	Fitted
0.053665461	3.2751	45.1706	
0.077824186	3.4987	48.2830	
0.106574940	3.7285	51.3824	*
0.135877231	3.9408	54.2113	*
0.163237219	4.1275	56.6908	*
0.188595088	4.2948	58.9330	*
0.213852389	4.4582	61.1303	*
0.238707954	4.6176	63.3032	*
0.263405375	4.7758	65.4875	*
0.288407930	4.9369	67.7418	*
0.313104034	5.0979	70.0202	
0.357549162	5.3950	74.3238	
0.397683828	5.6746	78.4880	
0.446861650	6.0373	84.0635	
0.496397055	6.4319	90.4326	
0.545717570	6.8629	97.8310	
0.594513636	7.3377	106.8632	
0.607888353	7.4780	109.7720	
0.620922246	7.6196	112.7842	
0.633541428	7.7617	115.9539	
0.645692079	7.9033	119.2176	
0.657391993	8.0446	122.6982	

BET SURFACE AREA

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Unit 1 Port 2 Serial #: 101 Page 9

Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started:	8/17/2012 8:44:16 PM	Analysis Adsorptive:	N2
Completed:	8/18/2012 6:33:42 PM	Analysis Bath Temp.:	-196.058 °C
Report Time:	8/30/2012 6:34:58 AM	Thermal Correction:	No
Sample Mass:	0.2555 g	Warm Free Space:	16.0907 cm³ Measured
Cold Free Space:	57.0915 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Sample Density:	1.000 g/cm³
Automatic Degas:	Yes		

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240

BET Surface Area Report

BET Surface Area:	217.1859 ± 0.2311 m²/g
Slope:	0.019839 ± 0.000021 g/cm³ STP
Y-Intercept:	0.000205 ± 0.000003 g/cm³ STP
C:	97.887751
Qm:	49.8911 cm³/g STP
Correlation Coefficient:	0.9999977
Molecular Cross-Sectional Area:	0.1620 nm²

Relative Pressure (p/p°)	Quantity Adsorbed (cm³/g STP)	1/[Q(p°/p - 1)]
0.077824186	48.2830	0.001748
0.106574940	51.3824	0.002322
0.135877231	54.2113	0.002901
0.163237219	56.6908	0.003441
0.188595088	58.9330	0.003944
0.213852389	61.1303	0.004450

BET Surface Area Plot

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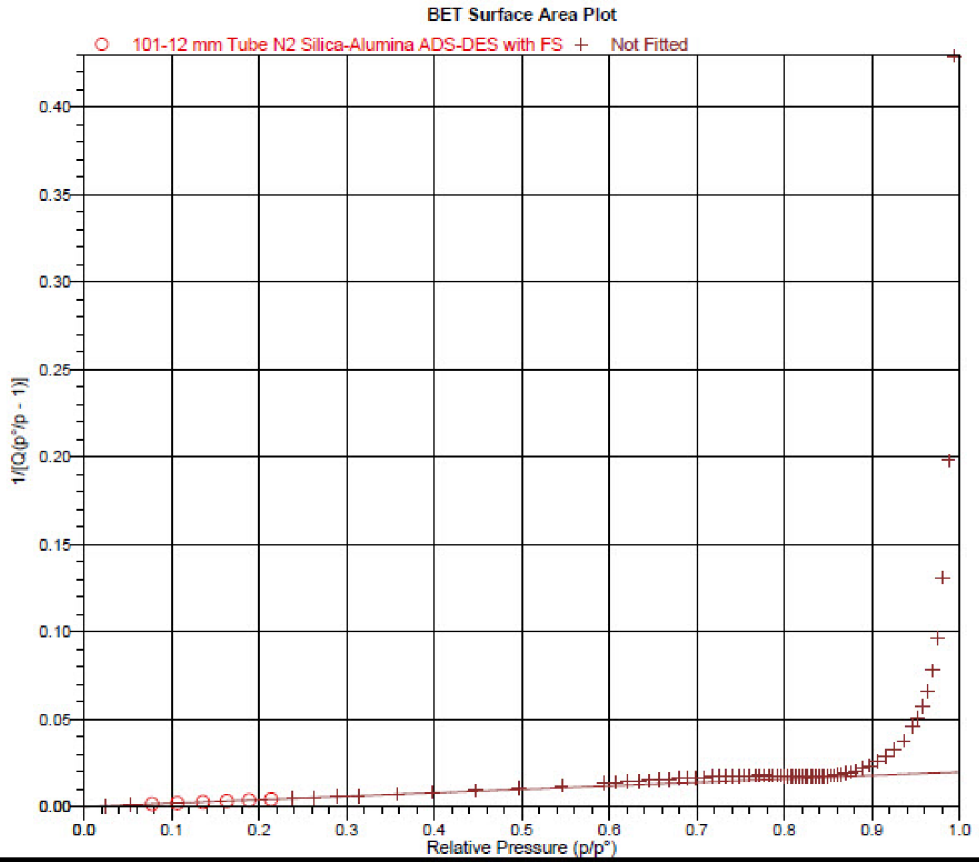
Unit 1 Port 2 Serial #: 101 Page 10

Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started:	8/17/2012 8:44:16 PM	Analysis Adsorptive:	N2
Completed:	8/18/2012 6:33:42 PM	Analysis Bath Temp.:	-196.058 °C
Report Time:	8/30/2012 6:34:58 AM	Thermal Correction:	No
Sample Mass:	0.2555 g	Warm Free Space:	16.0907 cm³ Measured
Cold Free Space:	57.0915 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Sample Density:	1.000 g/cm³
Automatic Degas:	Yes		

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240



ISOTHERM LINEAR PLOT

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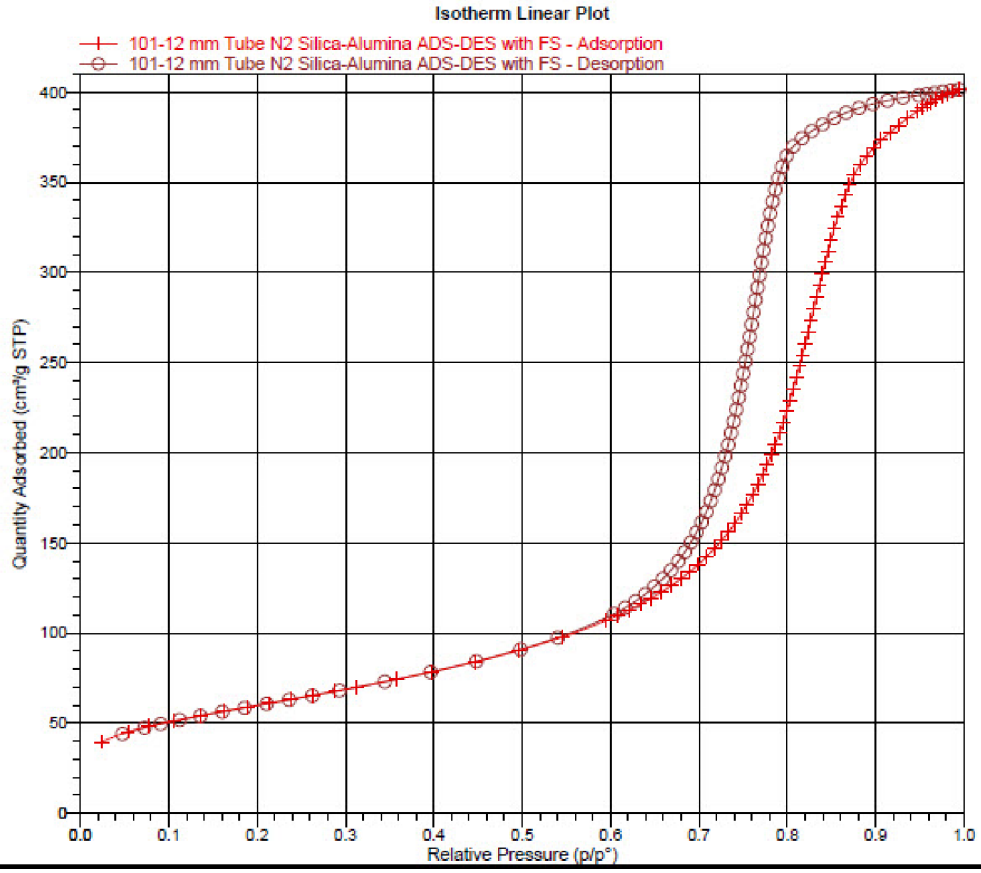
Unit 1 Port 2 Serial #: 101 Page 8

Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started:	8/17/2012 8:44:16 PM	Analysis Adsorptive:	N2
Completed:	8/18/2012 6:33:42 PM	Analysis Bath Temp.:	-196.058 °C
Report Time:	8/30/2012 6:34:58 AM	Thermal Correction:	No
Sample Mass:	0.2555 g	Warm Free Space:	16.0907 cm³ Measured
Cold Free Space:	57.0915 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Sample Density:	1.000 g/cm³
Automatic Degas:	Yes		

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240



BJH DESORPTION: CUMULATIVE PORE VOLUME

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Unit 1 Port 2 Serial #: 101 Page 24

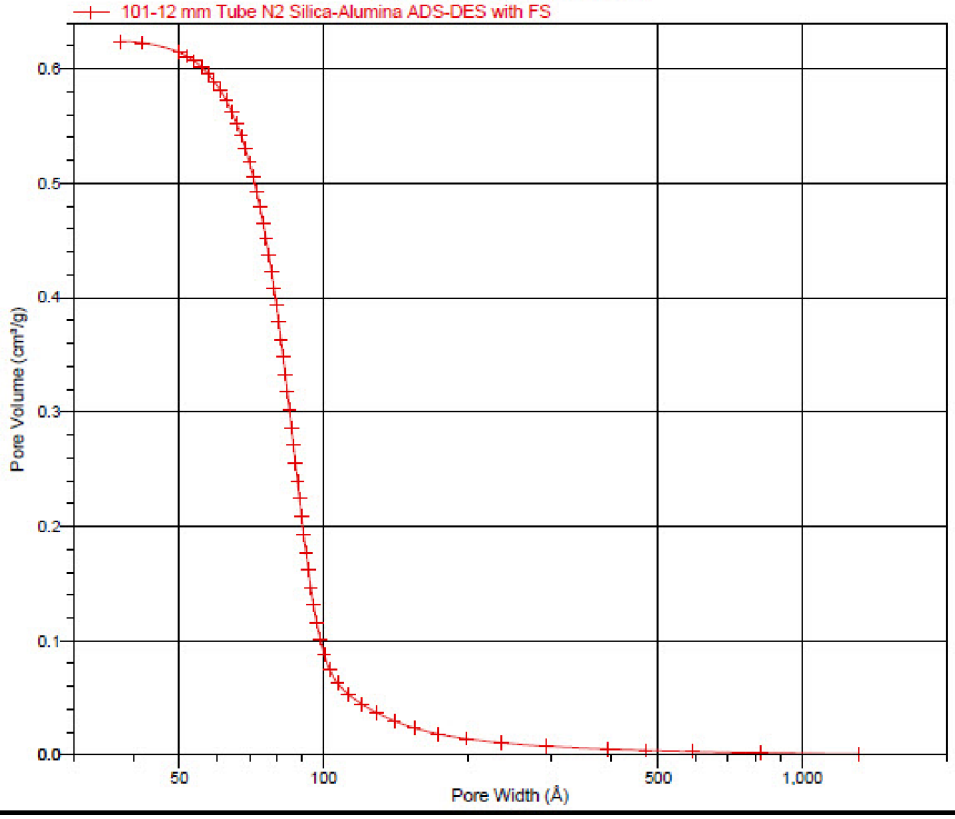
Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started:	8/17/2012 8:44:16 PM	Analysis Adsorptive:	N2
Completed:	8/18/2012 6:33:42 PM	Analysis Bath Temp.:	-196.058 °C
Report Time:	8/30/2012 6:34:58 AM	Thermal Correction:	No
Sample Mass:	0.2555 g	Warm Free Space:	16.0907 cm³ Measured
Cold Free Space:	57.0915 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Sample Density:	1.000 g/cm³
Automatic Degas:	Yes		

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240

BJH Desorption Cumulative Pore Volume (Larger)

Harkins and Jura : Faas Correction



BJH ADSORPTION: CUMULATIVE PORE VOLUME

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Unit 1 Port 2 Serial #: 101 Page 16

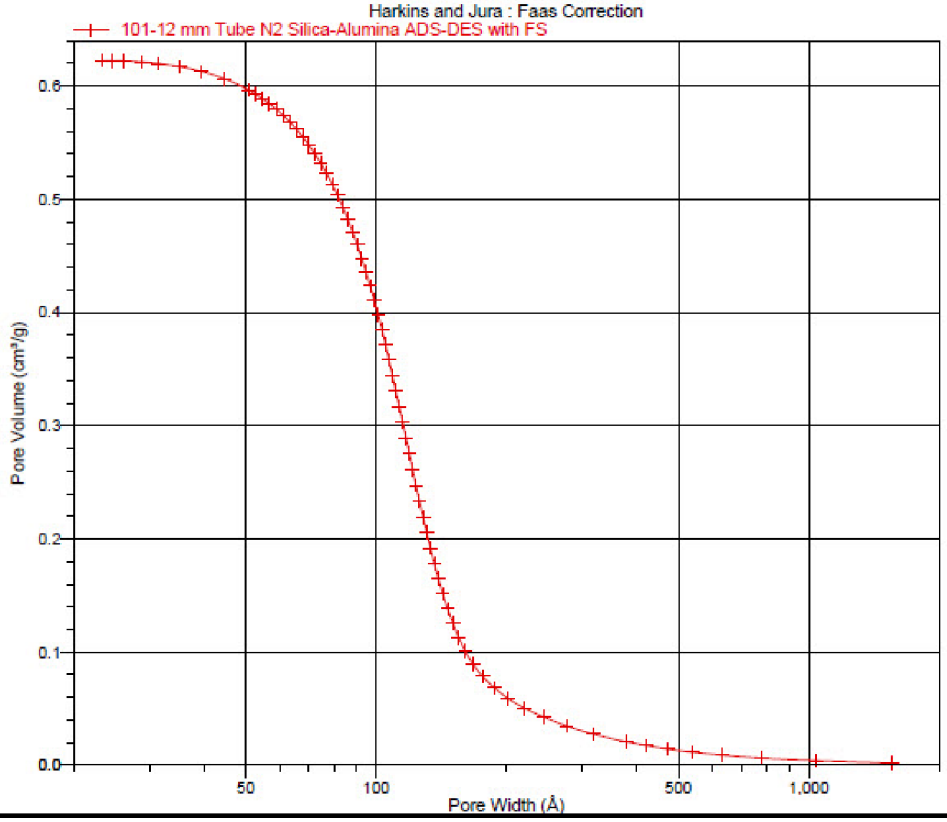
Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started:	8/17/2012 8:44:16 PM	Analysis Adsorptive:	N2
Completed:	8/18/2012 6:33:42 PM	Analysis Bath Temp.:	-196.058 °C
Report Time:	8/30/2012 6:34:58 AM	Thermal Correction:	No
Sample Mass:	0.2555 g	Warm Free Space:	16.0907 cm³ Measured
Cold Free Space:	57.0915 cm³	Equilibration Interval:	10 s
Low Pressure Dose:	None	Sample Density:	1.000 g/cm³
Automatic Degas:	Yes		

Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240

BJH Adsorption Cumulative Pore Volume (Larger)

Harkins and Jura : Faas Correction



8 Report Options

File > Open > [.RPO File]

(or click the *Report Options* tab when in *Advanced* option presentation)

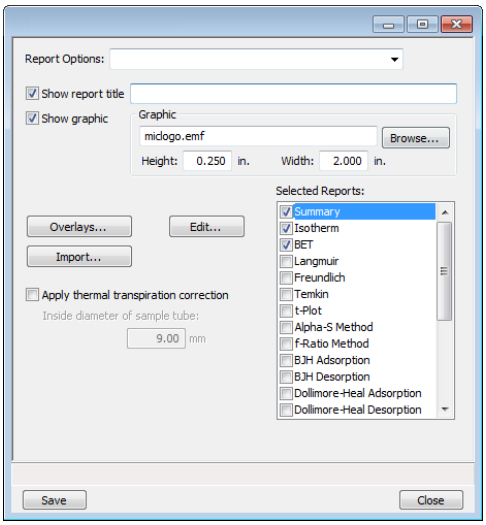
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

Use to specify report options for collected (from an analysis) or manually entered data. *Report Options* files also help in customizing report details such as axis scale, axis range, column headings, and components of thickness curve equations.

Reports can be generated for data:

- collected on a sample that has completed analysis
- collected on a sample currently being analyzed
- manually entered

Customized report options files can be created then loaded into a sample file, allowing quick generation of reports.



Report Options Fields and Buttons Table

Field or Button	Description
Apply thermal transpiration correction	Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Always use thermal transpiration when performing micropore analyses. See *Thermal Transpiration Correction on page B - 49*. • **Inside diameter of sample tube.** Enabled when *Apply thermal transpiration correction* is selected. Enter the inside diameter of the sample tube used in the analysis. If filler rods are used, enter the filler rod capillary diameter of 1 mm instead.
Import (for physisorption)	Import up to 25 pore distribution data files. These datasets are shown only in BJH and Dollimore-Heal reports.
Name column	Displays a list of files in the selected directory.
Overlays	See *Graph and Sample Overlays on page 7 - 29*.
Report Options drop-down list	Browse for a .RPO file that contains report options parameters to be used in the report.
Selected Reports list box	Select the report names to include in the report.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

SELECTED REPORTS

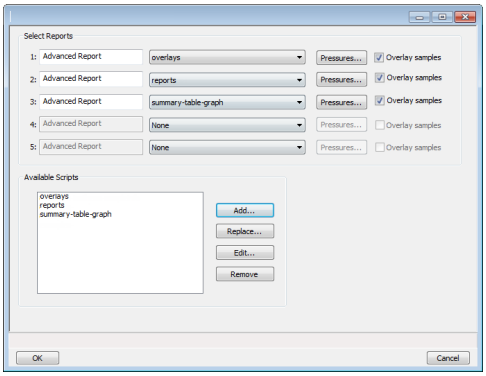
ADVANCED REPORT OPTIONS

See *Python Module - Advanced Reports on page H - 1*

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

Up to five Advanced reports, each with up to 10 summary reports, 10 tabular reports, and 10 graphical reports can be created. To use this feature, a file containing a Python script that imports a "mic" Python module must be created. An example of Python script and functions for the "mic" Python Module can be found in the Appendix section of this manual.

1. Create the Python script and save it in the *Scripts* directory.
2. Open a Sample File with a *Completed* status.
3. Select *Advanced* at the bottom of the window to return to the tabbed view.
4. On the *Report Options* tab, select *Advanced* in the *Selected Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add** in the *Available Scripts* group box to locate and select the Python script. Repeat for each script to be added.



6. In the *Selected Reports* group box, click the drop-down arrows to select up to five Python scripts previously added in the *Available Scripts* box.
7. Click **Pressures** to add pressure points to the report.
8. Click **OK** to return to the *Report Options* tab.
9. On the *Report Options* tab window, click **Preview**. The Python Reports will be included on the tabs across the top portion of the *Reports* window.
10. Select the *Overlay samples* checkbox to enable the overlay sample feature.

Advanced Report Options Fields and Buttons Table

Field or Button	Description
Add	Click to add additional Python reports.
Available Scripts	Lists the available reports and provides the option to add, replace, edit or remove reports.
Overlay samples	Use to overlay samples as defined by the function.
Advanced Report 1 through 5	Use the drop-down lists to select currently-defined functions used to define the report calculations and output.

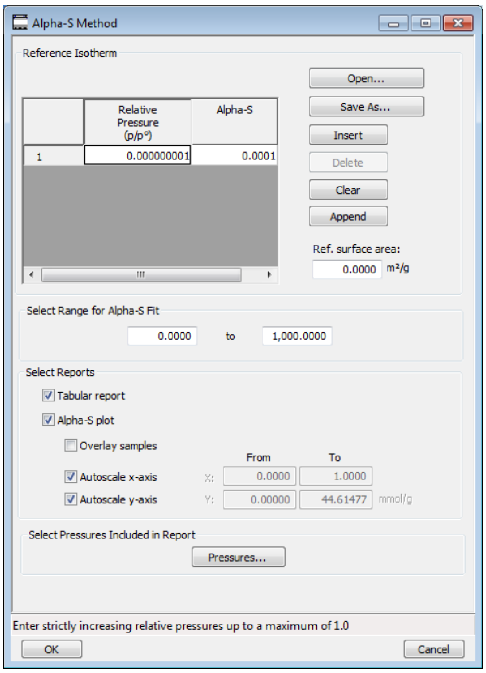
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

ALPHA-S METHOD REPORT OPTIONS

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

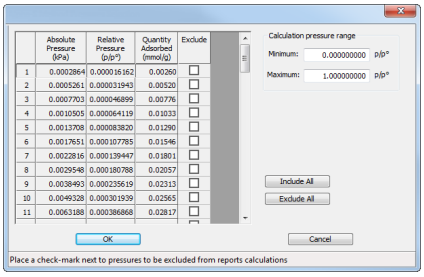
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *Alpha-S* plot converts the standard adsorption isotherm into a dimensionless isotherm using the quantity adsorbed at a relative pressure of 0.4.



One predefined curve is located in the *Reference* file directory. Use the table buttons to enter relative pressure and the alpha-s values.

Alpha-S Method Report Options Fields and Buttons Table

Field or Button	Description
Alpha-S plot	Use to plot data in graph format. • **Overlay samples.** Use to overlay sample files on the plot. • **Autoscale x-axis.** The x-axis field shows the relative pressure. • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.
Open	To import values from an existing thickness curve (.ALS file), click **Open** and select the file containing the values. The table to be imported must be saved as ASCII text with a .ALS file extension. It must have a two-column format with the relative pressures in the first column and the alpha-s values in the second column. Columns must be separated by a space or a tab.
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Ref. surface area	Enter the surface area from the reference curve. This value is used to calculate the sample surface area.

Alpha-S Method Report Options Fields and Buttons Table (continued)

Field or Button	Description
Select Range for Alpha-S Fit	Enter minimum and maximum relative pressures to determine the fit.

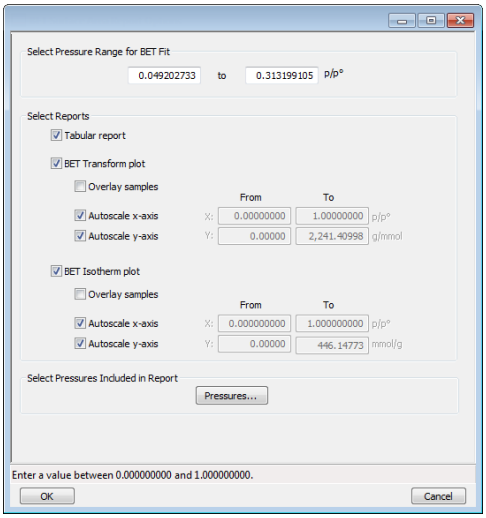
Selected Reports	• **Tabular Report.** Use to have a tabular report of data generated.
	• **Alpha-S Plot.** Use to plot data in graph format.
	  ○ **Overlay samples.** Use to overlay sample files on the plot.
	  ○ **Autoscale x-axis.** The x-axis field shows the relative pressure.
	  ○ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

BET Surface Area Report Options

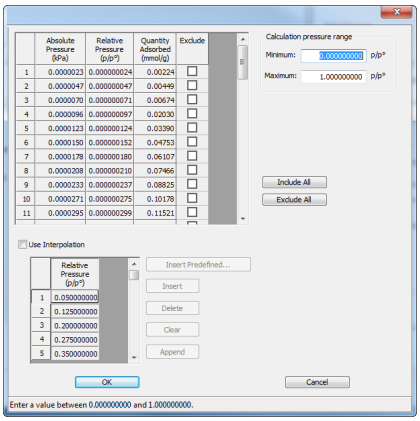
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.



The BET calculation obtains the sample surface area value by determining the monolayer volume of adsorbed gas from the isotherm data. BET uses a multilayer model.

BET Report Options Fields and Buttons Table

Field or Button	Description
Pressures	This option is available when the sample file has a status of *Analyzing* or *Complete*. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table if not using the *Use Interpolation* option. • **Use Interpolation.** Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only. • **Insert Predefined.** Click to insert a predefined (default) set of points into the report. *Use Interpolation* must be selected to enable this button. This button displays for BET reports only. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Select Pressure Range for BET fit	Enter values to indicate the fitted pressure range.

Selected Reports	• **Tabular report.** Use to have a table of measured and calculated values generated.
	• **BET Transform plot.** Use to generate a traditional BET surface area plot used to determine monolayer volume and BET C constant.
	  ○ **Overlay samples.** Use to overlay sample files on the BET transform plot.
	  ○ **Autoscale x-axis**. Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET.
	  ○ **Autoscale y-axis**. The y-axis field shows BET transformation.

BET Report Options Fields and Buttons Table (continued)

Field or Button	Description
	• **BET Isotherm plot.** Uses BET monolayer volume and constant to produce an isotherm. ◦ **Overlay samples.** Use to overlay sample files on the BET isotherm plot. ◦ **Autoscale x-axis**. Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET. ◦ **Autoscale y-axis**. The y-axis field shows the quantity of gas adsorbed.
	For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

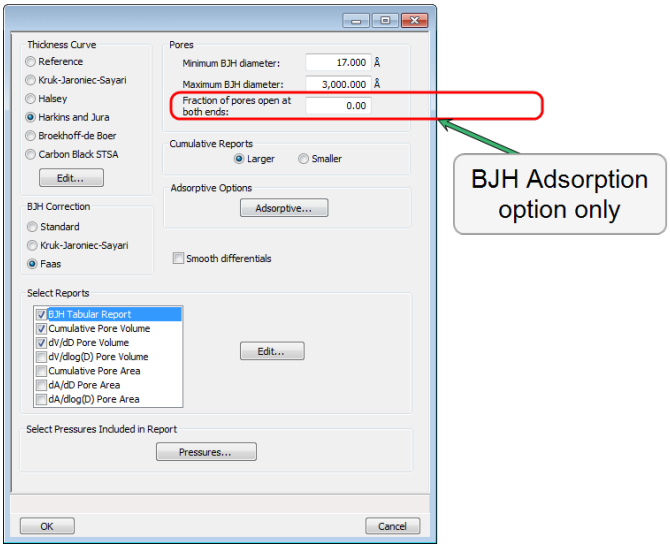
BJH Adsorption / Desorption Report Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

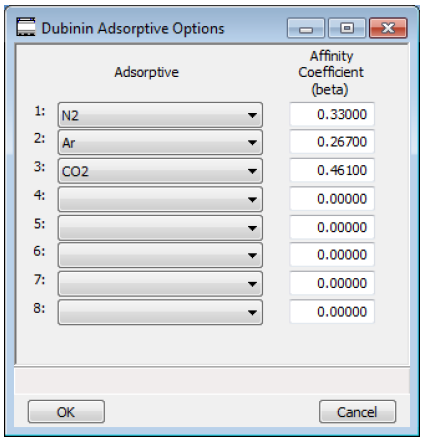
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The BJH calculation determines the mesopore volume / area distribution, which accounts for both the change in adsorbate layer thickness and the liquid condensed in pore cores. Reports can be generated from both adsorption and desorption data. The fields for both *BJH Adsorption Report Options* and *BJH Desorption Report Options* are identical unless otherwise specified.

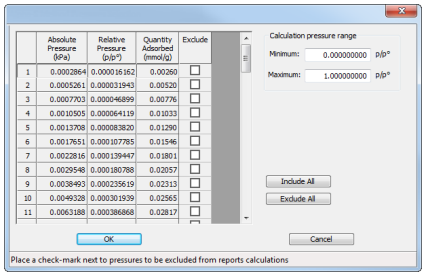
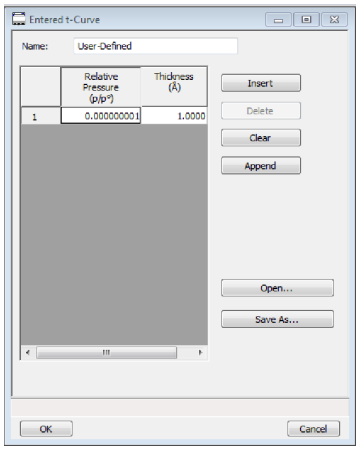
An incomplete pore distribution may be generated if a thickness curve selection is not a good match for the sample being analyzed.



BJH Adsorption / Desorption Report Options Fields and Buttons Table

Field or Button	Description
Adsorptive	Displays the *Adsorptive Options* window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified. 
BJH Correction	Select the type of correction to apply to calculations. The selected type displays in the report header. • **Standard.** Uses original BJH models. • **Kruk-Jaroniec-Sayari.** Good for reference thickness curves. • **Faas.** Good for statistical thickness curves.
Cumulative Reports	• **Larger.** Use to report the total volume found in pores larger than the current pore size. • **Smaller.** Use to report the total volume found in pores smaller than the current pore size.
Pores	Enter the minimum and maximum diameter (radius or width) of pores to include in the BJH reports. • **Fraction of pores open at both ends.** This field is not available for the *BJH Desorption Report Options* window. During adsorption calculations, the analysis program assumes that all pores are closed at one end. Occasionally, a percentage of pores may be open at both ends causing disagreement in the adsorption and desorption data or in the values for total volume and total BJH pore volume. Enter the fraction of pores open at both ends to compensate for this error.
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.

BJH Adsorption / Desorption Report Options Fields and Buttons Table (continued)

Field or Button	Description
	 • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Select Reports	Select the report names to include in the report. Highlight the report name, then click **Edit** to modify report parameters.
Smooth differentials	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.
Thickness Curve	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Reference.** Select *Reference*, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory. 

BJH Adsorption / Desorption Report Options Fields and Buttons Table (continued)

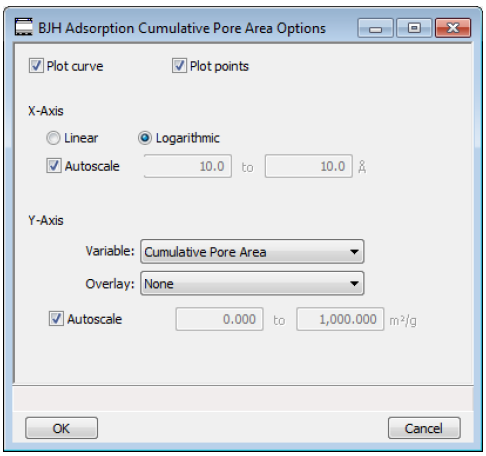
Field or Button	Description
	To import values from an existing thickness curve (.THK file), click **Open**, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space or a tab. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

BJH Plot Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.



The fields for all plot options are identical for specifying plotting methods and customizing plots. Highlight any plot option in the *Selected Reports* list box in the *BJH Report Options* window, then click **Edit**.

BJH Plot Options Fields and Buttons Table

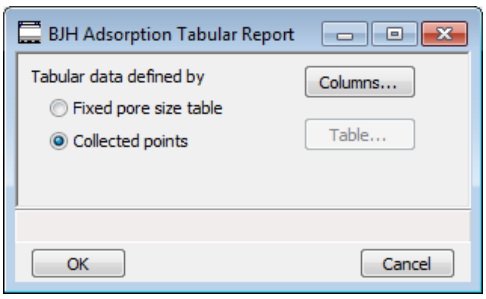
Field or Button	Description
Autoscale	When enabled on the report parameters windows, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y-ranges will be set so that all the data is shown. If *Autoscale* is not selected, the entered range is used.
Plot curve / Plot points	Select to plot points on the graph.
X-Axis	Use to have the x-axis on a logarithmic or linear scale.
Y-Axis	• **Variable.** Select a variable. • **Overlay**. Select an option to overlay onto the current report.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

BJH Tabular Report Options

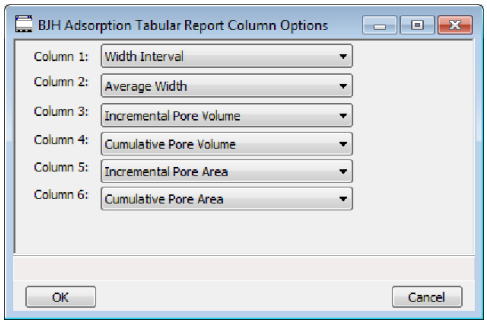
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.



Highlight *BJH Tabular Report* in the *Selected Reports* list box on the *BJH Adsorption Report Options* window, then click **Edit** to specify the method of data reduction.

BJH Tabular Report Options Fields and Buttons Table

Field or Button	Description
Collected points	Use to include all relative pressure points collected. Refer to the **Columns** button below.
Columns	Select the data types to include in the report. *Column [n]* indicates the column order and data contents for the report. 
Fixed pore size table	Use to specify exact pore sizes for volume or area data.Click **Table** to modify the fixed pore size table. Refer to **Table** and **Columns** buttons elsewhere in this table.
Table	The fixed pore size table must contain a minimum of two points. The points must be strictly decreasing. Enabled only when *Fixed pore size*

BJH Tabular Report Options Fields and Buttons Table (continued)

Field or Button	Description
	table is selected.

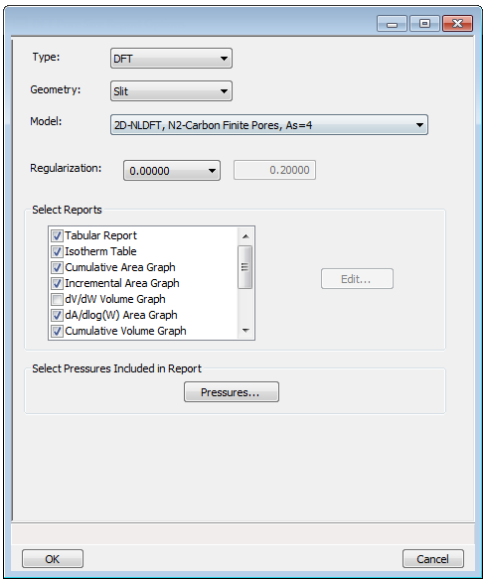
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

DFT Pore Size Report Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

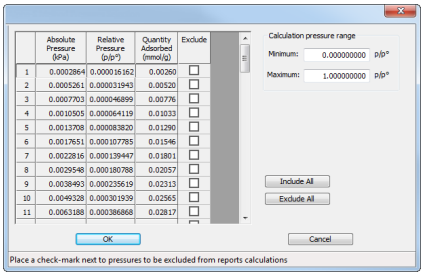
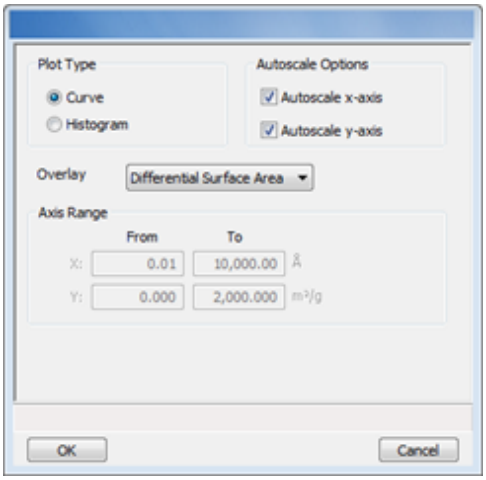
The *DFT Pore Size* report contains the results of pore size distribution analyses using a non-local DFT range of micro and mesopore ranges.



DFT Pore Size Report Options Fields and Buttons Table

Field or Button	Description
Geometry	Select the pore shape.
Model	Lists the models that meet the specified criteria and match the adsorbate and temperature of the sample data. If no models appear, no models meet the selected criteria. One model must be selected.
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.

DFT Pore Size Report Options Fields and Buttons Table (continued)

Field or Button	Description
	 • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Regularization	Select the extent of smoothing to apply to the data. If *0.20000 (user)* is selected, enter a number in the text box giving a relative weight for the smoothing during deconvolution. Larger values produce more smoothing.
Select Reports	Select the reports to generate. To edit graph details, highlight the graph option and click **Edit**. The *Log Goodness of Fit* and *Goodness of Fit* graphs cannot be edited.  • **Plot Type.** Select the method for data display. • **Autoscale Options.** Use to autoscale the x-axis and / or y-axes. • **Overlay.** Select an overlay for the report.

DFT Pore Size Report Options Fields and Buttons Table (continued)

Field or Button	Description
	• **Axis Range.** *From* / *To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and / or y-axes. ◦ **X-axis**. Shows the pore size. ◦ **Y-axis.** Shows the area.
Type	• **DFT.** Model based on the density functional theory. • **Classical.** Model based on the Kelvin equation and thickness for determining the pore size distribution. See ***DFT Models on page C - 1***.

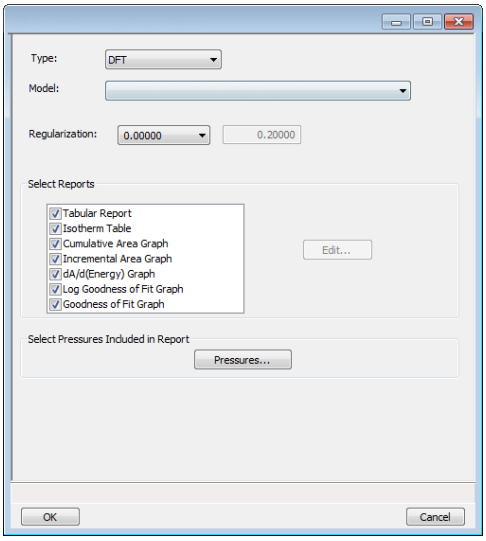
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

DFT SURFACE ENERGY REPORT OPTIONS

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *DFT Surface Energy* report contains the results of surface energy distribution analyses.



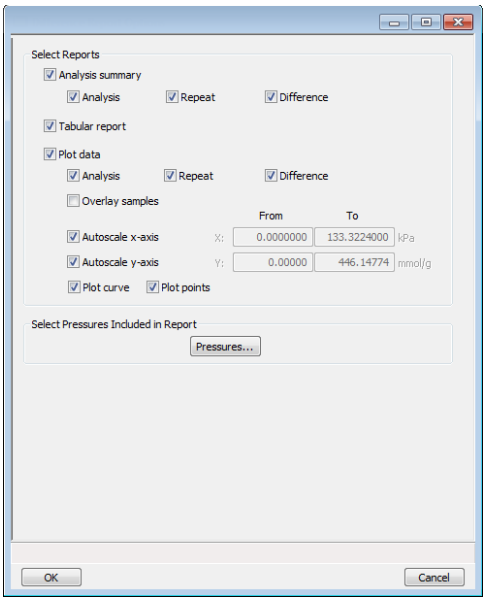
DFT Surface Energy Report Options fields and buttons are identical to the *DFT Pore Size Report Options*. See ***DFT Pore Size Report Options on page 8 - 18***.

DIFFERENCE METHOD REPORT OPTIONS

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

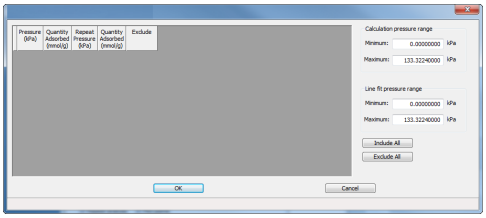
The *Difference Method Report* and the *Sinfelt Method Report* windows are identical unless otherwise specified.



The y-intercept quantity adsorbed (Q_0) is used for several calculations in the *Difference* and *Sinfelt* reports. This value can be determined in two ways. If one point selected, Q_0 is the quantity adsorbed for that point.

- **Difference Method.** The repeat isotherm data are subtracted from the primary isotherm. Q_0 is the y-intercept of a straight line through the difference data.
- **Sinfelt Method.** Both the primary and repeat isotherms are fitted to a straight line. Q_0 is the difference between the y-intercepts of the fit lines.

Difference and Sinfelt Report Options Fields and Buttons Table

Field or Button	Description
Analysis summary	• **Analysis.** Generates a summary of the following for the first analysis: ○ Percent metal dispersion ○ Metallic surface area ○ Volume adsorbed ○ Slope ○ Correlation coefficient • **Repeat.** (Sinfelt report only). Generates a line fit plot for the secondary analysis. • **Difference.** Generates a summary of the differences between the following information for the first and repeat analyses: ○ Percent metal dispersion ○ Metallic surface area ○ Average difference volume
Pressures	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Line fit pressure range.** Enter the minimum and maximum pressures for line fit. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Plot data	• **Analysis.** Includes a line fit plot for the primary analysis. • **Repeat.** Includes a line fit plot for the secondary analysis. • **Difference.** Plots the difference between the analysis and repeat analysis lines. • **Overlay samples.** Overlays data from the current sample with that of other samples. Click **Overlays** on the *Report Options* window to choose other sample files.

Difference and Sinfelt Report Options Fields and Buttons Table (continued)

Field or Button	Description
	• **Autoscale x-axis / Autoscale y-axis.** Select to have the X- and / or Y-axes automatically scaled. The application uses the highest values collected during analysis as the ending points for an axis range. X-axis shows the pressure. Y-axis shows the quantity of gas adsorbed. • **Plot curve** and **Plot points**. Use to specify how to plot data. Plot data as a curve, points, or both.
Tabular report	Select to have a report of the pressure points generated.

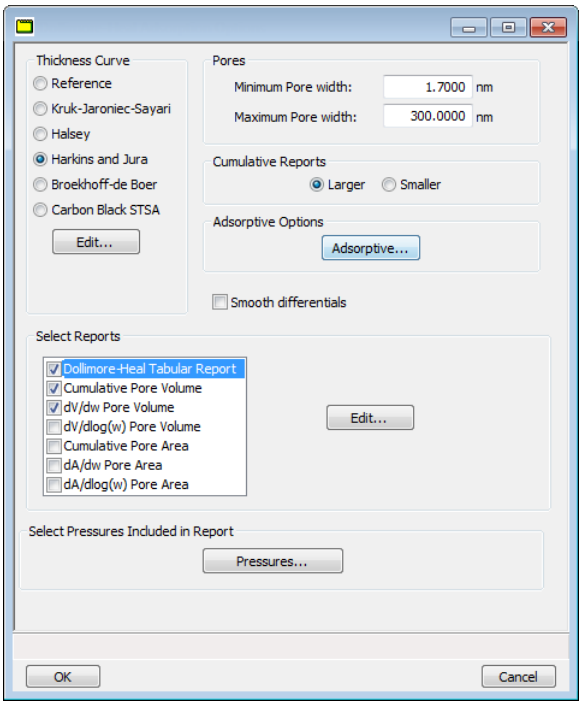
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

DOLLIMORE-HEAL ADSORPTION / DESORPTION REPORT OPTIONS

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

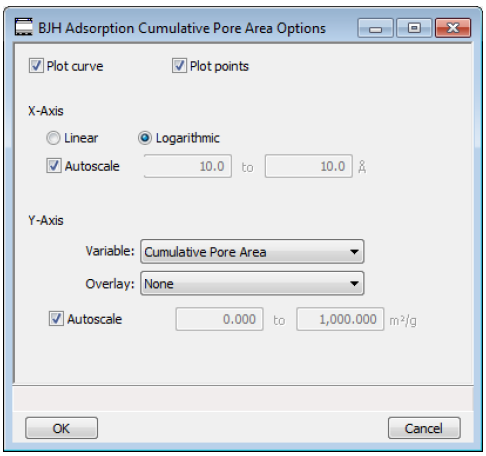
The *Dollimore-Heal Adsorption Report Option* and the *Dollimore-Heal Desorption Report Option* generate reports from both adsorption and desorption data. The fields and buttons for these reports are identical to the *BJH Adsorption / Desorption Report Options*. See ***BJH Adsorption / Desorption Report Options on page 8 - 11***.



Dollimore-Heal Plot Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

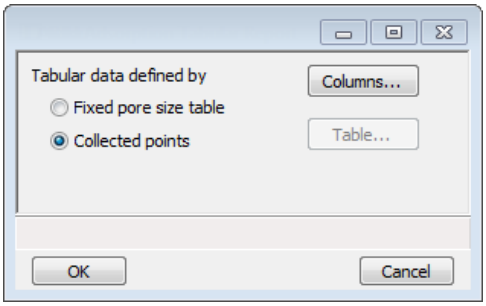
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.



The fields for all plot options are identical for specifying plotting methods and customizing plots. Highlight any plot option in the *Selected Reports* list box in the *BJH Report Options* window, then click **Edit**. The fields and buttons for these reports are identical to the *BJH Plot Report Options*. See ***BJH Plot Options on page 8 - 15***.

Dollimore-Heal Tabular Report Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.



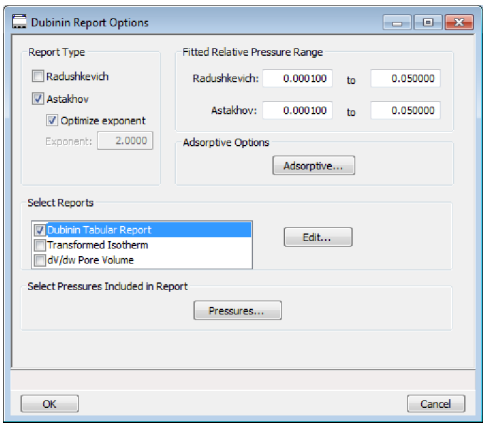
Dollimore-Heal Tabular Report Options are identical to the *BJH Tabular Report Options*. See ***BJH Tabular Report Options on page 8 - 16***.

DUBININ REPORT OPTIONS

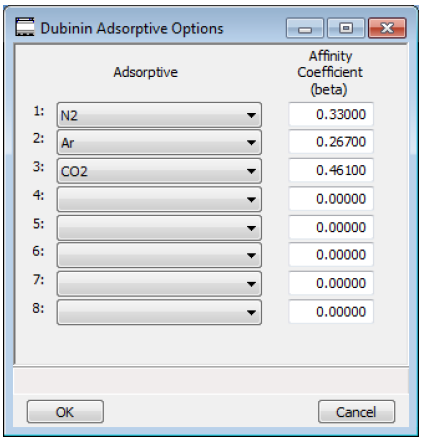
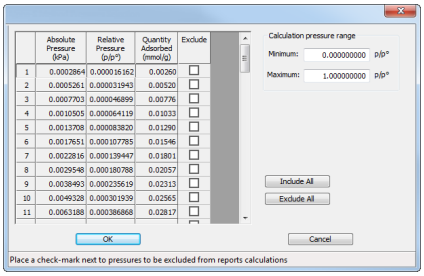
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *Dubinin* method provides pore volume distributions for microporous materials by making use of an expression for the adsorption potential.



Dubinin Report Options Fields and Buttons Table

Field or Button	Description
Adsorptive	Displays the *Adsorptive Options* window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified. 
Fitted Relative Pressure Range	Enter the minimum and maximum limits for Radushkevich or Astakhov relative pressures included in the line fit.
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.

Dubinin Report Options Fields and Buttons Table (continued)

Field or Button	Description
Report Type	Select report types. If *Astakhov* is selected, either select *Optimize exponent* or enter an appropriate exponent value in the text box.
Select Reports	Select the reports to generate. Highlight the report, then click **Edit** to modify report options.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

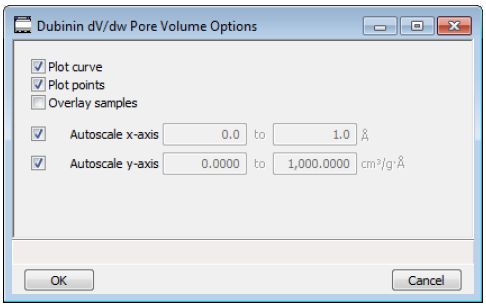
Dubinin Pore Volume Report Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

In the *Dubinin Report Options* window, highlight *dV/dw Pore Volume* in the *Selected Reports* list box, then click **Edit**.

This option plots differential pore volume as a function of pore width.



Dubinin Pore Volume Report Fields and Buttons Table

Field or Button	Description
Autoscale x-axis / Autoscale y-axis	Select an option to have the x- and / or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges. Enable to enter beginning and ending values manually.
Overlay samples	Use to overlay sample files on the plot.
Plot curve / Plot points	Select to plot points on the graph.

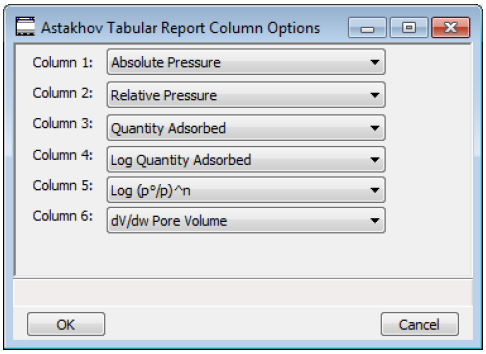
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

Dubinin Tabular Report Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

In the *Dubinin Report Options* window, highlight *Dubinin Tabular Report* in the *Selected Reports* list box, then click **Edit**. *Column [n]* indicates the column order and data contents for the report.



Log (p^o/p)^n. The value for *[n]* is the optimized exponent if *Optimize exponent* is selected on the *Dubinin Report Options* window. If not, then the value for *[n]* is the entered exponent value.

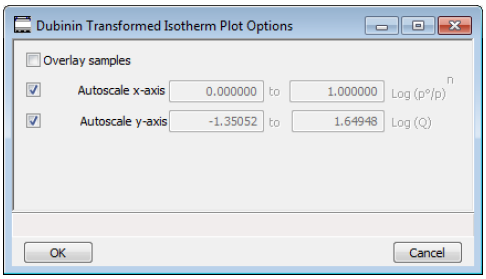
Dubinin Transformed Isotherm Plot Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

Highlight *Transformed Isotherm* in the *Selected Reports* list box in the *Dubinin Report Options* window, then click **Edit**.

The transformed Dubinin isotherm is the logarithm of quantity adsorbed as a function of the log of relative pressure raised to a power. Isotherms for which the Dubinin method is applicable produce straight lines when transformed in this way.



Dubinin Transformed Isotherm Plot Options Fields and Buttons Table

Field or Button	Description
Autoscale x-axis / Autoscale y-axis	Select an option to have the x- and / or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges. Deselect to enter beginning and ending values manually. • **Autoscale x-axis.** Shows the quantity of gas adsorbed at standard temperature and pressure. • **Autoscale y-axis.** Shows the log of relative pressure.
Overlay Samples	Use to overlay sample files on the plot.

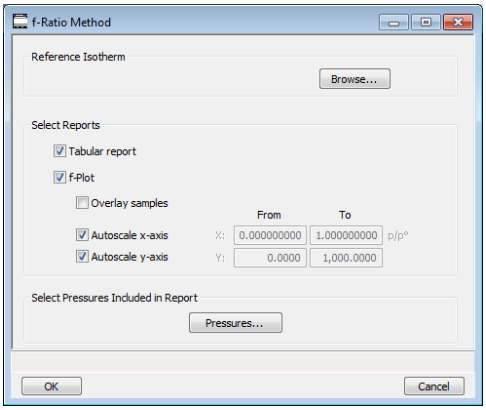
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

F-RATIO METHOD REPORT OPTIONS

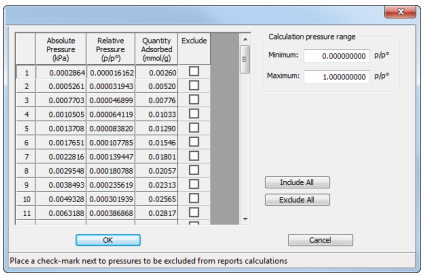
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *f*-Ratio report uses the measured isotherm and normalizes it using a reference isotherm.



f-Ratio Method Report Options Fields and Buttons Table

Field or Button	Description
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Reference isotherm	Click **Browse** to select a sample file to use as a reference for the isotherm. Select a file containing an isotherm measured from a non-porous sample of the same material as the current sample. When the referenced file is selected, the file name appears to the left of **Browse.**
Selected Reports	• **Tabular Report.** Use to have a tabular report of data generated. • ***f*-Plot.** Use to generate a normalized isotherm. ○ **Overlay samples.** Use to overlay sample files on the f-plot. ○ **Autoscale x-axis.** The X-axis field is dimensionless in units of f-ratio. ○ **Autoscale y-axis**. The Y-axis field shows the quantity of gas adsorbed.

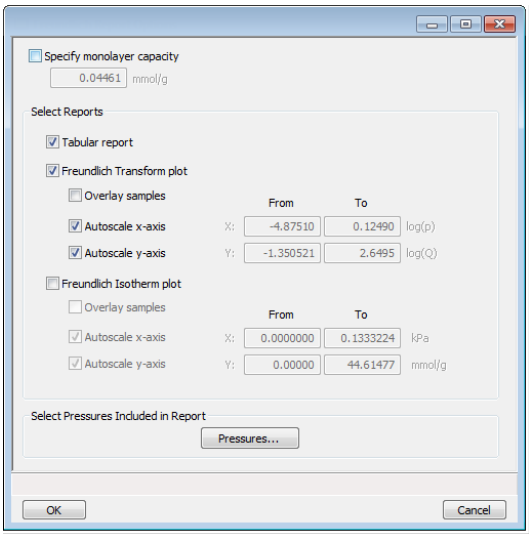
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

FREUNDLICH REPORT OPTIONS

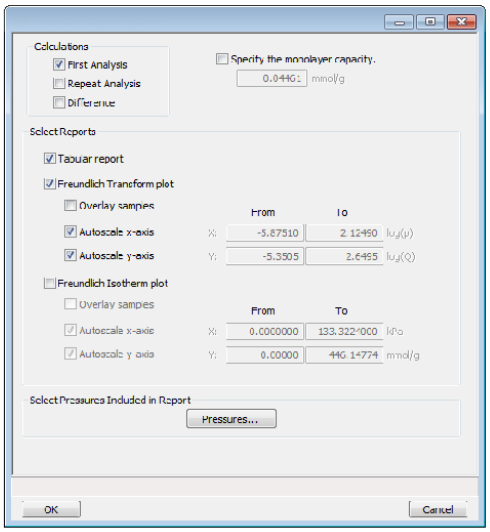
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *Freundlich Isotherm* is an empirical isotherm used to model low-pressure adsorption data. It can also be applied to model some micropore isotherms. In the *Selected Reports* list box, highlight *Freundlich*, then click **Edit**.

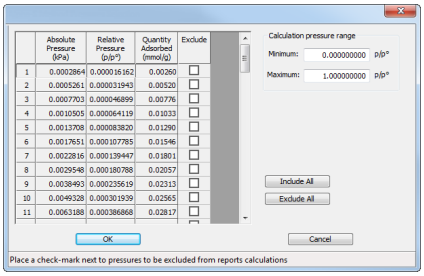
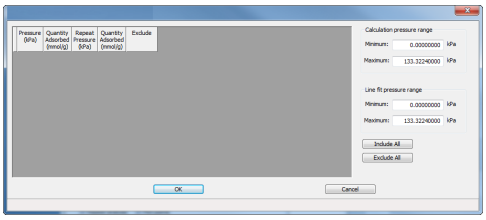


Physisorption



Chemisorption

Freundlich Report Options Fields and Buttons Table

Field or Button	Description
Calculations (for chemisorption)	Select from the various calculation options. • **First Analysis**. Includes a line fit plot for the primary analysis. • **Repeat Analysis.** Includes a line fit plot for the secondary analysis. • **Difference**. Plots the difference between the analysis and repeat analysis lines.
Pressures (for physisorption)	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Pressures (for chemisorption)	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Line fit pressure range.** Enter the minimum and maximum pressures for line fit. • **Include All.** Select to include all pressure points in the table.

Freundlich Report Options Fields and Buttons Table (continued)

Field or Button	Description
	• **Exclude All.** Select to exclude all pressure points in the table.
Select Reports	• **Tabular report.** Select to include pressure points included in the report. • **Freundlich Isotherm plot.** Plots the absolute pressure vs quantity adsorbed. Shows best fit line. ◦ **Overlay samples.** Use to overlay sample files on the Freundlich isotherm plot. ◦ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure. ◦ **Autoscale y-axis.** Y-axes begin at zero. The y-axis field shows the quantity of gas adsorbed. • **Freundlich Transform plot.** Plots the log(P) vs log(Q) and the best fit. ◦ **Overlay samples.** Use to overlay sample files on the Freundlich transform plot. ◦ **Autoscale x-axis.** The x-axis field shows the absolute pressure. ◦ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.
Specify monolayer capacity	Select and enter the monolayer capacity of the sample.
Tabular report	Use to have a report of the pressure points generated.

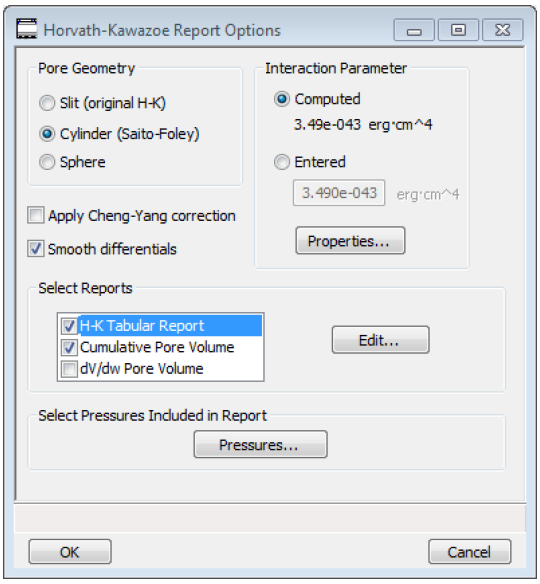
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

HORVATH-KAWAZOE REPORT OPTIONS

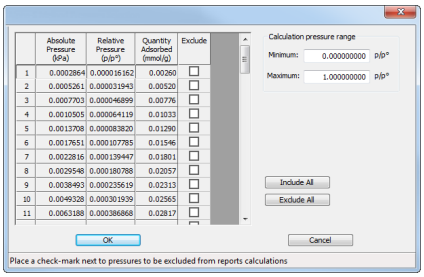
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

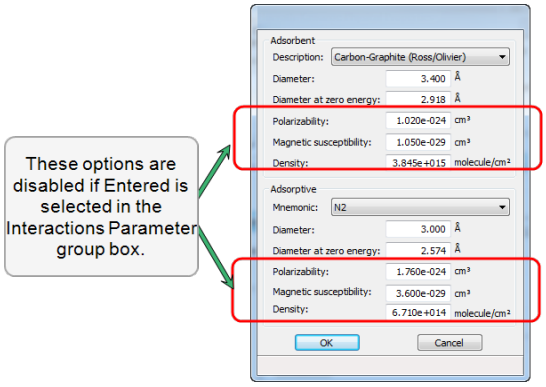
The *Horvath-Kawazoe* method plots individual peaks for different pore sizes even if the difference between one pore size and the next is only one angstrom (0.10 nm) or less.



Horvath-Kawazoe Report Fields and Buttons Table

Field or Button	Description
Apply Cheng-Yang correction	Use to apply the Cheng-Yang correction to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath-Kawazoe derivation.
Interaction Parameter	Use to determine which interaction parameter will be used in the report. These options are disabled if *Sphere* is selected in the *Pore Geometry* group box. • **Computed.** Use to calculate using the parameters on the *Horvath-Kawazoe Physical Properties* window (click **Properties** to display the *Physical Properties* window). The interaction parameter is recalculated each time a parameter in the *Physical Properties* window is edited. • **Entered.** Calculates using the value entered in the text box.
Pore Geometry	Select the option that best represents the physical geometry of the micropores in the sample material. When *Sphere* is selected, options in the *Interaction Parameter* group box are disabled.
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Properties	Click to view or edit the constants describing the physical properties of the adsorbent and adsorptive.

Horvath-Kawazoe Report Fields and Buttons Table (continued)

Field or Button	Description
	 **Adsorbent**. Contains the parameters for the sample. If using *Computed* for the interaction parameter, all fields are enabled. If using *Entered*, only the values in the *Diameter* and *Diameter at zero energy* text fields may be edited. • **Description.** Select the name of the sample used in the analysis. • **Diameter**. Enter the diameter of the sample atom. • **Diameter at zero energy**. Enter the diameter of an atom at zero interaction energy: $(2/5)^{1/6} \times$ diameter. • **Polarizability**. Enter the polarizability of the sample. • **Magnetic susceptibility.** Enter the magnetic susceptibility of the sample. • **Density.** Enter the density per unit area of the sample. **Adsorptive.** Contains the parameters for the adsorptives. If using *Computed* for the interaction parameter, all fields are enabled. If using *Entered*, only the values in the *Diameter* and *Diameter at zero energy* text fields may be edited. • **Mnemonic.** Select the mnemonic of the adsorptive gas in use. • **Diameter**. Enter the diameter of the gas phase atom. • **Diameter at zero energy.** Enter the diameter of an atom at zero interaction energy: $(2/5)^{1/6} \times$ diameter. • **Polarizability.** Enter the polarizability of the adsorptive. • **Magnetic susceptibility**. Enter the magnetic susceptibility of the adsorptive. • **Density**. Enter the density per unit area of the adsorptive.

Horvath-Kawazoe Report Fields and Buttons Table (continued)

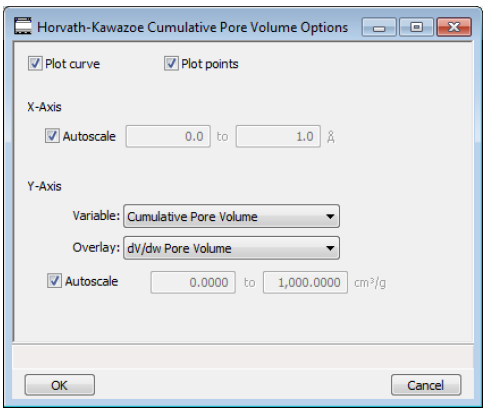
Field or Button	Description
Select Reports	Select the types of reports to generate. Highlight the report, then click **Edit** to modify report parameters.
Smooth Differentials	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

Horvath-Kawazoe Plot Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

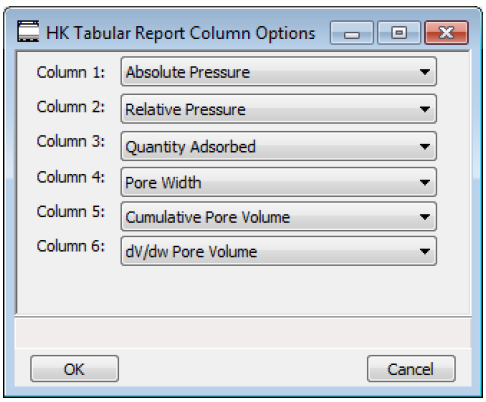


Highlight a plot option in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit** to customize the plotting method. See ***BJH Plot Options on page 8 - 15*** for additional information on fields and buttons for this report.

Horvath-Kawazoe Tabular Report Options

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.



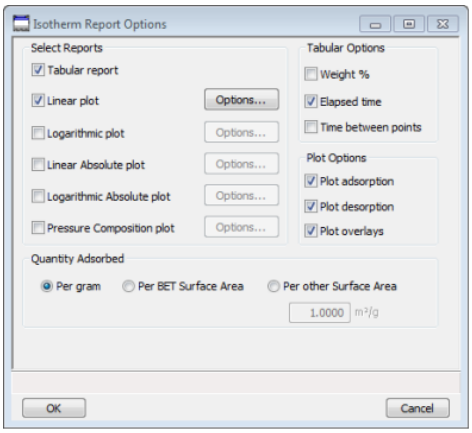
Highlight *H-K Tabular Report* in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit**. Select the data types to include in the report. *Column [n]* indicates the column order and data contents for the report.

ISOTHERM REPORT OPTIONS

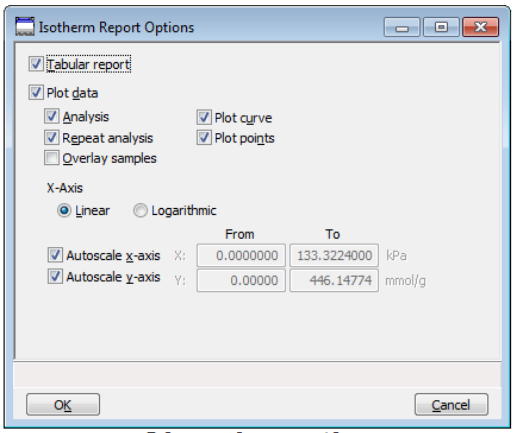
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

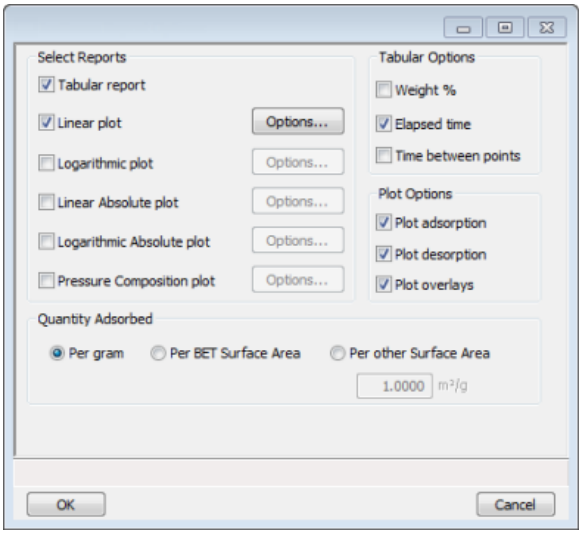
The *Isotherm* report indicates adsorption (up to saturation pressure) and desorption (down from saturation pressure) of a gas by a solid held at constant temperature.



Physisorption



Chemisorption



Physisorption Isotherm Report Options Fields and Buttons Table

Field or Button	Description
Options	Click to display related linear plot options. All plot windows contain identical fields. • **Plot curve / Plot points.**Select to plot points on the graph. • **Autoscale x-axis.** Linear x-axes begin at zero. Logarithmic x-axes begin at an appropriate value. The x-axis field shows the relative or absolute pressure. • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.
Plot Options	Select the types of isotherm to plot.
Quantity Adsorbed	Select how to report the quantity adsorbed. • per gram (cm^3/g) STP • per BET Surface Area (cm^3/m^2) STP or mmol/g • per other Surface Area (cm^3/m^2) STP or $mmol/m^2$
Select Reports	Select each option to include on the final report. Click the Options button of a selected item to include plot curve, plot points, and to autoscale x- and y-axes.
Tabular Options	Select the options to include on the report. • **Weight %.** Enter the mass percentage when plotting pressure composition • **Elapsed time.** Time elapsed during the analysis • **Time between points.** Time elapsed between points during the analysis

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

Chemisorption Isotherm Report Options Fields and Buttons Table

Field or Button	Description
Autoscale	• **Autoscale x-axis.** Linear x-axes begin at zero. Logarithmic x-axes begin at an appropriate value. The x-axis field shows the relative or absolute pressure. • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.
Plot data	Select each option to include in the final report.
Tabular report	Select to include tabular data in the report.
x-axis	Indicate if the x-axis should be in linear or logarithmic format.

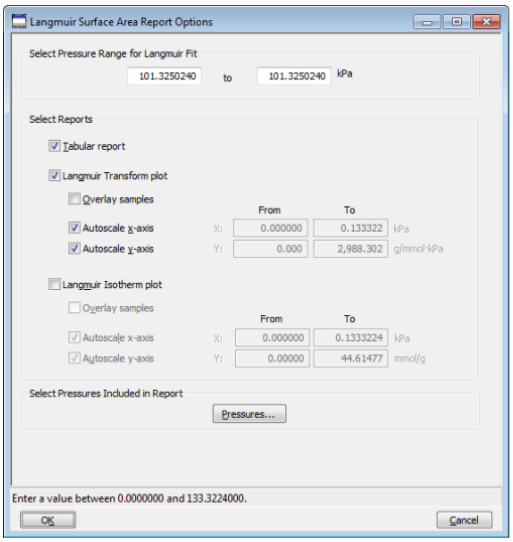
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

LANGMUIR REPORT OPTIONS

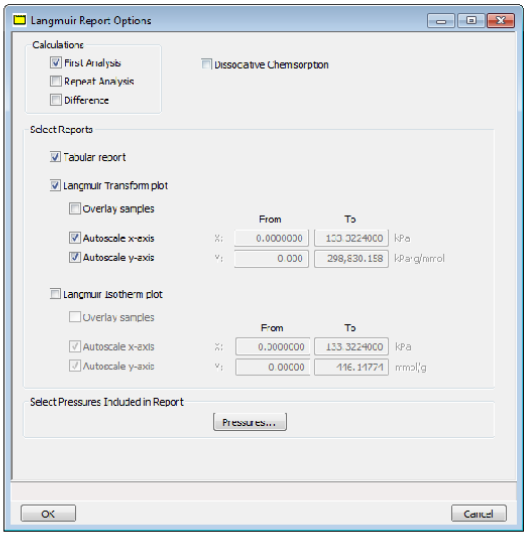
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The Langmuir calculation determines the surface area of a sample by relating the surface area to the volume of gas adsorbed as a monolayer. Langmuir uses a single layer model.

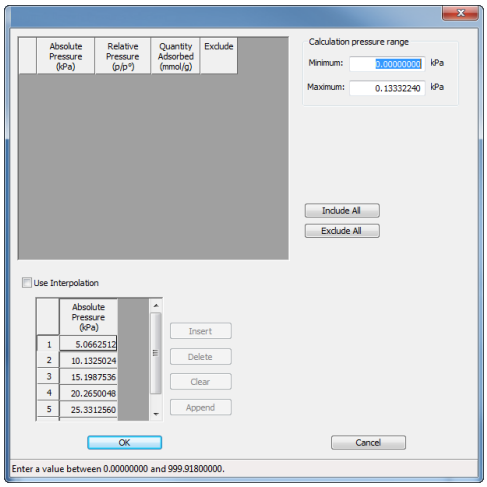


Physisorption

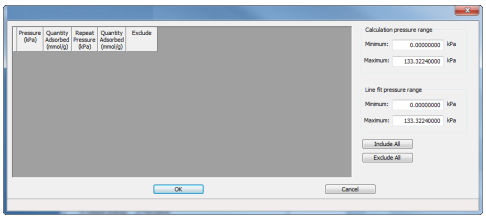


Chemisorption

Langmuir Report Options Fields and Buttons Table

Field or Button	Description
Calculations (for chemisorption)	Select one or more of the calculation options to be used for analysis.
Selected Reports	• **Langmuir Transform Plot.** Use to generate a traditional Langmuir surface area plot used to determine monolayer volume constant ◦ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. ◦ **Autoscale y-axis.** The y-axis field shows Langmuir transformation. ◦ **Overlay samples.** Use to overlay sample files on the Langmuir transform plot. • **Langmuir Isotherm Plot.** Uses the Langmuir monolayer volume and constant to produce an isotherm. ◦ **Overlay samples.** Use to overlay sample files on the Langmuir isotherm plot. ◦ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. ◦ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.
Pressures (for physisorption)	This option is available when the sample file has a status of *Analyzing* or *Complete*. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.**Enter the minimum and maximum pressures to be used in the pressure table.

Langmuir Report Options Fields and Buttons Table (continued)

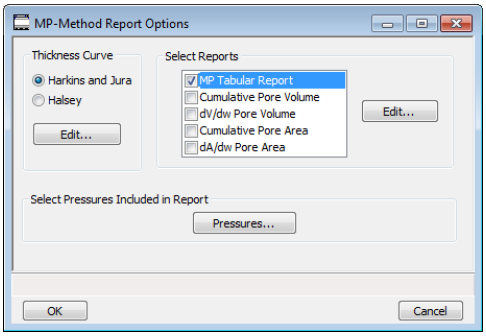
Field or Button	Description
	To exclude a point from the calculations used to generate the report, select *Exclude*. • **Use Interpolation.** Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only. • **Include All.**Select to include all pressure points in the table. • **Exclude All.**Select to exclude all pressure points in the table.
Pressures (for chemisorption)	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Line fit pressure range.** Enter the minimum and maximum pressures for line fit. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Select Pressure Range for Langmuir fit (for physisorption)	Enter values to indicate the fitted pressure range.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

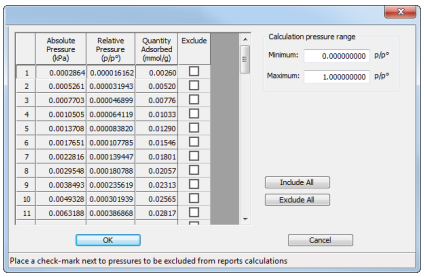
MP-METHOD REPORT OPTIONS

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *MP-Method Report Options* provides pore volume distributions for microporous materials by correlating quantity adsorbed with the thickness of the adsorbed layer as determined from a user-selected thickness curve. Pore size can be expressed in angstroms or nanometers. Go to ***Options > Units*** to specify the unit.



MP-Method Report Options Fields and Buttons Table

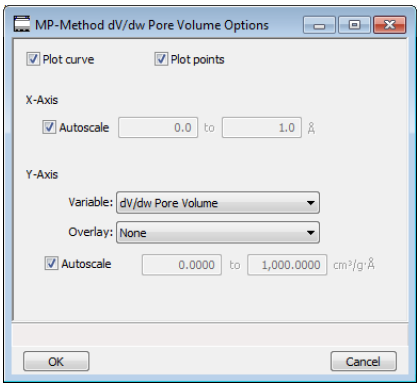
Field or Button	Description
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Select Reports	Select the reports to generate. Highlight the report, then click **Edit** to modify report options.
Thickness Curve	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

MP-Method Plot Report Options

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

In the *MP-Method Report Options* window, highlight a plot option in the *Selected Reports* list box, then click **Edit** to customize the plotting method.



MP Method Plot Options Fields and Buttons Table

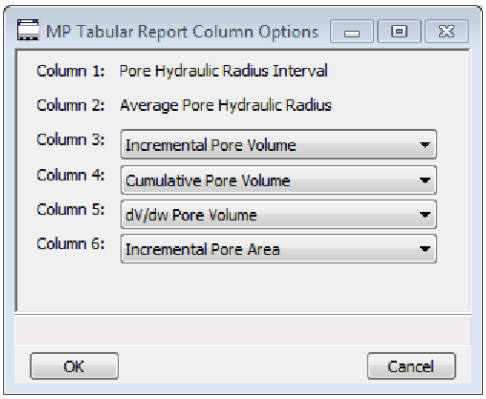
Field or Button	Description
Overlay drop-down list	Select an option to overlay on the current report.
Plot curve / Plot points	Select to plot points on the graph.
Thickness Curve	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve.
X-Axis	Use to have the x-axis autoscaled or enter beginning and ending values.
Y-Axis	• **Variable.** Select a variable. • **Overlay.** Select an option to overlay on the current report. • **Autoscale.** Use to have the y-axis autoscaled or enter beginning and ending values.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

MP-Method Tabular Report Options

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

In the *MP-Method Report Options* window, highlight *MP Tabular Report* in the *Selected Reports* list box, then click **Edit**. *Column [n]* indicates the column order and data contents for the report.



The MP Method reports hydraulic radius only. If Pore size in diameter is selected on the Unit Selection window, pore size in radius will be reports.

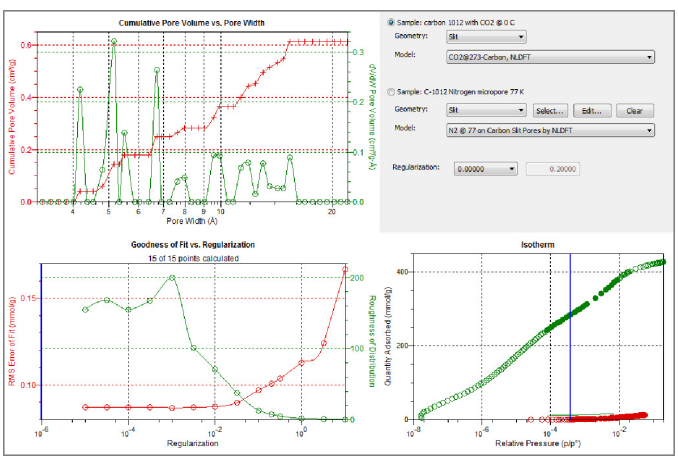
NLDFT ADVANCED PSD REPORT

A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *NLDFT Advanced PSD* report allows for more advanced computation of the pore size distribution of a material using two separate analyses and two non-local DFT models.

The *NLDFT Advanced PSD* report option provides the same calculations as the DFT Pore Size report option and more. The NLDFT report compares two sample files. The models that can be selected are restricted to only those models which have the same analysis temperature and analysis gas as the sample file that is open. For instance, if the sample file was analyzed with N2 at 77 degrees Kelvin, then only the N2 DFT models at 77 degrees Kelvin will be available in the *Model* drop-down list.



The model curve fit is shown in the lower right quadrant along with the adsorption isotherm. This curve fit is updated each time the calculation parameters change (selection of isotherm data points, choice of model, choice of regularization parameter).

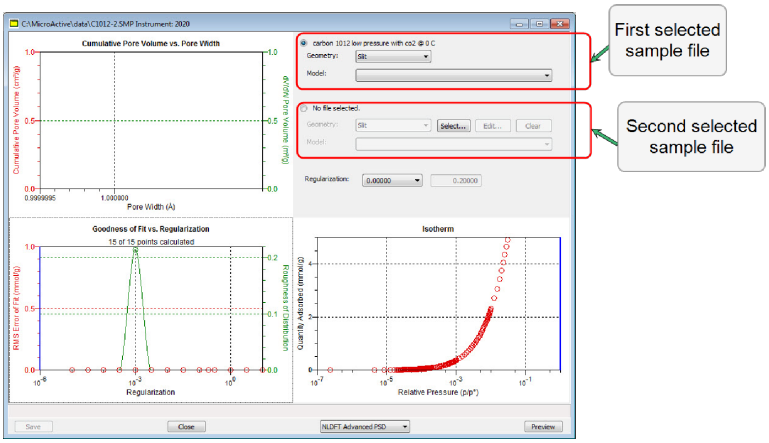
A second sample file and second model is used to compute a more accurate pore size distribution (PSD), which is shown in the upper left quadrant. Typically, the second sample file will have used the same sample material as the first sample file yet will have used a different analysis gas and temperature.

In general, the isotherm for this second sample will be different than the first sample. The advanced DFT calculation takes the data from both sample files and combines all this data into a more accurate calculation of the pore size distribution. More accurate means getting the pore distribution at smaller pore sizes (a few Angstroms) as well as larger pore sizes (one thousand Angstroms).

To make a successful *advanced* calculation, a second sample file must be selected using the **Select** button. A second model must also be selected. Use the options next to the two sample file names to select the isotherm data points for each sample. After selecting an option, the blue bars in the isotherm graph will be toggled to select either the red points or the green points. Once these selections have been done, the results will appear in the left-hand plots and a second isotherm will appear in the isotherm plot (lower right) as well as a second curve-fit. As the selection of points is adjusted, the DFT editor will recalculate the PSD results and also recalculate the two model curve fits.

To run the NLDFT report:

1. Go to ***File > Open***. Select a sample file with a *Complete* status, then click **Open**.
2. In the drop-down list at the bottom of the window, select *NLDFT Advanced PSD*. Graphs for the first sample file display and the sample description shows as the first group box title in the upper right corner of the window.

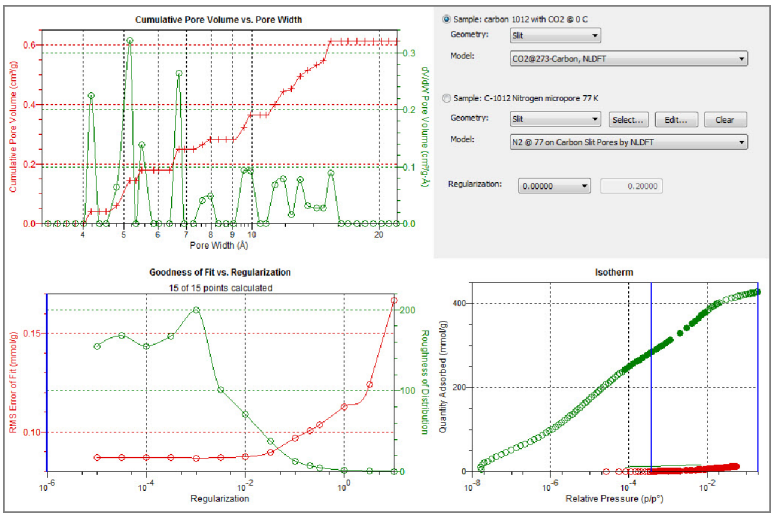


   a. Select the *Geometry* and *Model* from the drop-down lists for the first sample file.
   b. To select isotherm data points for calculation for the first sample file, ensure the option to the left of the first sample file description is selected. Slide the two blue bars on the isotherm graph to select data points. Without a second sample selected, the report will perform a single model DFT calculation and show the results in the two left-hand result windows.

3. To calculate data from the second sample file, click **Select** to locate and open the second sample file with a *Complete* status. Graphs for the second sample file display and the sample description displays as the second group box title in the upper right corner of the window.
   a. Select the *Geometry* and *Model* from the drop-down lists for the second sample file.
   b. To select isotherm data points for calculation for the second sample file, ensure the option to the left of the second sample file description is selected. Slide the two blue bars on the isotherm graph to select data points. Data are automatically calculated for both sample

files.

c. Click **Edit** to make any necessary modifications to the second sample file.



NLDFT Advanced PSD Report Fields and Buttons Table

Field or Button	Description
Geometry	Select the pore shape.
Model	Lists the models that meet the specified criteria and match the adsorbate and temperature of the sample data. If no models appear, no models meet the selected criteria. One model must be selected.
Regularization	Select the extent of smoothing to apply to the data. If 0.20000 (user) is selected, enter a number in the text box giving a relative mass for the smoothing during deconvolution. Larger values produce more smoothing.
Select	Use to select the second sample file.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

OPTIONS REPORT

The *Options* report for physisorption analyses lists the conditions used to perform the analysis— such as:

- Adsorptive properties
- Analysis conditions
- Analysis method
- Degas conditions
- Free space
- Saturation pressure (P_0) and temperature

The *Options* report for chemisorption analyses is a predefined collection of sample file parameters printed. If *Automatically collected* is selected in the *Type of Data* group box on the *Sample Description* tab, the following information is reported:

- **Task Summary.** Lists conditions specified for each task selected.
- **Analysis Task Options.** Details conditions specified for the analysis task.
- **Experiment Log.** Identifies actual conditions under which each task transpired.
- **Leak Test Results.** Identifies outgas rates and the outcome for each leak test performed.

Options reports cannot be edited.

SAMPLE LOG REPORT

This report provides information on:

- Manual control operations performed during analysis
- Information entered using *Add Log Entry* on the sample file editor
- Warnings and / or errors which occurred during analysis

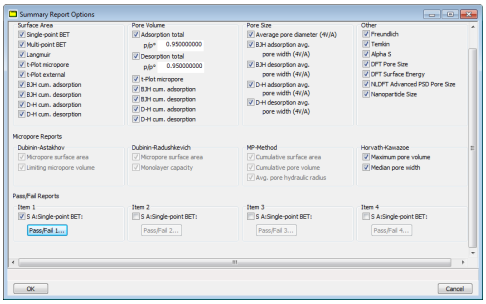
SINFELT AND DIFFERENCE METHODS

The *Difference Method Report* and the *Sinfelt Method Report* windows are identical unless otherwise specified. See ***Difference Method Report Options on page 8 - 22***.

SUMMARY REPORT OPTIONS

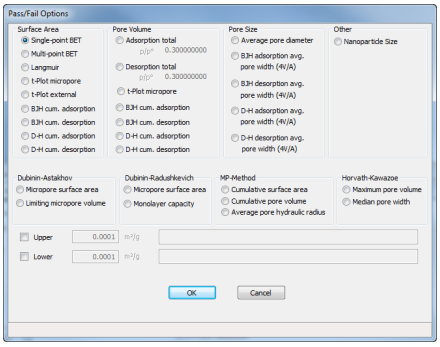
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *Summary Report* provides a condensed listing of selected data results.



In the *Pore Volume* group box, if *Adsorption total* or *Desorption total* is selected, the p/p^o field is enabled. Enter the relative pressure used to calculate the total pore volume.

Summary Report Fields and Buttons Table

Field or Button	Description
Item [n]	Use to enable the first *Pass/Fail* item. Until the *Summary Report* is selected, *S A Single-point BET* will be displayed by default. When selected, click **Pass/Fail**, then select pass/fail criteria options. • **S A: Single-point BET.** Use to enable **Pass/Fail [*n*]** in the *Item [n]* group box. • **Pass/Fail [n].** Click to display the *Pass/Fail Options* window for selection of pass/fail criteria.  • **Upper / Lower.** Specify upper and lower limits for the selected parameter. A range can be left open by not selecting the limit. In the text box to the right of *Upper / Lower*, enter operator instructions to be displayed if a failure is encountered.
Select All / Deselect All	Selects (or deselects) all options.

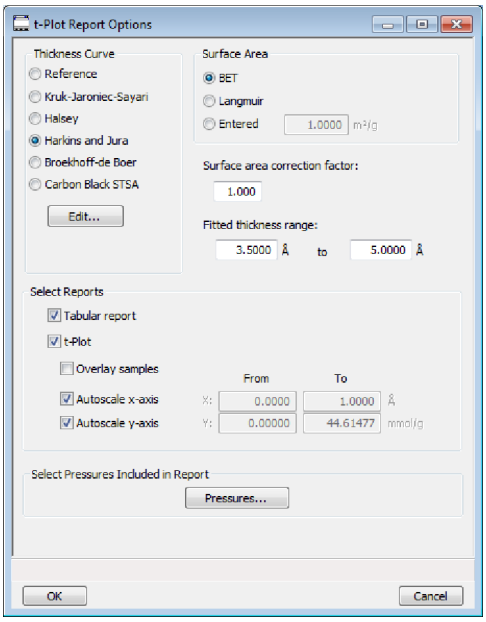
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

T-PLOT REPORT OPTIONS

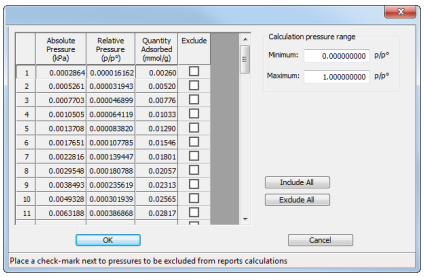
A tutorial is available for this topic. To view the tutorial, click the** Tutorial **tab in Online Help.

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

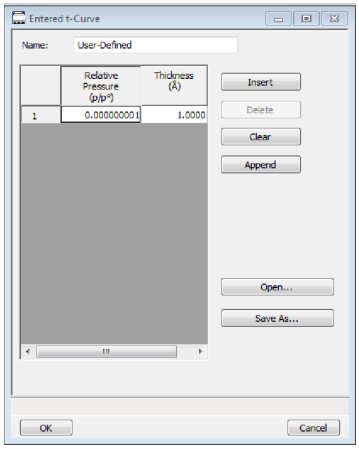
The *t*-Plot calculation allows quantitative analysis of the area and total volume ascribed to micropores. Matrix area (the area external to micropores) is directly determined and often proves to be a valuable way of characterizing complex mixed materials.



t-Plot Report Options Fields and Buttons Table

Field or Button	Description
Fitted thickness range	Enter the minimum and maximum thicknesses (in angstroms or nanometers) to include in the thickness curve. Go to ***Options > Units*** to specify default units.
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Selected Reports	• **Tabular Report.** Use to have a tabular report of data generated. • ***t*-Plot.** Use to have a graphical representation of data generated. ○ **Overlay samples.** Use to overlay sample files on the *t*-plot. ○ **Autoscale x-axis.** The X-axis field shows the statistical thickness of the adsorbed film. ○ **Autoscale y-axis.** The Y-axis field shows the quantity of gas adsorbed.
Surface area correction factor	Enter the value to correct for surface areas that are not smooth. This brings the values for BET surface area and micropore surface area into accordance. For most samples, the default value of 1.000 is adequate.
Surface Area	Select the surface area value used for thickness calculations. BET is the most commonly used option.
Thickness Curve	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Reference.** Select *Reference*, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined

t-Plot Report Options Fields and Buttons Table (continued)

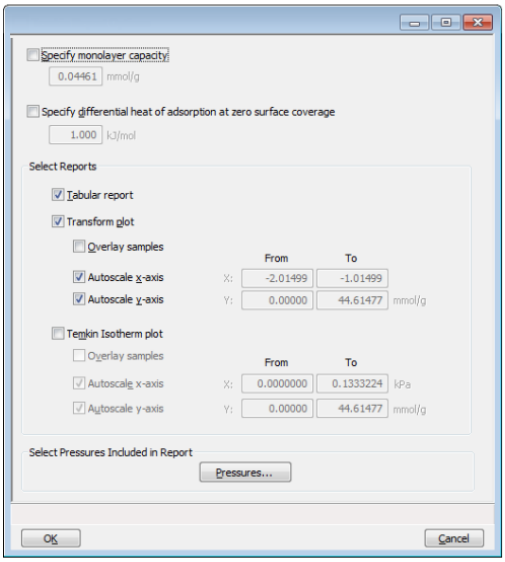
Field or Button	Description
	curve is shipped with the analysis program and is found in the *Reference* directory.  To import values from an existing thickness curve (.THK file), click **Open**, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space or a tab. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed.
t-Plot	Use to have a graphical representation of data generated. • **Overlay samples.** Use to overlay sample files on the *t*-plot. • **Autoscale x-axis.** The X-axis field shows the statistical thickness of the adsorbed film. • **Autoscale y-axis.** The Y-axis field shows the quantity of gas adsorbed.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

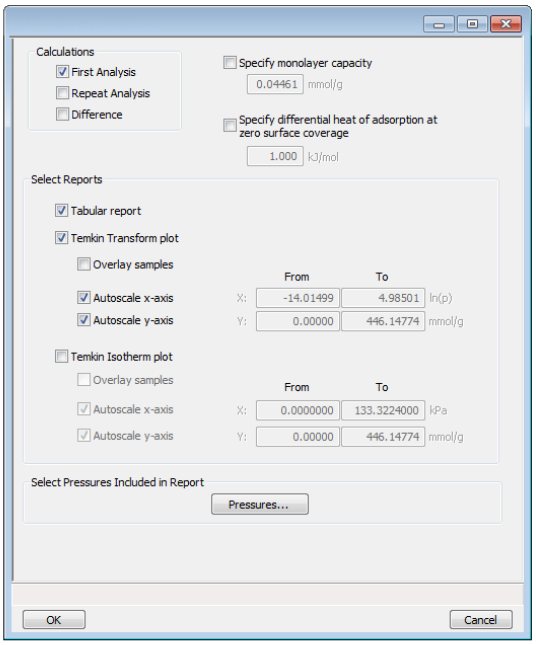
TEMKIN REPORT OPTIONS

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

The *Temkin* isotherm is used to model adsorption data where the heat of adsorption drops linearly with increasing coverage.

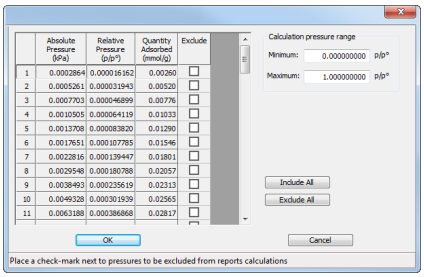
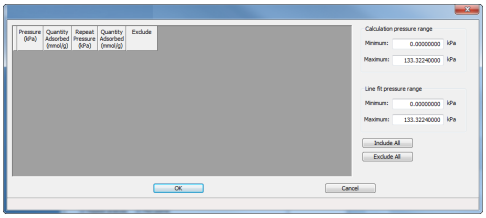


Physisorption



Chemisorption

Temkin Report Options Fields and Buttons Table

Field or Button	Description
Calculation Options (for chemisorption)	Select one or more of the calculation options to be used for analysis.
Pressures (for physisorption)	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Pressures (for chemisorption)	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Line fit pressure range.** Enter the minimum and maximum pressures for line fit. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Selected Reports	• **Tabular Report**. Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.

Temkin Report Options Fields and Buttons Table (continued)

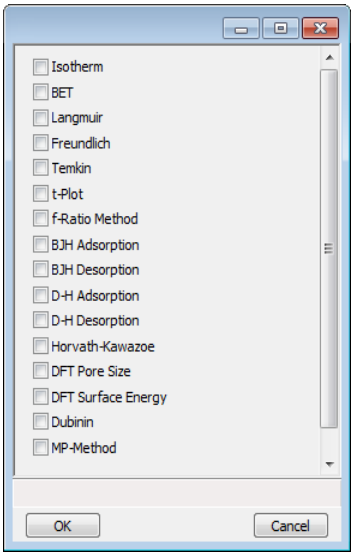
Field or Button	Description
	• **Temkin transform plot.** Plots a linear form of the Temkin transform plot. ◦ **Autoscale x-axis**. The X-axis field shows the logarithm of pressure (ln). ◦ **Autoscale y-axis.** The Y-axis field shows the quantity of gas adsorbed. ◦ **Overlay samples.** Use to overlay sample files on the transform plot. • **Temkin isotherm plot.** Overlays the Temkin isotherm with the analysis data. ◦ **Overlay samples.**Use to overlay sample files on the isotherm plot. ◦ **Autoscale x-axis.** Linear X-axes begin at zero. The X-axis field shows the absolute pressure. ◦ **Autoscale y-axis.** Y-axes begin at zero. The Y-axis field shows the quantity of gas adsorbed.
Specify differential heat of adsorption	Select and enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all Temkin constants.
Specify monolayer capacity	Select and enter the monolayer capacity of the sample.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

VALIDATION REPORT OPTIONS

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

This report allows data to be examined by the analysis program to determine if the results are within typical ranges. If the data for any reports selected for validation are determined to be out of range, a warning displays, and suggestions are given for corrective action. This information is detailed in the report and plotted on the graph as a unique plot symbol.



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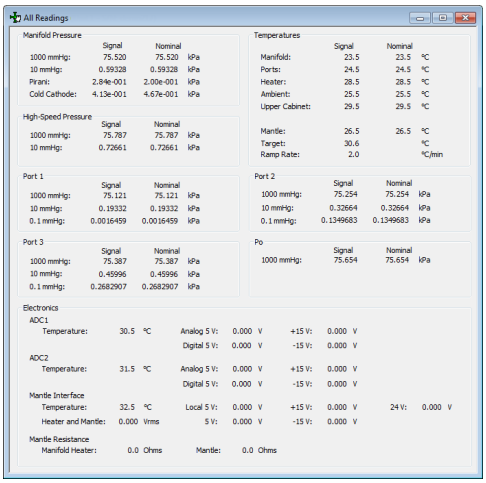
9 DIAGNOSTICS

SHOW ALL READINGS

Unit [n] > Diagnostics > Show All Readings

The *Show All Readings* window displays the calibrated and nominal readings of all sensors in the system.



START DIAGNOSTIC TEST

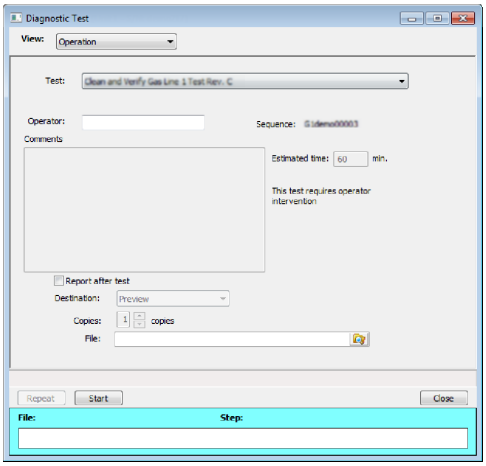
Unit [n] > Diagnostics > Start Diagnostic Test

Provides a method to start a diagnostic test immediately. To view the print options, resize the window. Upon completion of the diagnostic test, the file is saved as a .REP file which can be retrieved by going to ***Reports > Open Report*** and selecting the report file.

It is recommended to schedule the *Analysis Manifold Leak Test* and the P_0 *Port Leak Test* to run unattended on a weekly basis. These tests check for system leaks and require no operator intervention.

CAUTION

The P_0 *Port Leak Test* should only be run if the Psat tube is attached. If a vapor source is attached, this test should not be run.



Start Diagnostic Test Fields and Buttons Table

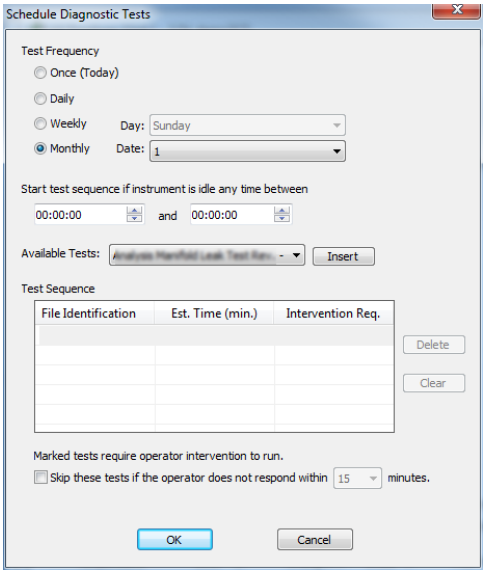
Field or Button	Description
Comments	Displays comments from the selected diagnostic test.
Estimated time (min.)	Approximate time for test completion.
File	Shows a status bar of steps complete once the test begins.
Next	Starts the next test.
Operator	Enter information to identify the person running the service test.
Repeat	Repeats the selected diagnostic test.
Report after test	Automatically generates reports to the selected destination when the test is complete.
Sequence	Sequence number assigned to the test.
Test	Select the diagnostic test to be performed.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

SCHEDULE DIAGNOSTIC TESTS

Unit [n] > Diagnostics > Schedule Diagnostic Tests

Allows the specification of one-time or periodic running of a sequence of diagnostic tests. A separate list of tests is saved for each of the possible test frequencies. Tests are categorized and flagged as requiring intervention or not. If tests requiring intervention are scheduled, the operator has the option of omitting the tests if the operator does not respond within a specified time after an initial prompt is displayed and before the test is started. Events are logged in the analyzer log for all starting, ending, and omitted tests.



It is recommended to schedule the *Analysis Manifold Leak Test* and the P_0 *Port Leak Test* to run unattended on a weekly basis. These tests check for system leaks and require no operator intervention.

CAUTION

The P_0 *Port Leak Test* should only be run if the Psat tube is attached. If a vapor source is attached, this test should not be run.

Schedule Diagnostics Test Frequency Fields and Buttons Table

Field or Button	Description
Available Tests	Select one or more tests to run unattended. Select the test, then click **Insert** for the test to display in the *Test Sequence* box.
Insert	Inserts the selected test in the *Available Tests* drop-down list.
Skip these tests if the	Check this option if any test requiring operator intervention should be omit-

Schedule Diagnostics Test Frequency Fields and Buttons Table (continued)

Field or Button	Description
operator does not respond within [*n*] minutes	ted if the operator does not respond within the specified time.
Start test sequence if instrument is idle any time between 00:00:00 and 00:00:00 .	Enter a from and to time for an unattended test to begin if the instrument is idle at any time during the entered time frame.
Test Frequency	Select how often the test is to run unattended.
Test Sequence	Provides the test file identification and estimated run time. A checkmark in the *Intervention Required* column indicates that operator intervention is required. To remove a test from the sequence, select the test, then click **Delete**. Alternatively, click **Clear** to clear the entire table of all entries. To add a test to the test sequence, highlight a row in the *Test Sequence* box, select a test from the *Available Tests* list, then click **Insert**. The new test will be inserted above the highlighted row.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

DIAGNOSTIC TEST REPORT

Unit [n] > Diagnostics > Diagnostic Test Report

Displays previously run diagnostic service tests. Separate directories store tests run once, daily, weekly, and monthly. Diagnostic test report files have a .SVT file extension and are stored in the ...\Service directory.

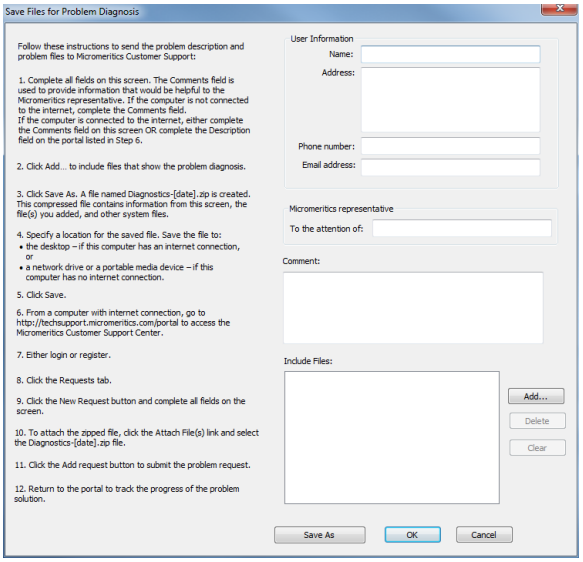
1. To open a diagnostic test report, select a service test report, then click **Open**. Alternatively, double click the report file name.
2. On the *Selected Reports* window, select the reports to display, then click **OK**.
3. The selected reports display on separate tabs at the top of the report window.

SAVE FILES FOR PROBLEM DIAGNOSIS

Unit [n] > Diagnostics > Save Files for Problem Diagnosis

Use to compress pertinent diagnostic information into a single zip file. This file can be sent to a Micromeritics Service Representative for problem resolution. The following files are included in the compressed file:

- *[instrument model]*.ini
- info[sn].dat
- cal [sn].dat
- [sn].dat
- UserInformation.txt
- Any files selected by the user



To send the problem description and problem files to Micromeritics Customer Support.

1. Complete all fields. The *Comments* field is used to provide information that would be helpful to the Micromeritics representative.
   - If the computer is not connected to the internet, complete the *Comments* field.
   - If the computer is connected to the internet, either complete the *Comments* field on this window OR complete the *Description* field on the portal listed in Step 6.
2. Click **Add** to include files that show the problem diagnosis.
3. Click **Save As**. A file named *Diagnostics-[date].zip* is created. This compressed file contains

information from this window, any added file(s), and other system files.

4. Specify a location for the saved file. Save the file to:
   - the desktop - if this computer has an internet connection, or
   - a network drive or a portable media device - if the computer is not connected to the internet.
5. Click **Save**.
6. From a computer with internet connection, go to http://techsupport.micromeritics.com/portal to access the Micromeritics Customer Support portal.
7. Either log in or register.
8. Click the *Requests* tab.
9. Click **New Request**, then complete all fields on the window.
10. To attach the zipped file, click the *Attach File(s)* link, then select the *Diagnostics-[date].zip* file.
11. Click **Add request** to submit the problem request.
12. Return to the portal to track the progress of the problem solution.

Save Files for Problem Diagnostic Fields and Buttons Table

Field or Button	Description
Comment	Enter information that would be helpful to the Micromeritics representative. If the computer is not connected to the internet, complete this field. If the computer is connected to the internet, this information can be completed on the Micromeritics Customer Support portal.
Include Files	• **Add.** Click to select additional files to send with this problem diagnosis. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Delete.** Select the file in the *Include Files* box, then click **Delete** to remove the file from the list. • **Clear**. Click to clear all files from the *Include Files* box.
Save As	Click to specify the name and location of the compressed file. Make a note of the file name and location. This file will need to be sent to your Micromeritics representative for problem resolution.

Save Files for Problem Diagnostic Fields and Buttons Table (continued)

Field or Button	Description
Micromeritics representative	Enter the name of your Micromeritics representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).
User Information	Enter information for the person to be contacted by a Micromeritics representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

10 About Calibration

Unit [n] > Calibration

Use to perform system calibrations. Disabled calibration options can be accessed only with the assistance of an authorized Micromeritics service representative. Calibrations can be saved to a file and reloaded later.

Load Calibration from File

Unit [n] > Calibration > Load from File

Use to load a previously saved calibration file.

It is recommended that the current calibration settings be saved using ***Unit [n] > Calibration > Save to File*** prior to loading another calibration file. When loading a previously saved calibration file, a backup of the current file is created and saved as *[SN]last.cal*. The backup file is overwritten each time a new one is created.

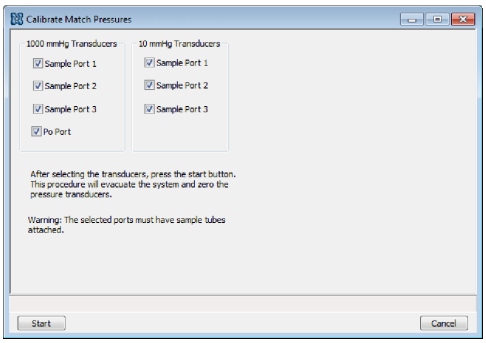
Changing the calibration may affect the analyzer's performance.

MATCH TRANSDUCERS

Unit [n] > Calibration > Match Transducers

Use to evacuate the system and zero the pressure transducers, then adjust the scale to match them to the manifold transducer near full scale pressure.

A blank sample tube or small plug must be installed on each selected port prior to starting this process.



1. Install a blank sample tube or small plug on each applicable port.
2. Ensure that all applicable transducers are selected, then click **Start**. The window closes when the operation is complete. Click **Cancel** to stop the calibration process.

Match Transducers Fields and Buttons Table

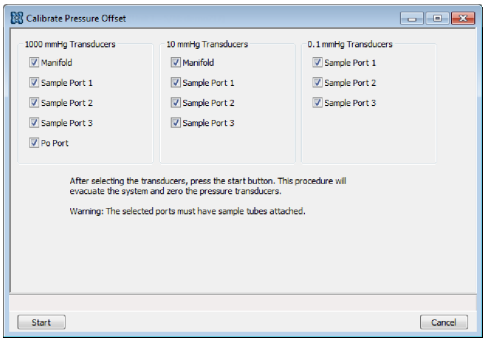
Field or Button	Description
1000 mmHg Transducers	Select the ports.
10 mmHg Transducers	Select the ports. Enabled only for ports with 10 mmHg transducers present.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

PRESSURE OFFSET

Unit [n] > Calibration > Pressure Offset

This procedure evacuates the system and zeroes the pressure transducers. This calibration should only be performed by qualified service personnel. In order to perform this procedure, sample tubes must be attached to each port.



1. Install a blank sample tube or small plug on each applicable port.
2. Ensure that all applicable transducers are selected, then click **Start**. The window closes when the operation is complete. Click **Cancel** to stop the calibration process.

Pressure Offset Fields and Buttons Table

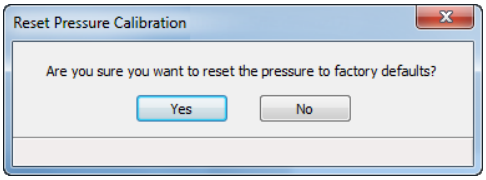
Field or Button	Description
1000 mmHg Transducers	Select the manifold and / or ports.
10 mmHg Transducers	Select the manifold and / or ports. Enabled only for the manifold and ports with 10 mmHg transducers present.
0.1 mmHg Transducers	Select the ports. Enabled only for ports with 0.1 mmHg transducers present.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

RESET PRESSURE CALIBRATION

Unit [n] > Calibration > Reset Pressure Calibration

This procedure resets the pressure calibration to the factory default settings.



Click **Yes** to reset the pressure calibration. Click **No** to leave the settings unchanged.

The servo valve should always be recalibrated after a pressure calibration has been performed. The pressure transducer should be calibrated before starting this procedure.

SAVE CALIBRATION TO FILE

Unit [n] > Calibration > Save to File

Use to save the current calibration settings to a backup file which can later be reloaded using ***Unit [n] > Calibration > Load from File*** menu option.

The default file naming convention for calibration files can be used or the file name can be changed. The default file name of 0217-2013-04-25.CAL is interpreted as:

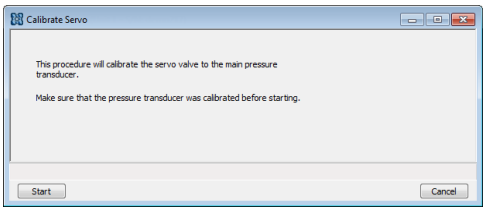
0217	is the analyzer serial number
2013-04-25	is the date the calibration file was saved
.CAL	is the file name extension

SERVO VALVE

Unit [n] > Calibration > Servo Valve

Use to calibrate the servo valve to the manifold pressure transducer. The servo valve should always be recalibrated after a pressure calibration has been performed. The pressure transducer should be calibrated before starting this calibration procedure.

Ensure the pressure transducer has been calibrated before performing this procedure. Go to ***Unit [n] > Unit Configuration*** and view the calibration information. Contact your service representative if calibration dates are not listed.



Click **Start**. The window closes when the calibration is complete. Click **Cancel** to stop the calibration process.

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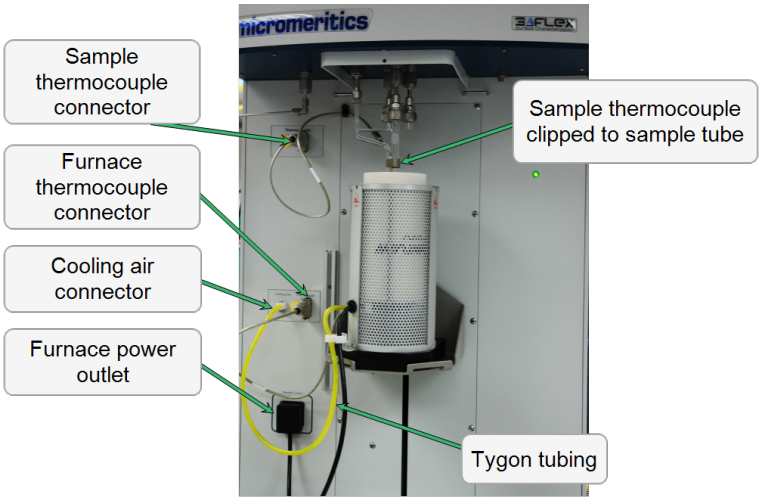
11 Hardware Components and Accessory Installation

Furnace Installation

The furnace uses same supply of compressed air as the instrument. There is an internal regulator that has been set at the factory for the correct flow rate of cooling air to the furnace.



NOTE: This photo is shown without the safety cover. The safety cover should always be installed prior to running an analysis.

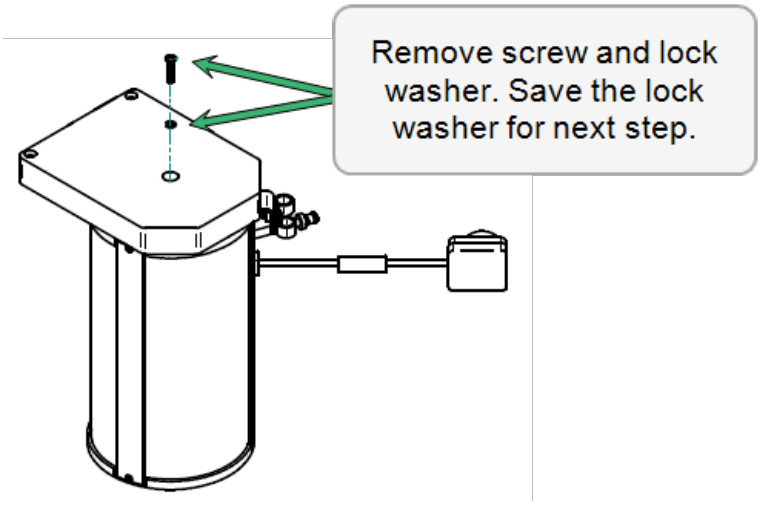
1. Place and center the furnace on the elevator.
2. Insert the furnace power cable into the power connector.
3. Connect the furnace thermocouple plug into the *Thermocouple* connector on the front of the analyzer.
4. Connect the cooling air tube into the *Cooling Air*connector on the front of the analyzer.

SECURE FURNACE TO ELEVATOR TRAY

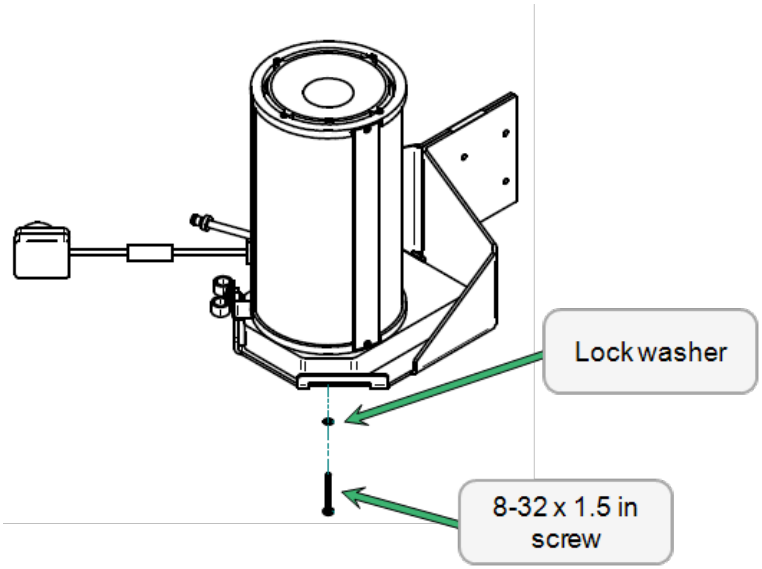
This is an optional procedure for securing the furnace to the elevator tray in areas prone to earthquakes.

Replace the screw that holds the black plastic base to the furnace and replace it with a 8-32 × 1.5 in. screw (part #004-28622-00).

1. Invert the furnace and remove the screw that holds the black plastic base to the bottom of the furnace. Save the lock washer and use it when inserting the longer screw.

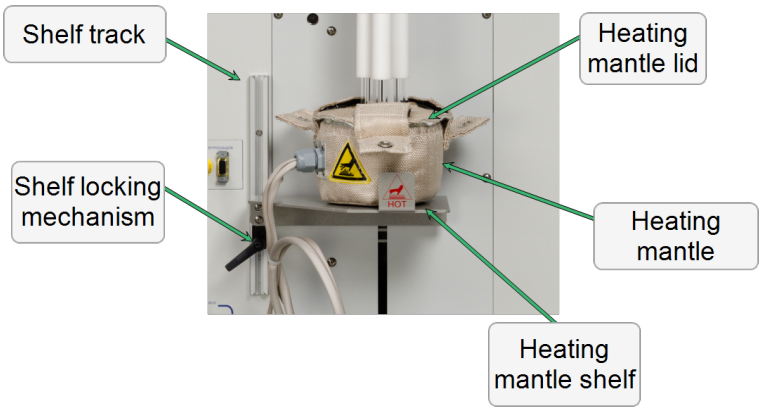


2. Place the black plastic base onto the elevator aligning the hole in the base with the hole in the elevator.
3. Position the furnace on top of the black plastic base aligning the hole in the furnace bottom with the hole in the furnace base.

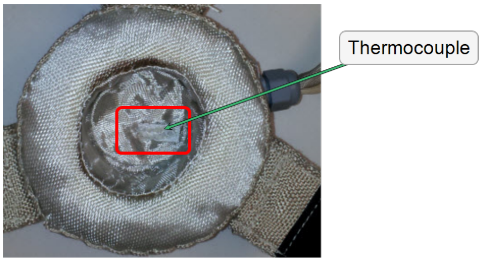


4. Raise the elevator. Position the lock washer in the hole in the elevator tray. Insert the 8-32 × 1.5 in. screw through the lock washer, the hole in the elevator, the black plastic base, and into the furnace.

HEATING MANTLE INSTALLATION



If using less than three sample tubes, the heating mantle position may need to be adjusted such that the bottom of a sample tube touches the thermocouple located on the bottom surface of the mantle's interior. A single sample tube must be installed on port 2.



1. Place the mantle around the sample tube bulbs. Ensure that the isothermal jackets are pushed up against the dewar lid to avoid damage to the jackets.

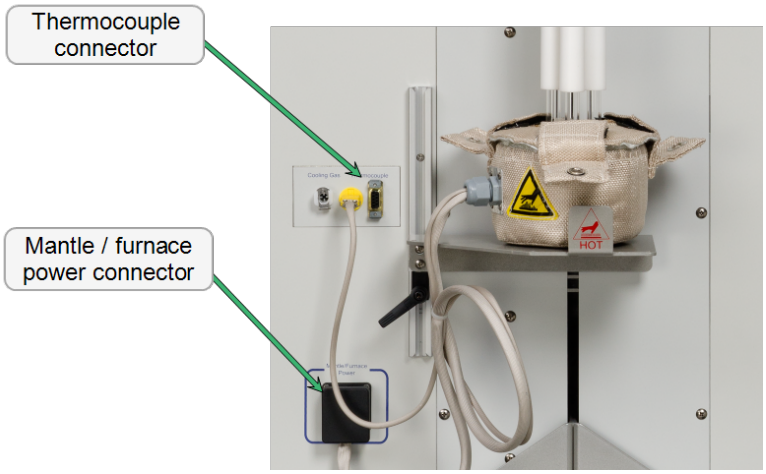
2. While supporting the heating mantle with one hand, slide the shelf locking mechanism into the shelf track. Raise the shelf on the track until the heating mantle rests securely on the shelf and the sample tubes touch the bottom of the inside of the heating mantle. Turn the locking mechanism clockwise to secure the shelf.


3. Slide the heating mantle cover between the sample tube bulbs and the bottom of the isothermal jackets so that the sample tubes fit within the slots of the mantle cover.


CAUTION

Do not to apply force to the tubes while installing the lid.

4. Secure the heating mantle tabs onto the hook and loop fasteners of the heating mantle cover. Ensure there is at least a 1/2 in. (12 mm) gap between the top of the mantle cover and the bottom of the isothermal jackets. This will prevent damage to the jackets. Replace any damaged jackets.
5. Insert the mantle thermocouple and the mantle power plug into the analyzer's front panel.



6. Acknowledge the prompt on the *Sample Analysis* window. The degas will proceed. When the degas is completed and the mantle has cooled below 45 °C, the *Sample Analysis* window will submit a prompt to remove the degas heating mantle and shelf, properly position the isothermal jackets and dewar lid, and install the dewar.

To prevent potential burns, do not touch the sample tube or the heating mantle until they have cooled.

7. To remove the heating mantle, take off the heating mantle cover, support the bottom of the heating mantle, then lower the shelf. The shelf must be removed prior to installing the dewar.

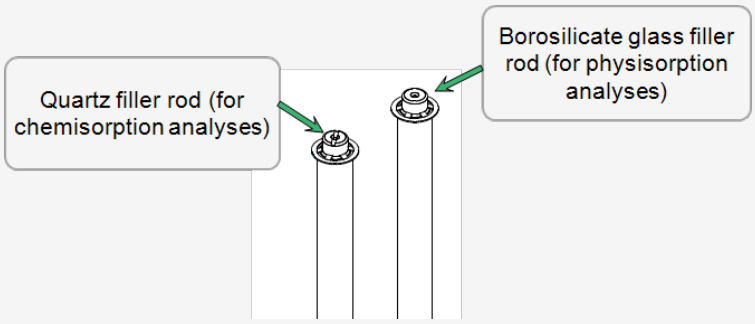
INSTALL THE SAMPLE TUBES FOR CHEMISORPTION

Wear latex gloves when handling the sample tube. The natural oils in human skin can chemically damage and weaken the quartz tube. It is also important that the sample tube and its components, as well as the sample and exhaust ports, be clean and free of debris. Dust particles from quartz wool or the insulator disc of previous analyses may adhere to the port and / or components, preventing a proper seal of the sample tube.

1. Remove the stopper from the sample tube stem and the cap from the exhaust stem.
2. Use a lint-free swab moistened with IPA and wipe the interior rims of the sample and exhaust ports.
3. Use a lint-free tissue moistened with IPA and wipe the O-ring, ferrule, and connector nuts for the sample and exhaust tubes. Place on a lint-free tissue.

Sample and exhaust ports, as well as all components that contact the sample and exhaust ports, must be clean, therefore it is recommended that the previous steps be repeated each time a sample tube is installed onto a port

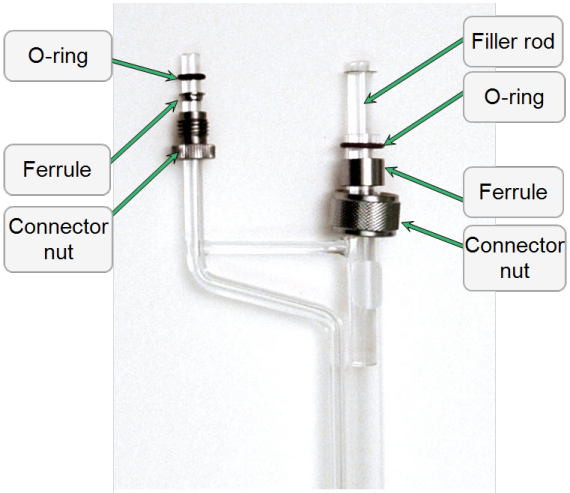
4. Remove the stopper from the sample tube stem and the cap from the exhaust stem.
5. If using a hanging filler rod (recommended), hold the sample tube slightly tilted and carefully place the filler rod into the tube.



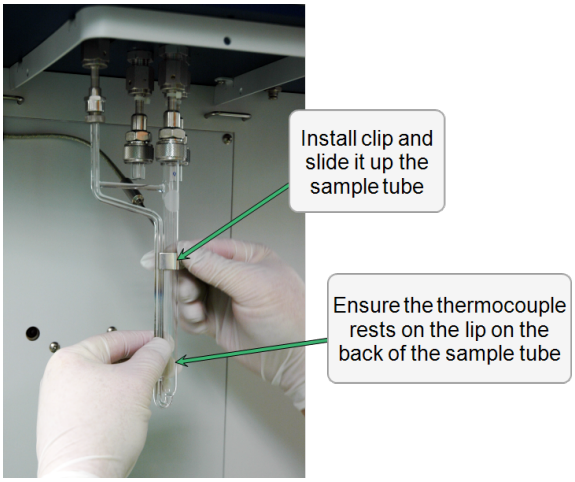
The quartz filler rod can be used for both physisorption and chemisorption analyses; however, the borosilicate glass filler rod can only be used with physisorption analyses. The quartz filler rod can be identified by the notch across the top of the rod.

Use of the borosilicate glass filler rod in a chemisorption analysis can cause damage to the instrument.

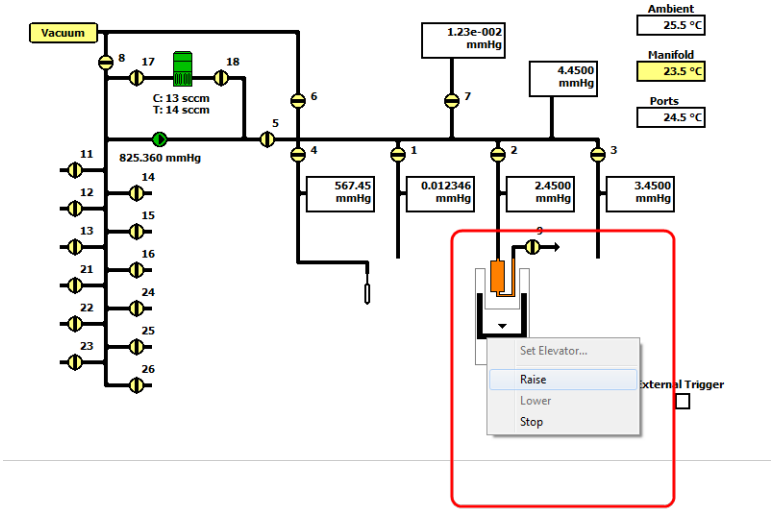
6. Assemble and install the sample and exhaust tube components.



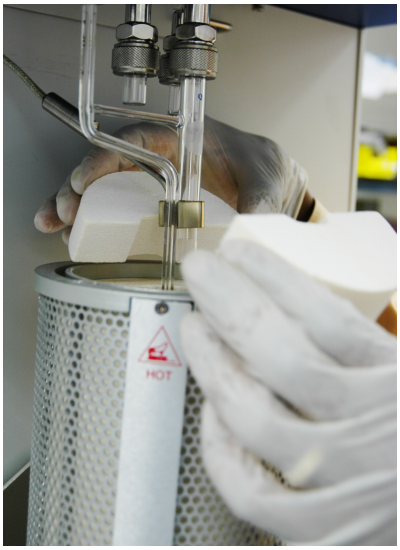
7. Insert the assembled sample tube into the exhaust port and analysis port #2.
8. Slide the connector nuts up the stems and screw the nuts clockwise to secure the tube in place. Hand tighten both connector nuts until snug.



4. Rest the sample thermocouple tip on the lip on the back of the sample tube and install the sample tube clip around the sample tube and thermocouple. Ensure the clip is high enough on the sample tube to clear the furnace disk when the elevator and furnace are raised.
5. Ensure the furnace is on the elevator shelf and manually raise the elevator. To raise the elevator, go to ***Unit [n]*** and ensure there is a check mark to the left of the *Chemisorption* option. If not, select ***Unit [n] > Chemisorption*** to select it.
6. Go to ***Unit [n] > Enable Manual Control***. Ensure a checkmark displays to the left of the menu item. The instrument schematic should display. If not, go to ***Unit [n] > Show Instrument Schematic.***



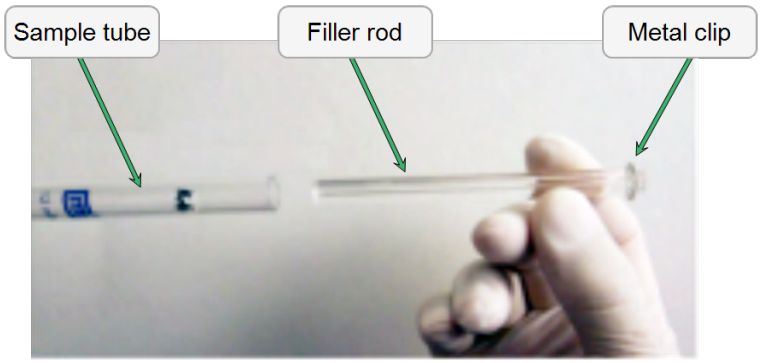
7. On the schematic, right click the furnace icon and select *Raise* to raise the elevator. If it is necessary to stop the elevator, right click the elevator icon again and select *Stop*.
8. When the elevator reaches the top, insert the two furnace disk halves on top of the furnace opening. Place the first disk behind the sample tube and the second disk in front of the sample tube. Ensure the clip remains above the furnace disks.


HOT

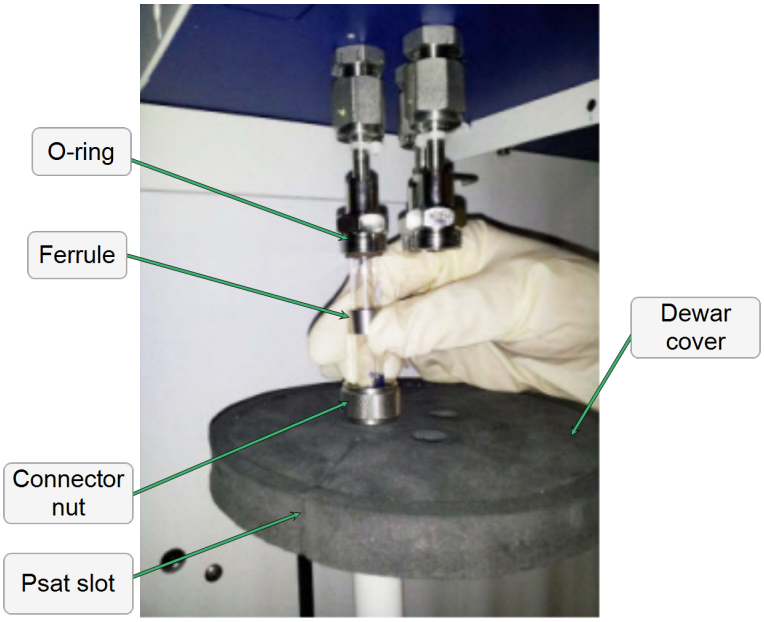
INSTALL THE SAMPLE TUBES FOR PHYSISORPTION

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

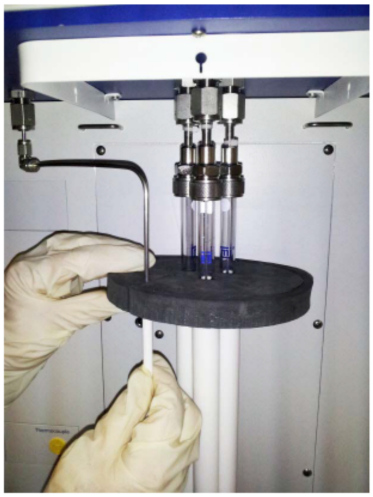
Repeat the following steps for each sample to be installed.

If using...	Then...
A rubber stopper	Remove it.
An isothermal jacket	Slide the jacket down over the stem of the sample tube until it touches the sample tube bulb. The top of the isothermal jacket should be aligned with the mark on the sample tube. If using sample material, insert it into the sample tube.
A filler rod	Hold the sample tube horizontally and carefully slide the filler rod into the tube until the metal clip touches the end of the tube. 

1. Loosen the connector nut on the Psat tube and rotate it out of the way.
2. If using a Check Seal, verify that the port has the Check Seal opener installed or if using a TranSeal, install it at this time.
3. Position the dewar lid so that the slot for the Psat tube is on the left between ports 1 and 2.



4. Insert the sample tube through one of the holes in the dewar lid.
5. Place the sample port nut, ferrule and O-ring onto the sample tube stem.
6. Insert the sample tube into the analysis port and ensure it is completely in the port. Securely hand tighten the sample port nut onto the analysis port.
7. Repeat for each sample tube.
8. Position the dewar lid approximately 3/4 in (19 mm) below the sample port nut.
9. Slide the Psat tube into the Psat slot in the dewar lid and retighten the Psat tube connector nut.
10. Insert the jacket onto the Psat tube. Ensure that the Psat tube jacket is below the dewar lid.



If using the TranSeal, see the instructions included with the TranSeal (part number 350-42803-00).
If using the Check Seal, see the instructions included with the Check Seal (part number 350-42802-00).

INSTALL A VAPOR SOURCE CONTAINER

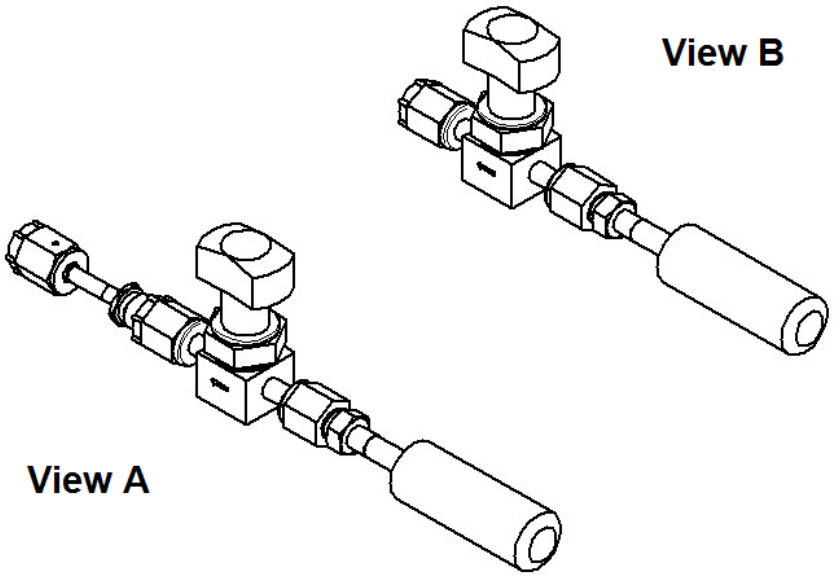
See *Vapor Purification on page I - 1*.

CAUTION

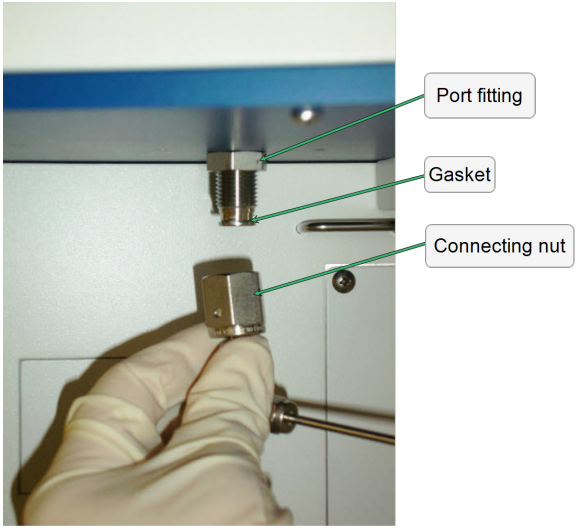
This device has been designed to be used for sample analysis via the analyzer control panel. Any other use may damage this device or the analyzer. Each time the Psat tube or vapor source container is replaced, a new gasket is required. Do not touch the sealing surfaces or the port fitting or gasket with bare hands.

For chemisorption: Only the vapor mantle or the sample furnace can be connected at the time of analysis.

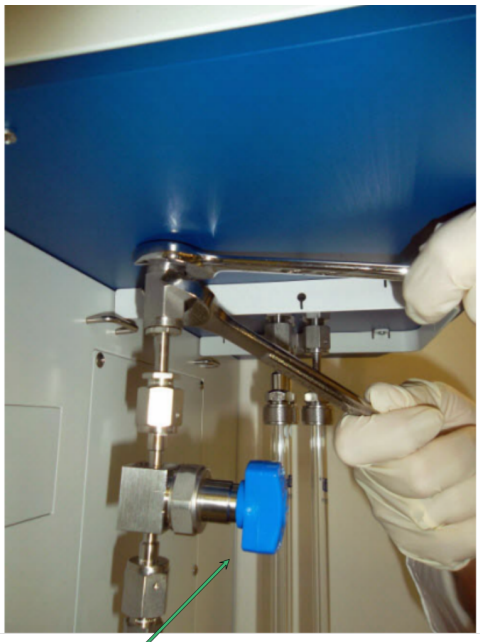
Your equipment may differ slightly. The photos in this section use View A, however, View B is installed in the same manner as View A. If using View B, the Vapor Source Mantle will be slightly shorter than shown in this section.



1. Use an appropriate wrench to loosen the connecting nut from the port fitting by turning the connecting nut counter-clockwise while using a second wrench to hold the port fitting stationary.

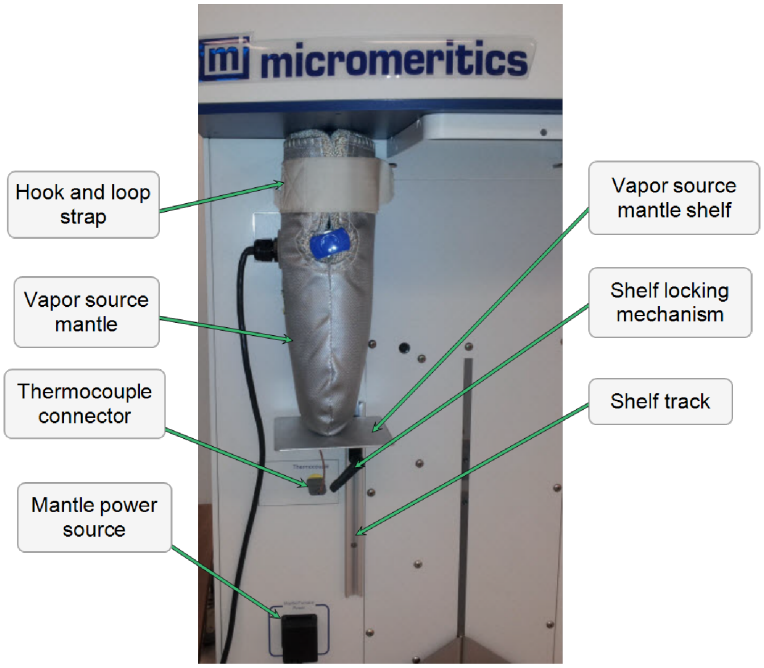


2. Remove the connecting nut and the attached assembly. After removal, the existing seal or a tight-fitting plastic cap can be used to protect the sealing surface assembly from scratches. Prior to reassembly, remove the existing seal or cap, then insert a new seal.
3. Install the vapor source container with a new seal by attaching the connecting nut to the port fitting. Hand tighten the connecting nut by turning clockwise. Use an appropriate size wrench to tighten the assembly an additional 1/8 to 1/4 turn beyond finger tight, while using a second wrench to hold the port fitting stationary on the analyzer.



Turn the vapor source isolation valve to adjust the vapor flow

4. Use the manual controls on the analyzer schematic, evacuate the space above the vapor source by opening valves 4 and 6 with all other valves closed. Then close valve 4 before turning the vapor source isolation valve to the vertical (open) position. The Po / vapor port pressure reading on the instrument schematic will show the vapor pressure.



5. Slide the vapor source mantle over the vapor source container. Extend the blue knob through the circular hole. Secure the hook and loop strap.
6. Insert the thermocouple plug into the connector labeled *Thermocouple*. Insert the power plug into the outlet labeled *Mantle/Furnace Power*.
7. Insert the thermocouple plug into the connector labeled *Thermocouple*. Insert the power plug into the outlet labeled *Mantle/Furnace Power*.
8. If using a support shelf, slide the shelf locking mechanism of the vapor source mantle shelf into the shelf track on the front of the analyzer. Raise the shelf until the vapor mantle is pushed as close as possible to the underside of the upper cabinet. To tighten the shelf, turn the locking mechanism clockwise.

12 Troubleshooting and Maintenance

The instrument has been designed to provide efficient and continuous service; however, certain maintenance procedures should be followed to obtain the best results over the longest period of time.

Most operational problems are caused by:

- Leaks (commonly found at the sample tube O-ring at the analysis port)
- Sample weighing errors
- Use of too much analysis bath fluid in the dewar at the start of an analysis
- Entry of incorrect system volume for analysis
- Impure gas supply

When unexpected analysis results occur, check the above first. Some common operational problems not indicated on the window and their respective causes and solutions are provided in the following table:

What Happened	Why	What to Do
Elevator cannot be raised (or lowered).	Elevator is stuck.	Check for possible obstruction to elevator movement.
Elevator is noisy.	The elevator screw may need greasing.	Contact your Micromeritics Service Representative.
Sample is not within specifications.	There may be a manifold leak.	See *Start Diagnostic Test on page 9 - 1*.
	Gas may be contaminated.	• Perform a blank analysis. If results are good, perform a reference material analysis. • Replace tank. • Check for line leak, which could cause contamination. • Flush the lines occasionally to help prevent contamination.
	Incorrect type of gas line.	Ensure the gas line is all metal. It is best to use the one shipped with the analyzer. Do not use polymer gas lines or flexible gas lines that may be coated internally with a polymer.
High vacuum pump indicator light does not come on.	No power to the high-vacuum pump.	Remove the lower panel on the front of the instrument. Check the power supply plug to the pump. Turn the high-vacuum pump power switch off then back on.

What Happened	Why	What to Do
Vacuum gauge shows reading above 20 mmHg, even after extended pumping through unrestricted valve with analysis or degas ports closed.	Port filter is dirty.	Replace the port filter.
	No power to the vacuum pump.	Check the pump power plug, power switch, and line circuit breaker.
	Oil-based Pumps:	
	Port filter is dirty.	Replace the port filter.
	Vacuum pump oil is low, causing ineffective evacuation	Add or change vacuum pump oil. Add oil to proper level according to the pump's indicator window.
	Alumina in the oil vapor trap is holding moisture because it was not sufficiently dried before being added to the alumina trap.	Replace or dry the alumina.
	Oil free Pumps:	
	High Vacuum pump may have timed out.	Remove the lower front panel from the analyzer. Turn the high-vacuum pump power switch off, then back on.
	The pump diaphragm is worn or damaged.	Contact your Micromeritics service representative.
Vacuum pump is noisy.	Sample tube connector is loose.	Tighten fitting. Replace O-ring.
	Sample tube O-ring is worn or cracked.	Replace O-ring. See *Replace the Sample Port Frit on page 12 - 22*.
	Sample tube is cracked.	Replace with new sample tube.
	No sample tube loaded on a selected port.	Install plug or empty sample tube.
	Gas inlet valve open while vacuum valve open.	With manual control enabled, use the instrument schematic to close gas inlet valve.
Vacuum pump system makes loud continuous noise.	Sample tube connector nut is loose.	Turn the connector nut clockwise to tighten.
	Sample tube fitting is loose.	Tighten the fitting using a wrench.
	Sample tube O-ring is work or cracked.	Replace sample tube O-ring.
	Sample tube is cracked.	Replace the sample tube.
	No sample tube is loaded on selected port.	Ensure the port valve is closed. Install a plug or empty sample tube on the port.
	A gas inlet valve is open while the vacuum valve is open.	Enable manual control then use the analyzer schematic to close the gas inlet valve. See *Show Instrument Schematic on*

What Happened	Why	What to Do
		page 2 - 22.
Valves cannot be operated.	Cable from computer to the instrument is loose.	Reconnect the cable.
	Circuit was opened by the circuit breaker.	Press the **Breaker** button located on the side of the analyzer near the power entrance. Contact your Micromeritics service representative if the button will not stay depressed.

CALIBRATE THE SYSTEM

A calibration file was created specifically for the analyzer and included with the accessories. It is not necessary to recalibrate the system unless it seems out of calibration. Certain calibrations are not allowed unless performed under the direction of a Micromeritics service representative. Those calibrations are disabled on the *Calibration* menu.

To review calibration details of the analyzer, go to ***Unit [n] > Unit Configuration***.

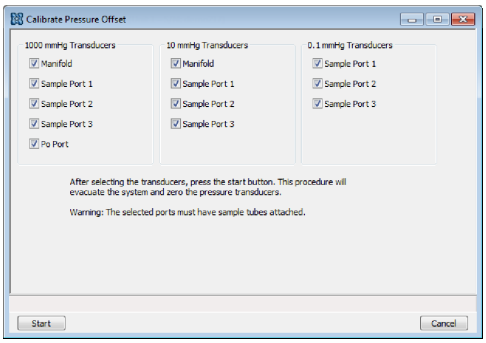
The following calibrations can be performed without the assistance of a service representative:

- Pressure Offset
- Match transducers
- Servo valve

PRESSURE OFFSET

Unit [n] > Calibration > Pressure Offset

This procedure evacuates the system and zeroes the pressure transducers. This calibration should only be performed by qualified service personnel. In order to perform this procedure, sample tubes must be attached to each port.



1. Install a blank sample tube or small plug on each applicable port.
2. Ensure that all applicable transducers are selected, then click **Start**. The window closes when the operation is complete. Click **Cancel** to stop the calibration process.

Pressure Offset Fields and Buttons Table

Field or Button	Description
1000 mmHg Transducers	Select the manifold and / or ports.
10 mmHg Transducers	Select the manifold and / or ports. Enabled only for the manifold and ports with 10 mmHg transducers present.
0.1 mmHg Transducers	Select the ports. Enabled only for ports with 0.1 mmHg transducers present.

For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

MATCH TRANSDUCERS

Unit [n] > Calibration > Match Transducers

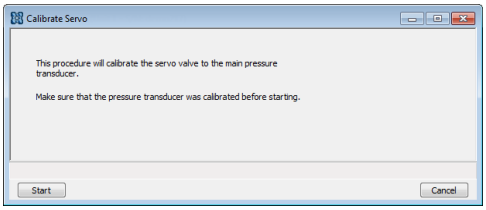
- *Match Transducers on page 10 - 2*

SERVO VALVE

Unit [n] > Calibration > Servo Valve

Ensure the pressure transducer has been calibrated before performing this procedure. Go to ***Unit [n] > Unit Configuration*** and view the calibration information. Contact your service representative if calibration dates are not listed.

Click **Start** to begin the calibration process. The window closes when the calibration is complete.



CLEAN THE POWER SUPPLY AIR FILTER

Two power supply air filters are located on the lower rear panel of the analyzer and should be cleaned or replaced every 30 days (more often in environments with increased levels of dust).

1. Use a flat blade screwdriver to pry the air filter cover from the base. Do not remove the screws. Use caution when removing the cover to avoid breakage.

2. Use an air compressor to remove the dust, or rinse with tap water and dry thoroughly.
3. Replace the filter and cover by pressing the cover back into the base.

GUIDELINES FOR CONNECTING GASES

- Place gas cylinders within 6 feet of the gas inlets of the instrument. Using gas line extenders on gas cylinders located in remote areas may degrade gas quality and reduce pressure. Gas lines are typically five to six feet long. Place the cylinders close enough to allow for proper connection at the analyzer inlet.
- Use a retaining strap (or other appropriate tether) to secure the gas cylinder.
- Always use the gas lines provided with the analyzer. It is very important that proper gas lines are used with the analyzer.
    - **Do not use** polymer tubing for the gas line.
    - **Do not use** flexible gas lines. Some flexible lines may appear to be appropriate, such as those with a herringbone covering, but the line may be coated internally with a polymer.
- Long gas lines, such as those used with gas cylinders placed in remote areas, must be evacuated for an extended period of time to remove ambient gases. When possible, avoid placing gas cylinders in remote locations. It is always better to have gas cylinders located near the analyzer.
- Carefully route the gas lines from the cylinder to the analyzer avoiding overlapping or entangling gas lines. This will help avoid confusion when maintenance is required.
- Label the gas line at the analyzer inlet for proper identification and maintenance.
- Replace gas cylinders before gas is depleted. It is best to replace a gas cylinder when the pressure reads approximately 200 psi on the high pressure gauge. Contaminants absorbed to the walls of the cylinder will desorb as the pressure decreases.
- Ensure the gas cylinder is closed before connecting to the analyzer.

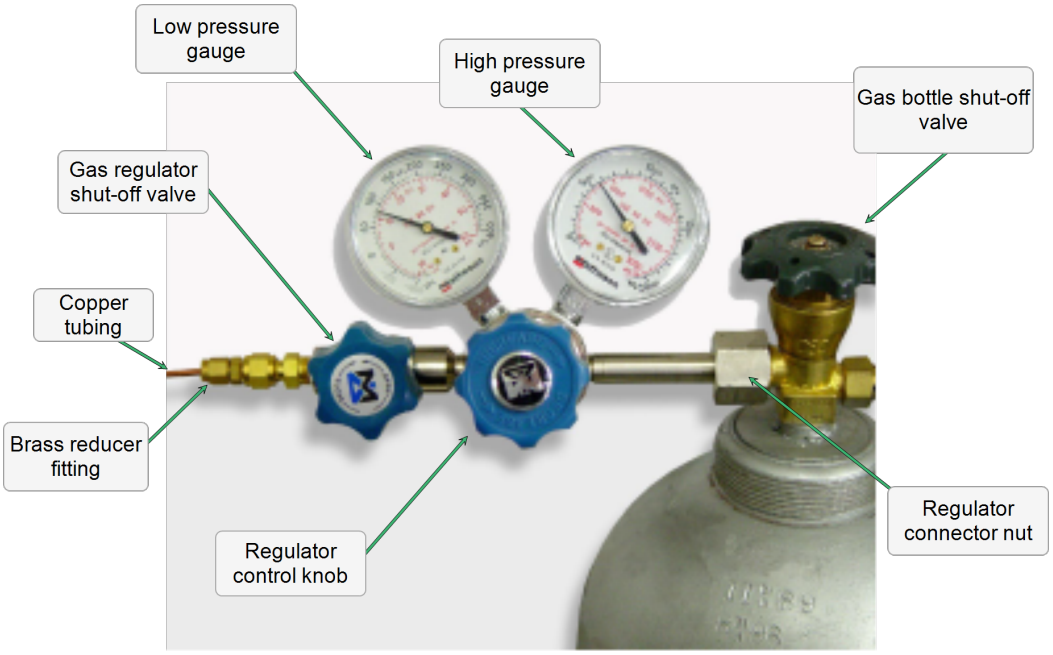
To use oxygen, the analyzer must be equipped with an oxygen-compatible vacuum pump that uses Fomblin® (or a suitable equivalent) pump oil or a dry pump. Failure to use the proper vacuum system could result in hazardous conditions, including fire and personal injury.

CLEAN AND VERIFY THE GAS LINE

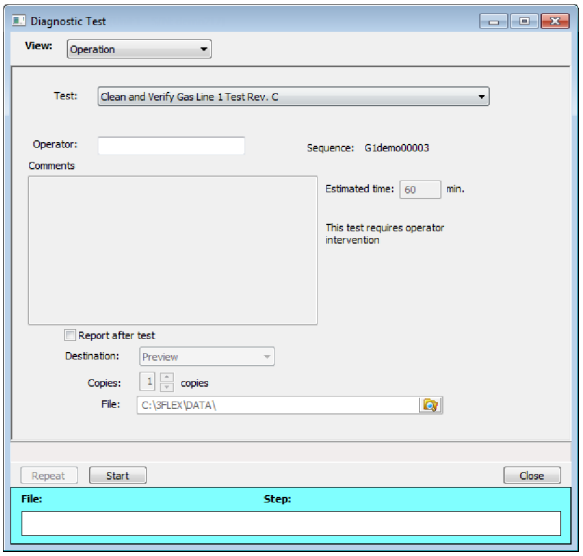
Unit [n] > Diagnostics > Start Diagnostic Test

Always clean the gas lines and verify there are no leaks at the connections after a gas cylinder is connected. This test examines the gas line from the analyzer to the gas cylinder, then from the analyzer to the regulator shutoff valve. A report is generated at the completion of the test to verify that it has passed or failed. Causes and corrective action for a failure are provided.

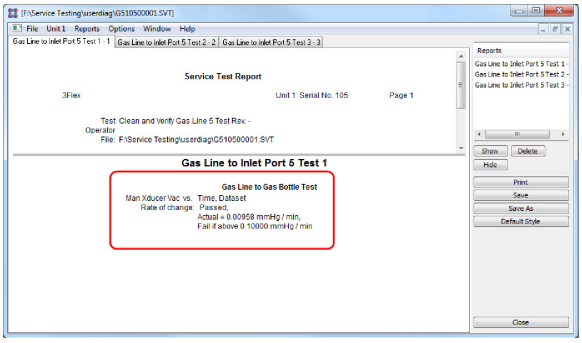
Before beginning, confirm that the state for valves and the low-pressure gauge are as follows:



1. Click the down arrow to the right of the *Test* field, then select *Clean and Verify Analysis Gas Line [n] Test Rev [n]*. The length of time a test will run is also indicated on the window. The *Sequence* field indicates the file created as a result of this test.



2. Resize the window (if necessary) to display the *Report after test*, then select *Preview* as the destination. Click **Start**.
3. From the *View* drop-down list, select either *Operation*, *Instrument Log*, or *Instrument Schematic*.
4. The following series of prompts display on the window requiring operator response.
   a. This is the gas line clean and leak check test for inlet port [*n*]. Inlet ports being tested must be connected to a gas cylinder according to the user manual. A Nupro 'isolation' valve should be installed on the line between the instrument and the regulator.
   b. The test starts with a manual leak check (requires Snoop or equivalent, and IPA), then the line and regulator are evacuated for 20 minutes for cleaning. Next, the leak rate of the gas line is determined.
   c. With the regulator set to 15 psig, open the bottle, regulator shutoff valve, and isolation valve. Check each joint for bubbles with Snoop or equivalent. If a joint is leaking, attempt tightening (without over-tightening) or replace ferrules.
   d. When there are no leaking joints, use IPA to remove water from each joint, then wipe dry.
   e. Close the gas cylinder valve. Leave the regulator shutoff and isolation valves open.
   f. User will be needed in 30 minutes to close the isolation valve. Click **OK** to begin automated testing.
5. A popup window indicates the test is complete. Click **OK**. The reports display.



6. Click each tab across the top portion of the window and look for a reading of *Passed*. A *Passed* reading indicates all valves are in a proper state for operation. If any test shows a *Failed* reading, refer to the following table to help determine the location of the gas leak.

Tab	Test	If *Failed* status, then...
Gas Line to Inlet Port [*n*] Test 1	Gas Line to Gas Cylinder Test	This test will show a reading of *Failed* if any of the other tabs has a *Failed* reading. Correct the failed connection and rerun the test.
Gas Line to Inlet Port [*n*] Test 2	Gas Line to Isolation Valve Test	Check for a leak between the gas line and the isolation valve. Correct the problem and rerun the test.
Gas Line to Inlet Port [*n*] Test 3	Isolation Valve To Bottle Leak Rate	Check for a leak between the isolation valve and the gas cylinder. Correct the problem and rerun the test.

If the *Fail if above* field indicates *Failed*, one or more valves is not in the proper position. Set the valves as shown below, then ensure the appropriate pressure is displayed on the low-pressure gauge.

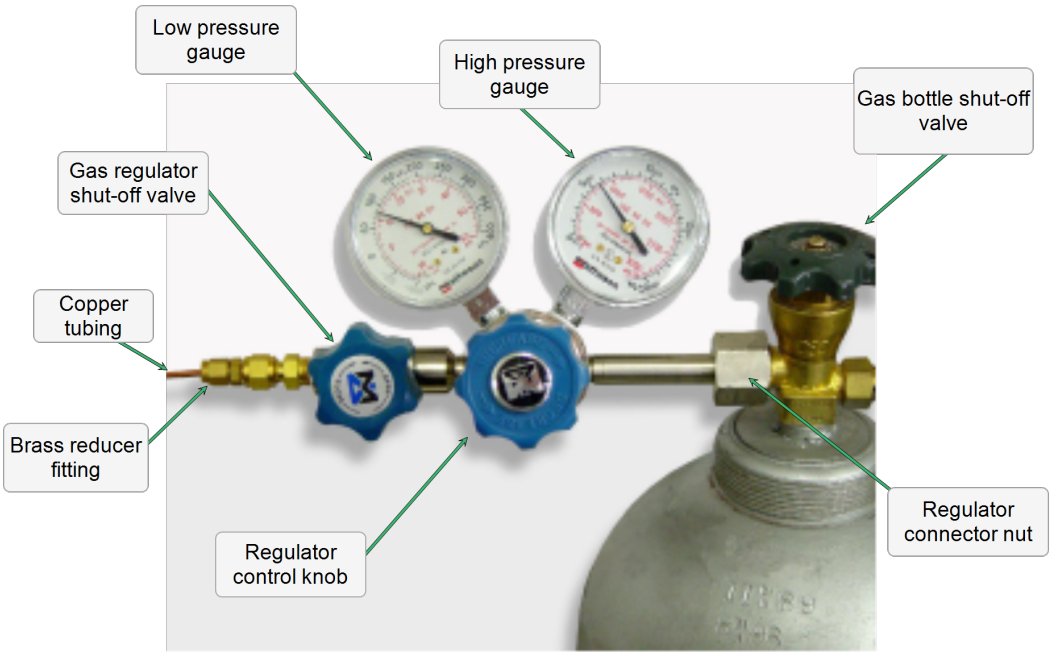
If re-running the test, close the gas cylinder valve before starting the test.

7. Click **Close** to close the test report. Click **Close** again to close the test.

REPLACE A GAS CYLINDER

These instructions apply to working with inert gases only. When working with hazardous gases, follow the safety procedures established by your lab.

A power failure or loss of cryogen can result in dangerous pressures in the sample chamber. When using toxic or flammable gases, additional venting of the cabinet may be required.



Disconnect the Depleted Gas Cylinder

1. Close the gas cylinder shutoff valve by turning the valve clockwise.
2. Disconnect the gas line from the regulator and allow the regulator line to purge. The gas will begin to vent. It is not necessary to disconnect the gas line from the analyzer inlet.
3. Open the gas regulator Shutoff valve by turning the valve counter-clockwise. The gas will continue to vent.
4. Turn the regulator control knob clockwise to open and vent any remaining gas. Both gauges should read at or near zero. If not, open the gas regulator shutoff valve to release gas. It is not necessary to disconnect the gas line from the regulator or the analyzer.

5. Close the regulator control knob.
6. Use an appropriate wrench to loosen the nut at the regulator / gas cylinder connection then remove the regulator from the bottle.
7. Replace the protective cap on the depleted bottle. Disconnect the retaining strap and move the bottle to an appropriate location.

Connect A Gas Cylinder

Regulator Pressure Settings

Analyzer Series	Gauge should indicate
3Flex	15 psig (103 kPag)
AccuPyc	15 psig (103 kPag)
ASAP	10 psig (69 kPag)
AutoChem	5 psig (35 kPag)
AutoPore	45 - 50 psig (310 - 345 kPag)
Gemini	19.5 psig (134.4 kPag)
TriStar	15 psig (03 kPag)

Move the replacement bottle close to the analyzer and tether it into place. It is not necessary to disconnect the gas line from the regulator or the analyzer.

1. Use an appropriate cylinder wrench to remove the protective cap from the replacement gas cylinder. Place the protective cap in a secure location. It will be needed to recap the gas cylinder when it is depleted and replaced.
2. Attach the gas regulator to the gas cylinder connector. Hand tighten the nut, then use an appropriate wrench to tighten an additional 3/4 turn.

CAUTION

Over-tightening the fitting may cause a leak.

3. Check for leaks at the high-pressure side of the regulator and in the connector.
   a. Turn the regulator control knob fully counter-clockwise.
   b. Slowly open the gas cylinder S*hut-off* valve, then quickly close it.
   c. Observe the pressure on the high-pressure gauge for approximately one minute:
      - If the pressure is stable, proceed with the next step.
      - If the pressure decreases, tighten the regulator connector nut until it becomes stable. If the pressure does not remain stable, remove the reg-

ulator and clean all contacts at the regulator connection, then reinstall the regulator.

4. Purge the air from the lines.

Purge the regulator before proceeding to prevent contamination of the analysis gas supply.

   a. Open the gas cylinder valve to pressurize the regulator, then close the valve.
   b. Adjust the *Pressure Control* knob to approximately 5 psi.
   c. Turn the regulator *Shut-off* valve counter-clockwise to open. Allow gas to flow until both gauges ready approximately zero.
   d. Close the regulator *Shut-off* valve to stop gas flow.
   e. Reconnect the gas line to the regulator.
   f. Use two 7/16 in. (11 mm) wrenches to tighten the gas line connection. One wrench fitting steady and the other is used to tighten the connector nut.

5. Set the analyzer pressure.
   a. Turn the *Regulator Control* knob clockwise until the low pressure gauge indicates the appropriate pressure. See ***Regulator Pressure Settings on the previous page***.
   b. Open the regulator *Shut-off* valve.
   c. Open the gas cylinder *Shut-off* valve and flow gas for 10 to 30 seconds.
   d. Close the gas cylinder *Shut-off* valve.
   e. Close the gas cylinder valve.
6. If the gas line to the instrument inlet was previously disconnected, reconnect it now.

SPECIFY GAS PORTS

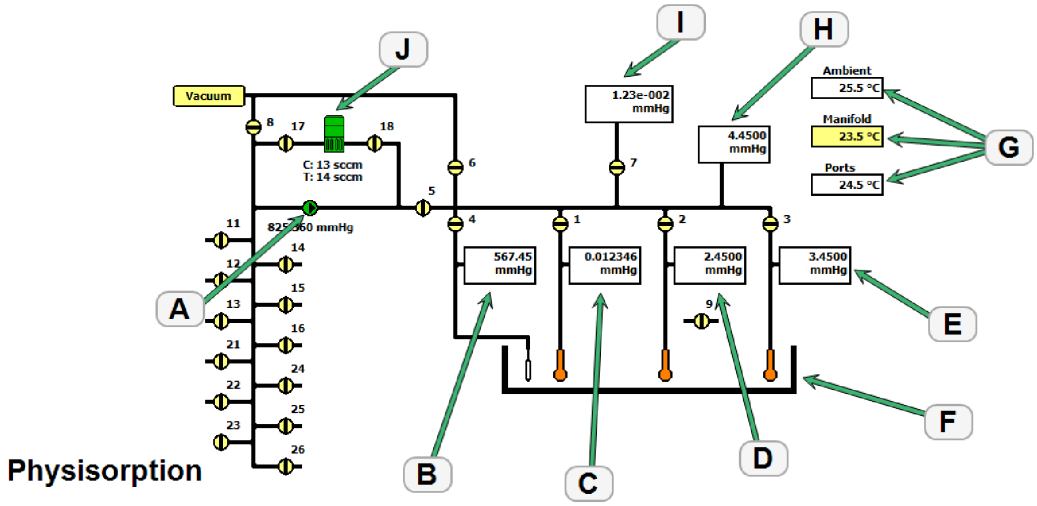
- ***Specify Gas Ports on page 2 - 19***

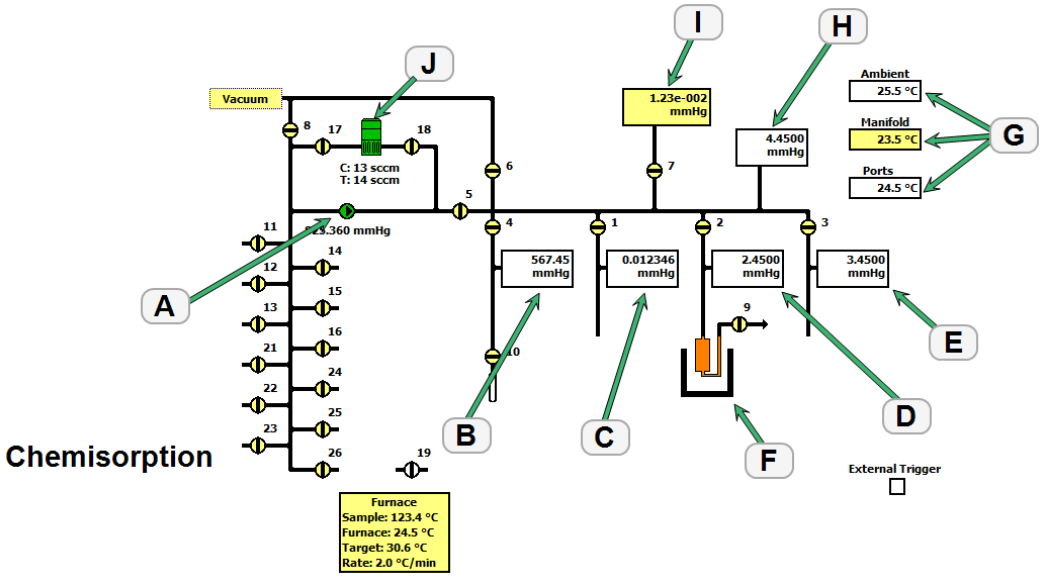
ENABLE MANUAL CONTROL

Unit [n] > Enable Manual Control

Use to enable the manual control of certain system valves and elevator components. When this option is enabled, a checkmark appears to the left of ***Unit [n] > Enable Manual Control***.

If the analyzer schematic is not immediately visible, go to ***Unit [n] > Show Instrument Schematic***.





Analyzer Schematic Components Table

Schematic Components	Description
1-3	Sample ports
4	Po port
5	Servo isolation valve
6	Manifold vacuum
7	Vacuum gauge isolation valve
8	Inlet vacuum
9	Exhaust valve for chemisorption only
10	Reference volume (shown in Service Test mode only)
11-16 and 21-26	Inlet valves
A	Servo valve
B	Po pressure
C	Port 1 pressure
D	Port 2 pressure
E	Port 3 pressure
F	Elevator
G	Temperature sensors
H	Manifold pressure
I	Vacuum gauge
J	Mass flow control

Analyzer Schematic Icon Table

Icon or Symbol	Description
	**Open Valve**. Green indicates an open valve.
	**Closed Valve**. Yellow indicates a closed valve.
	**Servo Valve**. Closed.
	**Servo Valve**. Open.
	**Physisorption Elevator.**

Analyzer Schematic Icon Table (continued)

Icon or Symbol	Description
	Chemisorption Sample Tube and **Furnace Elevator.** The sample tube icon is white when the sample and furnace temperatures are 50 °C or lower. If either the sample or furnace temperature exceeds 50 °C, the sample tube icon turns orange. Temperature readings and ramp rate are displayed below and to the left of the icon. The furnace icon resides on the elevator.
	Chemisorption Mass Flow Controller (MFC). Controls the flow of gas into the sample port. The current (C) rate and the target (T) rate are shown to the right of the MFC icon. Applicable only for the gas used in the Flow prep tasks. The mass flow controller constant is preset for gases provided with the application. See *Chemisorption Tasks on page 4 - 14*.
	Physisorption Sample Tube. Cannot be manually controlled.
Shield Removed	A *Shield Removed* warning indicates the safety shield is not in place.

O-RING COMPATIBILITY

O-ring selection for chemical adsorption measurements is based on temperature, time, and chemical compatibility. Chemical compatibility should be the first consideration when selecting an appropriate o-ring. The time at which the furnace, and subsequently the sample cell, is at elevated temperature can also affect the performance of the O-rings and should be a secondary consideration. Common O-ring materials include Buna-N (nitrile), Viton (fluoroelastomer), and Kalrez (perfluoroelastomer). Kalrez has historically been used extensively for chemical adsorption measurements due to compatibility with a wide range of chemicals and temperatures and should be suitable for all applications of the chemisorption option. Viton or Buna-N may also be suitable for analyses similar to the reference material example file. The ability to re-use Buna-N or Viton O-rings may be limited, while the re-use of Kalrez O-rings should be more broad. Frequency of use and, potentially, several other factors affect the duration of O-ring use, so rigid rules cannot be specified for these materials. Leak rate and ultimate vacuum levels may be used as indicators for O-ring performance.

PERFORM A LEAK TEST

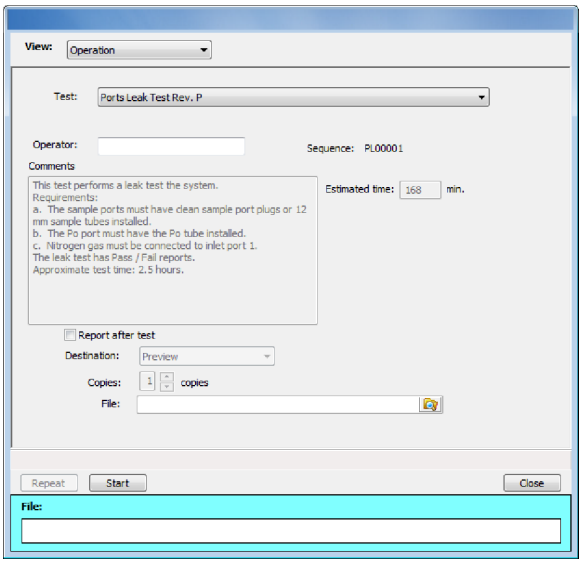
Unit [n] > Diagnostics > Start Diagnostic Test

A service representative may request that a leak test be performed to determine if there is a system leak and may also require a copy of the report generated by this test.

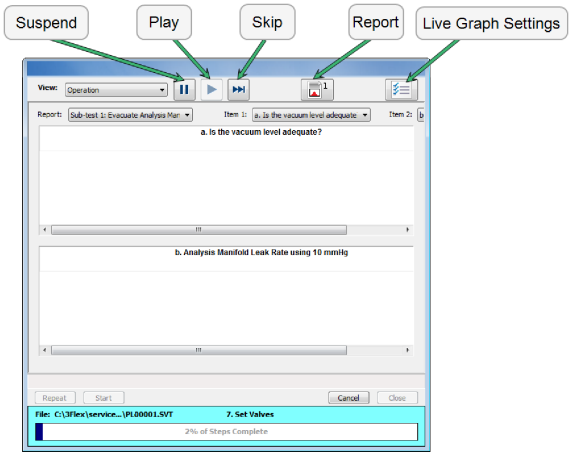
The test provides::

- Prompts on preparing the analyzer for the test
- Approximate time period of the test
- Prompts in which an operator response is required

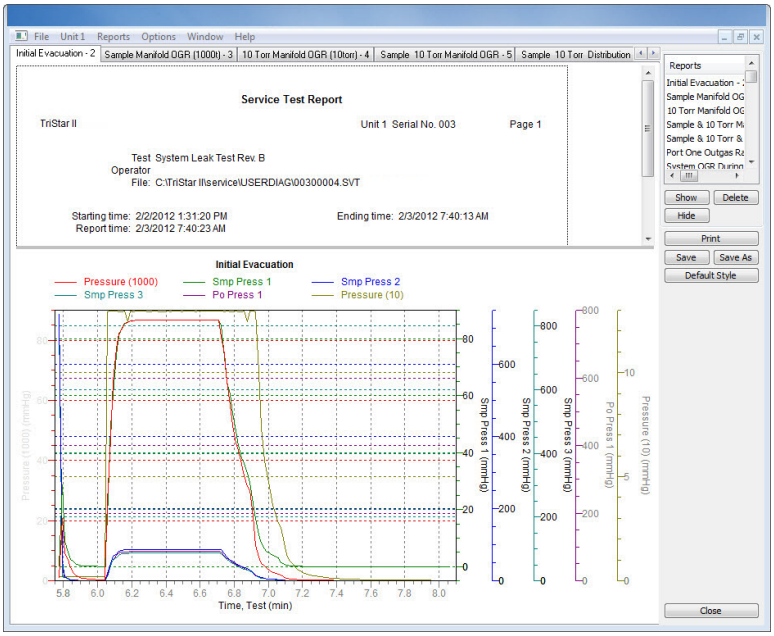
1. Go to ***Unit [n] > Diagnostics > Start Diagnostic Test***.
2. Click the down arrow in the *Test* field, then select the appropriate test.
3. Resize the window so that the *Report after test* section displays at the bottom of the window. Select *Report after test*, *Preview*, and the destination.



4. Click **Start**.
5. Click **Next**. Data will be inserted into the window as they are collected.



6. Use the *Report* drop-down list to select a report to run.
7. Use the *Item 1* and *Item 2* drop-down lists to change the report details that display in the top and bottom boxes.
8. When the test is complete, the report will be displayed.



9. To save the report, click **Save**. Alternatively, click **Save As** to specify a library location, then change the default file name.
10. E-mail the file to the service representative requesting the report.

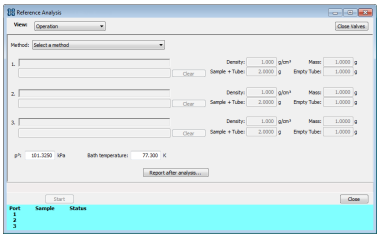
PERFORM A REFERENCE ANALYSIS

Unit [n] > Reference Analysis

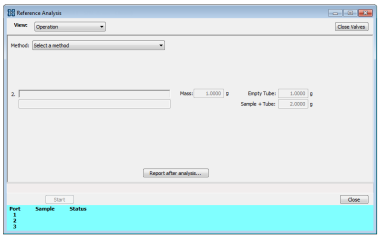
A reference analysis is used to verify the analyzer is operating properly and producing optimum results. These methods provide specifications for critical report quantities and reporting of whether quantities are in or out of specification.

When running a reference analysis, use the appropriate reference material provided in the accessories kit to perform this analysis. The results should match those shown on the label of the reference material bottle, within the tolerance level.

1. Go to ***Unit [n] > Reference Analysis***.
2. Select *Operation* from the *View* drop-down list. The analysis will not start if another view is selected.
3. Click **Browse** from the *Method* drop-down list, select a method, then click **Open**.
4. To manually close all analyzer valves, click **Close Valves**.



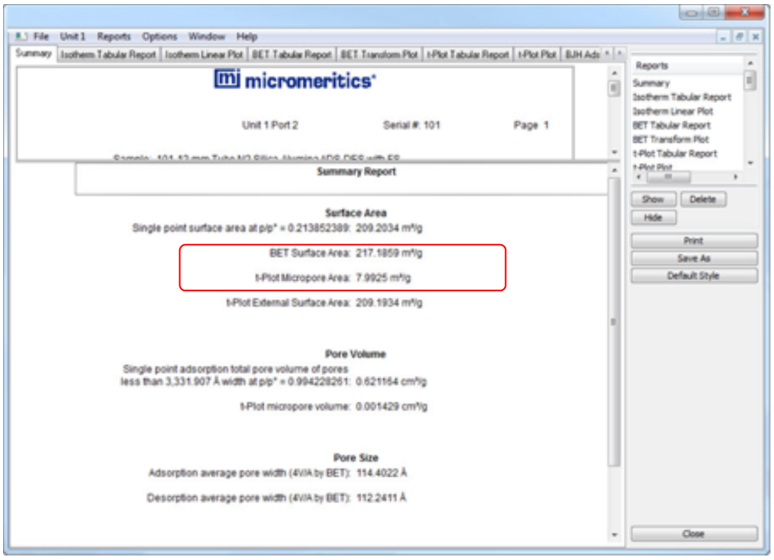
Physisorption



Chemisorption

5. Enter *Mass* for each sample. Verify the populated information is correct and modify if necessary. This information is pulled from the selected file. If a *Blank Analysis* is selected, enter a sample mass of 1 g.For physisorption, the *Density* value is applicable only if using the *Calculate* method for the free space determination.
6. For physisorption: Edit the *po* and *Bath temperature* fields, if necessary.
7. Click **Report after analysis** to generate reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK**.
8. Click **Start** to begin the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.
9. When the analyses are complete, click the *Report Port 1* icon and compare the *BET Surface Are*a shown on the *Summary Report* with the *BET Surface Area* shown on the reference material bottle. The values should match within the tolerance level shown on the bottle. Repeat on ports 2 and 3 if running a physisorption analysis.

   If a *Blank Analysis* was selected, look at the *Blank Analysis Report*. The isotherm points should all fall between the minimum and maximum specification lines.



- If the results are within tolerance, the analyzer is operating properly. Click **Close**.
- If the results are not within tolerance, refer to the following *Cause and Action* table. After performing the action, perform the reference analysis again.

Cause and Action Table

Cause	Action
The sample was not degassed properly.	Degas the sample again.
The gas lines are not clean.	Perform the procedure for cleaning and verifying gas lines, then try again.
The measured free space is too high.	This indicates the helium is not pure enough. Use helium that is 99.999% pure, then try again.

PREVENTIVE MAINTENANCE

Perform the following preventive maintenance procedures to keep the analyzer operating at peak performance. Micromeritics also recommends that preventive maintenance procedures and calibration be performed by a Micromeritics Service Representative every 12 months.

Maintenance Required	Frequency
Dewar	Check and clean weekly.
Power supply air filters	Clean and replace every 30 days (more often in environments with increased levels of dust).
Analyzer exterior	Clean a needed or every 6 months.
Vacuum pump diaphragm	Replace every 12 months.
Port gasket	Replace every 3 to 6 months (depending on the types of analyses that were run).
Sample tube O-ring	Replace as required or every 3 to 6 months.
Test analyzer for leaks	As required or every 12 months.

CLEAN THE ANALYZER

The exterior casing of the analyzer may be cleaned using a clean cloth, dampened with isopropyl alcohol (IPA), a mild detergent, or a 3% hydrogen peroxide solution. Do not use any type of abrasive cleaner.

CAUTION

- Do not allow liquid to penetrate the casing of the analyzer. Doing so could result in damage to the unit.
- Use only a mild detergent in water to clean safety shields. The use of isopropyl alcohol can damage the shield surface.

CLEAN THE DEWAR

When handling dewars, follow the precautions outlined in *Dewar Precautions on page 6 - 1*.

Ice and suspended frost particles may accumulate in the bottom of an analysis port dewar. Particles or deposits exceeding 1/4 in. (0.64 cm) in depth may jam between the bottom of the sample tubes and the bottom of the dewar, causing the dewar not to raise fully. Accumulations of fine particles impede liquid nitrogen circulation around the bottom of the sample tubes. This causes the sample temperature to be slightly higher, which can cause pore volume measurement errors in those samples exhibiting high isotherm slope above 0.97 relative pressure.

Accumulated ice is likely to melt and form a pool of water in the dewar if all liquid nitrogen evaporates. The water must be removed; otherwise, it will solidify when liquid nitrogen is added and could press on the bottom, causing a sample tube breakage.

To ensure problems do not develop due to ice accumulation, check the dewar after each use. Clean the dewar on a weekly basis.

1. Go to ***Unit [n] > Enable Manual Control***. Ensure a checkmark displays to the left of the menu item. The instrument schematic should display. If not, go to ***Unit [n] > Show Instrument Schematic.***
2. .Right click the elevator icon, then select *Lower* to lower the elevator to its lowest position.
3. Remove the dewar, then pour the liquid nitrogen from the dewar into an appropriate cryogenic container.

CAUTION

Do not pour liquid nitrogen directly into a sink. Doing so may cause drain pipes to burst.

4. Rinse with warm water to melt any ice accumulation which may remain in the dewar, then dry thoroughly.

LUBRICATE THE ELEVATOR DRIVE ASSEMBLY

The elevator screw is lubricated before it leaves the factory and should not require lubricating. If the elevator starts to vibrate or becomes noisy when traveling, contact a Micromeritics Service Representative for disposition.

Should lubrication become necessary, apply a light coat of lithium grease to the elevator screw, accessed from the rear of the instrument, as needed.

REPLACE THE PSAT FITTING GASKET

A gasket is attached to the Psat fitting. Instructions for replacing the Psat fitting gasket are located in the following two links.

CAUTION

Each time the Psat tube or vapor source container is replaced, a new gasket is required. To avoid degassing problems, do not touch the sealing surfaces of the port fitting or gasket with bare hands.

REPLACE THE SAMPLE PORT FRIT

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

The chemisorption port frit is changed in the same manner as the sample port frit.

A frit is located in the connecting nut attached to each analysis port. If the frit becomes contaminated, the contaminant may adsorb or desorb during analysis, affecting the results. A contaminated frit on the analysis port may be indicated as a leak or a free space reading much lower than normal.

CAUTION

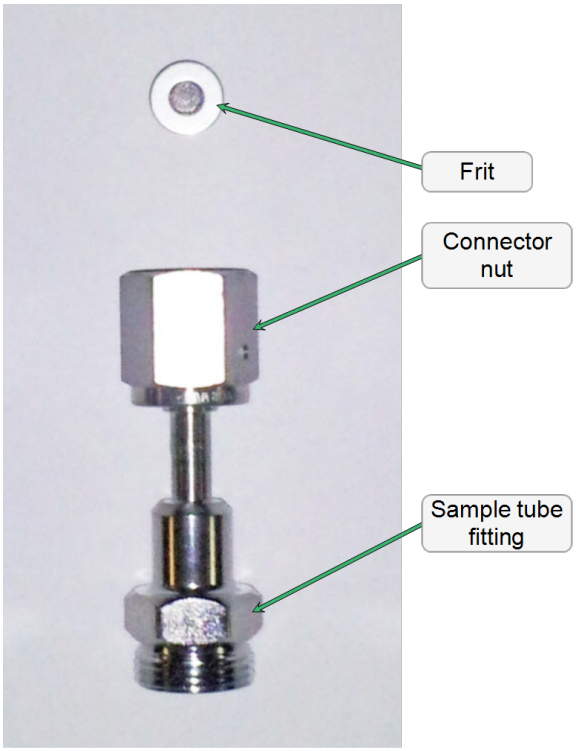
Use a 20 µm frit. The analyzer will not operate properly if an incorrect size is used.

1. Go to ***Unit [n] > Enable Manual Control***. Ensure a checkmark displays to the left of the menu item. The instrument schematic should display. If not, go to ***Unit [n] > Show Instrument Schematic.***
2. Right click on the valve of the appropriate port. If the valve is open, click **Close** to close the valve.
3. Use a wrench to remove the connecting nut from the sample port while using a second wrench to hold the port fitting stationary. Remove and discard the used frit.

CAUTION

To avoid degassing problems, the frit should be clean and should not be touched with bare hands.

4. Place a new frit into the connecting nut.



5. Attach the connecting nut to the sample port fitting and finger tighten. Use a wrench to tighten the nut 1/8 to 1/4 turn past finger tight while using a second wrench to hold the port fitting stationary.

REPLACE THE SAMPLE TUBE O-RING

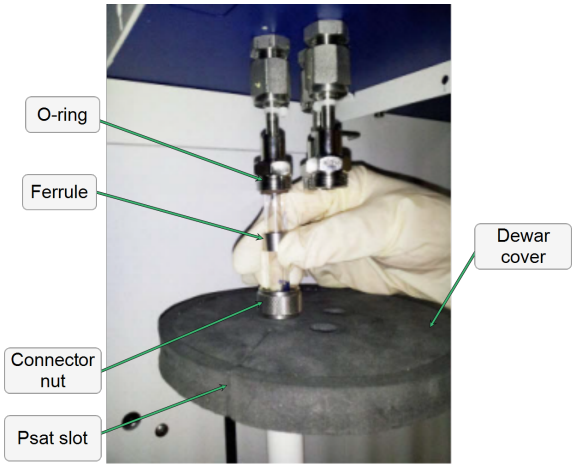
The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

It is important to maintain a vacuum-tight seal near the top of the sample tube stem. If an O-ring becomes worn or cracked, it does not provide a good seal and will need to be replaced.

Before removing (or installing) a sample tube, ensure that the port valve is closed. Observe the analyzer schematic to verify valve status.

1. Carefully remove the dewar from the elevator. Take care not to bump the sample tube bulbs with the dewar during this process. Place the dewar aside.

2. Hold the sample tube firmly with one hand, loosen the sample tube connector nut by turning counter-clockwise.
3. Carefully pull the sample tube down until it is free from the port. It may be necessary to grasp the sample tube with both hands.



4. Remove the O-ring from the top of the sample tube and replace it with a new one.

If the O-ring remains inside the sample port, use a pair of tweezers or needle-nose pliers to remove it.

5. After the new O-ring is in place, insert the sample tube back into the sample port until it is fully seated.
6. Slide the sample tube connector nut up the tube until it comes in contact with the port fitting (the ferrule and O-ring will move along with the connector nut). Then, turning clockwise, hand tighten the connector nut to the sample connector.

REPLACE THE PSAT TUBE FERRULES

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

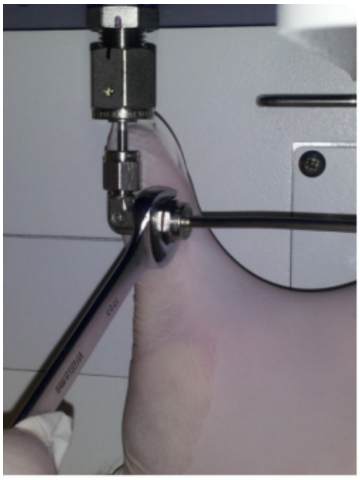
Over an extended period of time, pivoting the Psat tube may cause wear on the nylon and teflon ferrules housed in the Psat tube nuts. If the recommended weekly scheduled P_0 *Port Leak Test* detects a leak by reporting *Failed* on the *Evacuated* or *Pressured* report, the first time a leak is detected, tighten the Psat nuts 1/2 turn and rerun the test. If the leak is still present, replace the nylon and teflon ferrules.

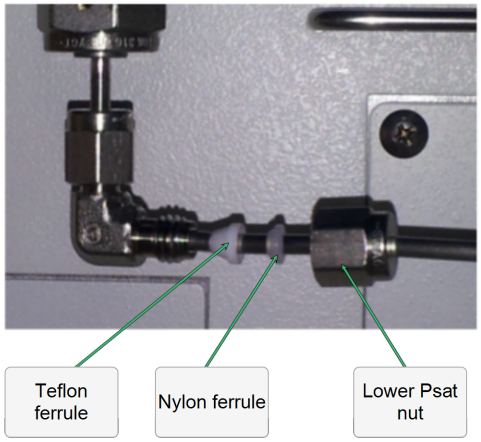
CAUTION

It is recommended that the VCR connector not be removed from the port fitting for this process.

Two ferrule sets are located in the upper and lower Psat nuts. Both sets should be replaced. Additional ferrule sets were included in the analyzer's accessory kit.

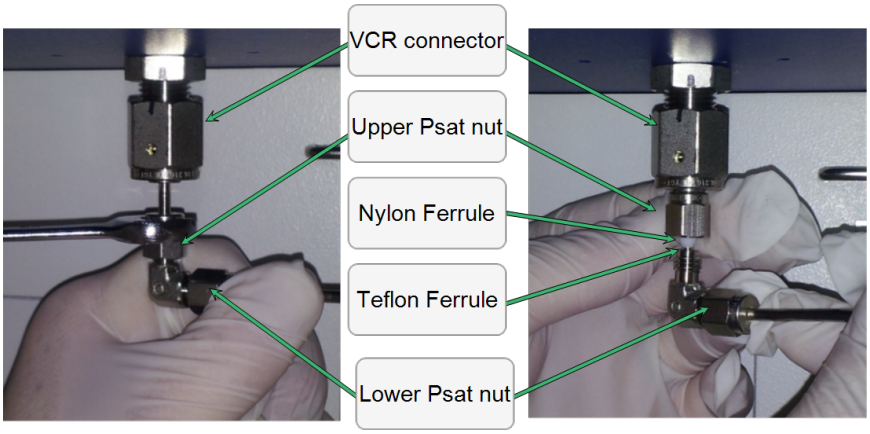
1. Ensure the Psat tube is filled at atmospheric pressure with gas before loosening the Psat nut.
2. To remove the lower Psat nut, use a 7/16 in. (11 mm) wrench to loosen the Psat nut by turning the nut counter-clockwise.





3. Remove the Psat tube from the Psat elbow.
4. Remove the nut from the Psat tube and remove the set of teflon and nylon ferrules from inside the nut. Orient the ferrules as shown with the cone pointed out of the nut.

5. Insert the Psat nut onto the Psat tube, followed by a nylon ferrule, then a teflon ferrule.
6. Insert the Psat tube into the Psat elbow.
7. Hand tighten the Psat nut by turning the nut clockwise. Then use an appropriate size wrench to tighten the nut an additional 3/4 turn while holding the elbow so it does not move.
8. To remove the upper Psat nut, use a 7/16 in. (11 mm) wrench to loosen the Psat nut by turning the nut counter-clockwise.



9. Remove the Psat tube from the VCR connector.
10. Remove the nut from the Psat tube and remove the set of teflon and nylon ferrules from inside the nut.
11. Reinsert the Psat nut onto the VCR connector, followed by a nylon ferrule, then a teflon ferrule. Orient the ferrules as shown with the cone pointed out of the nut.
12. Insert the Psat elbow into the upper Psat nut.
13. Hand tighten the upper Psat nut by turning the nut clockwise. Then use a 7/16 in. (11 mm) wrench to tighten the nut an additional 3/4 turn while holding the elbow so it does not move.

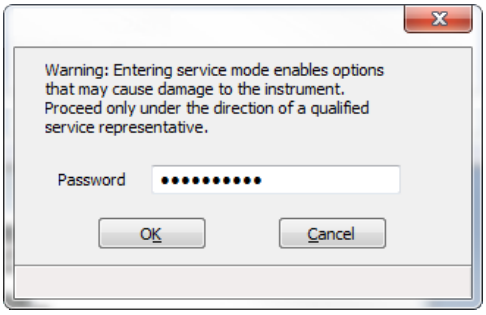
RECOVER FROM A POWER FAILURE

The analyzer saves entered and collected data in case of power failure. File parameters and any other data entered will still be present when power is restored. If an analysis was in progress when the power failure occurred, it will be canceled when the analyzer restarts. Any data collected during the analysis will still be present, but the analysis should be restarted in order to produce complete results.

SERVICE TEST MODE

Options > Service Test Mode

Service Test Mode is a password protected option used to perform certain service tests with the assistance of a trained Micromeritics service representative. This password is supplied by your service representative.



13 Parts and Accessories for the 3Flex

Order system components and accessories using one of the following methods:

- Call our Customer Service Department at 1-770-662-3636
- Email orders to Orders@Micromeritics.com
- Contact your local sales representative

Part Number	Item and Description
Analyzer Optional Equipment	
060-00035-00	FlowPrep 060, degasses up to six samples at up to 400 ºC with flowing gas. A gas regulator is required.
061-00035-00	VacPrep 061, degasses up to six samples at up to 400 ºC; uses flowing gas or evacuation by vacuum (evacuation requires a vacuum pump) and a required gas regulator.
065-00035-00	SmartPrep 065, Windows interface allowing programmable ramp and soak rates; degasses up to six samples with flowing gas. This is the recommended degassing unit because it can be controlled through the software. A gas regulator is required.
067-00000-00	Smart VacPrep (no vacuum pump)
067-00000-11	Smart VacPrep with 110/120V oil-sealed vacuum pump system
067-00000-22	Smart VacPrep with 220/240V oil-sealed vacuum pump system
067-00001-00	Smart VacPrep with hybrid turbo vacuum pump system
350-33015-00	Vapor Adsorption option - provides more thermal control over vapor generation
350-33602-00	Port Upgrade Kit for Micropore option - adds 10 torr and 0.1 torr transducer on each port to be upgraded to micropore capability
Cables	
003-63801-01	Cable, Ethernet straight-thru
Dewar and Accessories	
240-25901-00	Dip stick for checking liquid nitrogen level in dewar
350-25825-00	Dewar, Glass, 3.2 liters
350-25853-04	Shield assembly for dewar and chemisorption furnace
350-31700-00	Dewar lid, for 9 mm sample tubes
350-31701-00	Dewar lid, for 12 mm sample tubes
Furnace	
004-28622-00	(For chemisorption) 8-32 × 1.5 in. long binder head screw, Phillips; used to secure furnace to the elevator tray
Gas Cylinder Accessories	

Part Number	Item and Description
004-25318-00	(For chemisorption) Reducer, 1/4 stub × 1/8 tbg, SST
004-25549-00	Reducer, 1/8 in tube × 1/4 in tube, brass
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator
004-33602-00	Pressure Relief Kit; prevents excessive gas pressure in the event of regulator failure (not to be used with toxic gases)
004-62230-32	Gas Regulator, CGA 320, 30 psig (CO2)
004-62230-58	Gas Regulator, CGA 580 fitting, 30 psig (He, N2, Kr, Ar)
201-25818-00	(For chemisorption) Gas Inlet Line, SS 6 ft
290-25846-00	Gas Inlet Line, 1/8 in × 6 ft, copper
290-25846-01	Gas Inlet Line, 1/8 in × 16 ft, copper
Heating Mantle	
350-53700-00	Degas Mantle Top; replacement top for 350-26000-00 Degas mantle
350-26001-00	Vapor Heating Mantle
350-53701-00	Degas Mantle; for use on instruments below serial number 200
350-53701-01	Degas Mantle Top; replacement top for 350-53701-00 and 350-26002 Degas Mantles
350-26002-00	Degas mantle; for use on instruments above serial number 200
Isothermal Jacket	
350-25812-02	Isothermal Jacket, 9 mm ID
350-25812-03	Isothermal Jacket, 12 mm ID
Miscellaneous	
004-32187-00	(For chemisorption) Glove, Cotton Canvas, 8 oz
Operating Supplies	
004-25891-00	(For chemisorption) Clip, Sample Thermocouple for use with bulb sample tube.
004-28410-01	(For chemisorption) Sample Thermocouple Clip for use with straightwall sample tube.
350-31720-00	Furnace Insulator Set
350-32801-00	(For chemisorption) Exhaust tube for vacuum pump, 3/8 in. ID x 10 ft long
350-32802-00	(For chemisorption) Exhaust tube for instrument, 1/4 in. ID x 10 ft long
350-33609-00	Extended Operating Supplies, 9 mm
350-33610-00	Extended Operating Supplies 12 mm
350-33632-00	Chemisorption Extended Operating Supplies (12-month supply); includes sample tube o-rings, sample port dust frit, reference material, furnace insulator set, quartz hanging filler rod assembly, quartz wool, quartz filter discs, sample tube brush, sample tube stoppers, exhaust tubing.

Part Number	Item and Description
	Sample tubes are not included in the 3500 Chemisorption Extended Operating Supplies kit. Sample tubes must be ordered separately.
350-33635-00	Chemisorption Operating Supplies (6-month supply); includes sample tube o-rings, sample port dust frit, reference material, furnace insulator set, quartz hanging filler rod assembly, quartz wool, quartz filter discs, sample tube brush, sample tube stoppers. Sample tubes are not included in the 3500 Chemisorption Extended Operating Supplies kit. Sample tubes must be ordered separately.
350-63835-00	(For chemisorption) Cable, Sample Thermocouple, DB9, Type K
Reference Material	
004-16821-00	Reference Material, Silica alumina, ~ 215 m^2/g,10 g
004-16825-00	(For chemisorption) Reference material, Platinum on alumina chemi
004-16844-00	Reference material, Y Zeolite
Sample Tubes and Accessories	
004-25013-02	O-Ring, -013 70 Duro Viton, brown
004-25040-05	Gasket, 1/4 in, SS, retainer assembly
004-25040-06	Gasket, 1/2 in, SS, retainer assembly
004-25079-10	(For chemisorption) O-Ring, -009, Viton, brown
004-25225-02	(For chemisorption) Ferrule, 6 mm, rear, stainless
004-25474-00	(For chemisorption) O-ring, -013 Kalrez, Kalez 7075
004-25678-00	(For chemisorption) O-ring, -009 Kalrez, Kalrez 7075
004-27070-00	Frit, 20 µm, 1/4 in. (VCR style)
004-32004-00	Rubber stopper for sample tubes (fits various sample tube sizes)
004-32164-01	(For chemisorption) Quartz Wool, 4 in × 6 in bag
004-32604-08	(For chemisorption) Cap, plastic, .225 D × 5/16,Vinyl
004-54104-01	16 in Brush, for cleaning sample tubes
004-54609-01	(For chemisorption) Brush, 3 mm Dia × 20 mm × 300 mm LG
004-54618-00	Tool for removing the sample port O-ring
004-54805-00	(For chemisorption) Tool, Stuffing Extractor
240-14855-00	Rack for sample tube holder
240-25853-00	Funnel for sample tube
240-32000-00	(For chemisorption) Stopper, for 1/2 in OD tubes
248-32702-00	(For chemisorption) Filter Disc, Quartz (qty. 100)
300-32800-00	Support, sample weighing
350-25843-01	Ferrule for 9 mm sample tube
350-25843-00	Ferrule for 12 mm sample tube

Part Number	Item and Description
350-25864-00	Check Seal Assembly for 12 mm sample tube
350-25874-03	(For chemisorption) Nut, Sample Port, 6 mm
350-25875-00	(For chemisorption) Assembly, Stand, Sample Tube Weighing
350-33032-00	Chemisorption Quartz Filter Discs Kit; includes quartz filter discs (pack of 100), quartz filter discs instructions, glass rod for filter disc installation
350-33033-00	(For chemisorption) Quartz flowthru bulb sample tube kit (includes 2 Quartz flowthru bulb sample tubes and 1 thermocouple clip for use with the bulb sample tube.
350-33603-00	Sample Tube Kit, 12 mm; includes 6 O-rings, 6 stoppers, 3 isothermal jackets, 4 ferrules, 1 Dewar lid, 6 sample tubes, 3 hanging filler rods, 1 sample tube brush
350-33604-00	Sample Tube Kit, 9 mm; includes 6 O-rings, 6 stoppers, 3 isothermal jackets, 4 ferrules, 1 Dewar lid, 6 sample tubes, 3 hanging filler rods
350-33607-00	Check Seal Kit, 12 mm, includes 3 openers, 6 Check Seals, and 1 extractor tool
350-33608-00	TranSeal Kit, 12 mm
350-33636-00	(For chemisorption) Kit, Kalrez O-rings sample tube
350-61002-02	Sample Tube, 9 mm, flat bottom
350-61002-03	Sample Tube, 12 mm, flat bottom
350-61003-00	Hanging Filler Rod for 9 mm sample tube
350-61003-01	Hanging Filler Rod for 12 mm sample tube
350-61003-02	(For chemisorption) Assembly Hanging Filler, Quartz
350-61030-00	(For chemisorption) Tube, Sample Flowthru, Quartz, 12 mm
350-61032-00	(For chemisorption) Tube, Sample Flowthru, Quartz, 12 mm, 6 cc, bulb tube
350-61033-00	(For chemisorption) Glass rod for filter disc installation
Software and Manuals	
350-20800-00	3Flex - current version software
350-42800-00	3Flex - operator's manual
Vacuum Pump and Accessories	
004-25509-00	Clamp, NW 10/16
004-25626-04	Flex Tube, 3/4 in OD × 48 in, NW 16
004-25630-00	Centering Ring, NW 16
004-28998-02	(For chemisorption) Clamp, Hose, 7/32 in -5/8 in
004-62023-03	Kit, FFKM Valves for part # 004-62023-00
062-62803-00	Dry Diaphragm Vacuum Forepump without tray
350-34006-00	Hybrid Pump, 5×10^{-9} mbar

Part Number	Item and Description
Valves	
004-22601-06	Diaphragm Sealed Valve, 1/4 in male
004-62002-05	Solenoid Valve, 24 VDC, 2-way, without BASE
248-60802-36	Servo Valve cable, .030, SST

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A Atomic Weights and Cross Sectional Areas

Atomic Weights and Cross-sectional Areas for Selected Metals

Metal	Symbol	Atomic Weight (g/mole)	Cross-sectional Area (sq nm)	Density (g/mL)
chromium	Cr	51.996	0.0635	7.19
cobalt	Co	58.933	0.0662	8.9
copper	Cu	63.54	0.0680	8.96
gold	Au	196.967	0.08696	18.9
hafnium	Hf	178.490	0.0862	13.3
iridium	Ir	192.220	0.0769	22.4
iron	Fe	55.847	0.0613	7.89
manganese	Mn	54.938	0.0714	7.43
molybdnum	Mo	95.940	0.0730	10.22
nickel	Ni	58.710	0.0649	8.9
niobium	Nb	92.906	0.0806	8.57
osmium	Os	190.220	0.0629	22.6
palladium	Pd	106.400	0.0787	12.02
platinum	Pt	195.090	0.0800	21.45
rhenium	Re	186.2	0.0649	21.02
rhodium	Rh	102.905	0.0752	12.1
tuthenium	Ru	101.070	0.0613	12.4
silver	Ag	107.868	0.0869	10.5
tantalum	Ta	180.947	0.0800	16.6
thorium	Th	232.038	0.1350	11.7
tin	Sn	118.710	0.1082	4.54
tungsten	W	183.850	0.0741	19.3
vanadium	V	50.942	0.0680	6.11
zirconium	Zr	91.220	0.0877	6.51

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B CALCULATIONS

ALPHA-S METHOD

The alpha-S curve is calculated from the reference isotherm by dividing each quantity adsorbed by the quantity adsorbed at 0.4 relative pressure.

$$\alpha_s = Q/Q_{0.4}$$

where $Q_{0.4}$ is found by linear interpolation.

A least-squares analysis fit is performed on the (α_i,Q) pairs. The following are calculated:

a. Slope (s cm^3/g STP)
b. Y-intercept (Q_0 cm^3/g STP)
c. Uncertainty of the slope (u(s) cm^3/g STP)
d. Uncertainty of the Y-intercept (u(Q_0) cm^3/g STP)
e. Correlation coefficient

Surface area is calculated as:

$$A_s = A_{ref} s / Q_{0.4}$$

where A_{ref} is the entered reference surface area.

Pore size is calculated as:

$$V_p = Q_0 / D$$

BET SURFACE AREA

The BET[1] transformation is calculated as:

$$y = \frac{1}{Q\left(\rho^0 / \rho - 1\right)}$$

A least-squares fit is performed on the (P_{rel}, y). The following are calculated:

a. Slope (S cm^3/g STP)
b. Y-intercept (y_0 cm^3/g STP)
c. Uncertainty of the slope (u(s) cm^3/g STP)
d. Uncertainty of the Y-intercept (u(y_0) cm^3/g STP)
e. Correlation coefficient

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

BET Surface Area (m^2/g):

$$A_s = \frac{A_m N_A}{V_{m(s+y_0)}} \times 10^{-18} m^2 / nm^2$$

BET C value:

$$C = s / y_0 - 1$$

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

$$Q_m = 1 / C y_0 = \frac{1}{s + y_0}$$

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

$$u(A_s) = \frac{\sqrt{u^2(s) + u^2(y_0)}}{s + y_0}$$

[1] Brunauer, S.; Emmett, P.H.; and Teller, E., J. Am. Chem. Soc. 60, 309 (1938).

BJH Pore Volume and Area Distribution

For adsorption data, the relative pressure and quantity adsorbed data point pairs collected during an analysis must be arranged in reverse order from which the points were collected during analysis. All calculations are performed based on a desorption model, regardless of whether adsorption or desorption data are being used.

The data used in these calculations must be in order of strictly decreasing numerical value. Points which do not meet this criterion are omitted. The remaining data set is composed of relative pressure (P), quantity adsorbed (Q) pairs from (P_1, Q_1) to (P, Q_n) where ($P_n = 0$, $Q_n = 0$) is assumed as a final point. Each data pair represents an interval boundary (or desorption step boundary) for intervals $i=1$ to $i=n-1$ where n = total number of (P, Q) pairs.

Generally, the desorption branch of an isotherm is used to relate the amount of adsorbate lost in a desorption step to the average size of pores emptied in the step. A pore loses its condensed liquid adsorbate, known as the core of the pore, at a particular relative pressure related to the core radius by the Kelvin[1] equation. After the core has evaporated, a layer of adsorbate remains on the wall of the pore. The thickness of this layer is calculated for a particular relative pressure from the thickness equation. This layer becomes thinner with successive decreases in pressure, so that the measured quantity of gas desorbed in a step is composed of a quantity equivalent to the liquid cores evaporated in that step plus the quantity desorbed from the pore walls of pores whose cores have been evaporated in that and previous steps. Barrett, Joyner, and Halenda[2] developed the method (known as the BJH method) which incorporates these ideas. The algorithm used is an implementation of the BJH method.

Explanation of Terms

A pore filled with condensed liquid nitrogen has three zones:

a. The *core* - evaporates all at once when the critical pressure for that radius is reached; the relationship between the core radius and the critical pressure is defined by the Kelvin equation.
b. The *adsorbed layer* - composed of adsorbed gas that is stripped off a bit at a time with each pressure step; the relationship between the thickness of the layer and the relative pressure is defined by the thickness equation.
c. The *walls of the cylindrical pore* - the diameter of the empty pore is required to determine the pore volume and pore area. End area is neglected.

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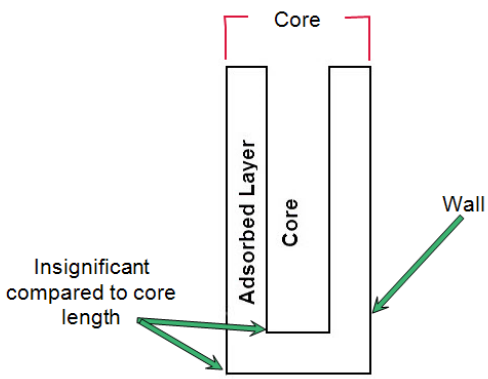
1) Kelvin, J. (published under the name of Sir William Thomson), Phil. Mag. 42, 448-452 (1871).
2) Barrett, E.P.; Joyner, L.S.; and Halenda, P.., J. Am. Chem. Soc. 73, 373-380 (1951).



CALCULATIONS

The quantities adsorbed (Qa) are converted to the liquid equivalent volumes (Vl, cm^3/g):

$$Vl_i = \frac{Q_i V_{mol}}{22414 cm^3 STP}$$

where V_{mol} is the liquid molar volume from the fluid property information.

The relative pressure (P_i) is assumed to be close to unity so that substantially all the pores in the sample are filled.

The corresponding Kelvin core radius is calculated. Only pores smaller than this size will be included:

$$Rc_i = \frac{-A}{(1+F)\ \ln(P_i)}$$

where

A	=	adsorbate property factor (from the *BJH Adsorptive Options* window)
F	=	fraction of pores open at both ends (from the *BJH Adsorption Report Options* window or the *BJH Desorption Report Options* window); assumed to be zero for desorption
Rc	=	Kelvin radius (Å) of core

This radius will be adjusted for the thickness of the adsorbed layer during subsequent calculation steps.

The following calculations (a-c) are made for each relative pressure interval based on the increment of volume desorbed during that interval:

where

i	=	interval number, that is $i=1$ for the first interval from P_1 to P_2, and so on

j	=	each previous interval during which new pores were found
k	=	the total number of intervals in which new pores have been found. It is also the number of lines reported on the BJH table for collected data

a. The thickness of the adsorbed layer at the end of the interval is calculated using the equation located in ***Thickness Curve on page B - 50***.

For the last pressure interval from the lowest Pr_i to zero relative pressure, reference the calculations from the equations in ***Thickness Curve on page B - 50***.

For the first pressure interval, there are no previously opened pores so the volume of liquid desorbed from walls of previously opened pores is zero ($Vd_1 = 0$), and the remainder of Step (a) is skipped.

The change in thickness of the wall layer due to desorption from previously opened pores is calculated as:

$$\Delta Tw = Tw_1 - Tw_{i+1}$$

The annular cross-sectional area of the wall layer desorbed is calculated for all previously opened pores:

$$CSA_j = \pi\left[\left(Rc_j + \Delta Tw\right)^2 - Rc^2{}_j\right]\left(10^{-16}\ \frac{cm^2}{\text{Å}^2}\right)$$

The total volume of gas desorbed from walls of previously opened pores is calculated:

$$Vd_i = \sum_j \left(LP_j\right)\left(CSAa_j\right)$$ for all previously opened pores

where LP_j = length of previously opened pores as calculated in Step b(2).

b. The physical processes occurring for this pressure interval are determined as:

1. If Vd_i is greater than the current increment of volume desorbed (Vl_i - Vl_{i+1}), desorption from walls only is occurring. Total surface of walls exposed thus far (cm^2/g) is calculated as:

$$SA_w = \sum_j \pi\left(LP_j\right)\left(D_{avg,j}\right)\left(\frac{10^{-8}cm}{\text{Å}}\right)$$ for all previously opened pores

where

$D_{avg,j}$ = weighted average pore diameter calculated in Step b.2.

A new layer thickness (ΔTw) that will not overcompensate for the actual volume desorbed in this interval is calculated:

$$\Delta Tw = \frac{\left(Vl_i - Vl_{i+1}\right)\left(10^8 \frac{\text{Å}}{cm}\right)}{SAw_i}$$

Since no cores are evaporated in this pressure interval, no new pores are revealed. Thus no ending Kelvin radius and average pore diameter are calculated for this interval. Note that this means the report may have fewer tabulated intervals on the collected data report than experimental pressure intervals.

2. If Vd_i is less than the volume increment desorbed during this interval (Vl_i-Vl_{i+1}), the remaining volume is due to new pores with core evaporation taking place in this interval. K, the number of intervals with new pores exposed, is increased by 1. (For the interval from the lowest Pr_1 to zero relative pressure, no new pore volume is calculated and the rest of Step b is skipped.)

The volume desorbed from newly opened pores in this interval is calculated as:

$$Vc_i = (VI_i - VI_{i+1}) - Vd_i$$

The Kelvin radius for the end of the interval is calculated as:

$$Rc_{k+1} = \frac{-A}{(1+F)\ln(P_{i+1})}$$

All new pores opened in this interval are represented by one pore having a length-weighted average pore diameter and a corresponding length sufficient to account for the required volume of adsorbate. The weighted average pore diameter is calculated as:

$$D_{avg,k} = \frac{2(Rc_k + Rc_{k+1})\,(Rc_k)\,(Rc_{k+1})}{Rc_k^{\,2} + Rc_{k+1}^{\,2}}$$

$D_{avg,k}$ is the diameter of a pore which would have a surface area that is the average of the areas for pores radius Rc_k and Rc_{k+1}, if its length was the mean of the lengths at those radii.

The relative pressure corresponding to $D_{avg,k}$ is calculated as:

$$P_{avg,k} = \ln^{\exp}\left[\frac{-2A}{\left(1+F\right)\left(D_{avg,k}\right)}\right]$$

The thickness of the adsorbed layer at this pressure is calculated as:

$$Tw_{avg,k} = HP1\left[\frac{HP2}{\ln\left(P_{avg,k}\right)}\right]^{HP3}$$

The decrease in thickness of the wall layer by desorption from the walls of new pores during the lower portion of the pressure interval is calculated as:

$$\Delta Td = Tw_{avg,k} - Tw_{i+1}$$

The cross-sectional area of the newly opened pores is calculated as:

$$CSAc_k = \left[\frac{D_{avg,k}}{2} + \Delta Td\right]^2\left(\frac{10^{-16}cm^2}{\text{Å}^2}\right)$$

The length of the newly opened pores is calculated as:

$$LP_k = \frac{Vc_i}{CSAc_k}$$

Pore diameters and radii are adjusted for the change in thickness of the adsorbed wall layer during this interval. If new pores were opened during this interval, the average diameter is adjusted by the change in layer thickness during the second portion of the desorption interval as:

$$D_{avg,k,new} = D_{avg,k,old} + 2\left(\Delta Td\right)$$

The layer thickness change during the whole interval is added to diameters of previously opened pores as:

$$D_{avg,k,new} = D_{avg,k,old} + 2\left(\Delta Tdw\right)$$

(not including $D_{avg,k}$)

The layer thickness change desorbed during this interval also is added to the radii corresponding to the ends of the pressure intervals as:

$$Rc_{j,new} = Rc_{j,old} + \Delta Tw$$

for all except Rc_{k+1}.

Steps a to c are repeated for each pressure interval.

After the above calculations have been performed, the diameters corresponding to the ends of the intervals are calculated as:

$$Dp_j = 2\left(Rc_j\right)$$

for all Rc_j including Rc_{k+1}.

The remaining calculations are based on Dp_i, $D_{avg,i}$, and LP_i. These calculations are only done for $D_{avg,i}$ values that fall between the Minimum BJH diameter and the Maximum BJH diameter specified by the operator on the *BJH Adsorption Report Options* window or the *BJH Desorption Report Options* window.

(1) Incremental Pore Volume (Vp_i, cm^3/g):

$$Vp_i = \pi\left(Lp_i\right)\left[\frac{D_{avg,i}}{2}\right]^2\left[\frac{10^{16}cm^2}{\text{Å}^2}\right]$$

(2) Cumulative Pore Volume ($Vp_{cum,i}$, cm^3/g):

$$VP_{cum,i} = \sum_j Vp_j for\left(J \le 1\right)$$

(3) Incremental Surface Area (SA_i, m^2/g):

$$SA_i = \pi\left(LP_i\right)\left(\frac{10^{-2}m}{cm}\right)\left(D_{avg,i}\right)\left(\frac{10^{-10}m}{\text{Å}}\right)$$

(4) Cumulative Surface Area ($SA_{cum,i}$, m^2/g):

$$SA_{cum,10} = \sum SA_j \quad \text{for} \quad J \le 1$$

(5) dV/dD pore volume (dV/dD_i, cm^3/g-A):

$$\frac{dV}{dD_i} = \frac{VP_i}{Dp_i - Dp_{i+1}}$$

(6) *dV/dlog(D)* pore volume ($dV/dlog(D)_i$, cm^3g):

$$\frac{dDv}{d\log D_i} = \frac{VP_i}{\log\left(\frac{Dp_i}{Dp_{i+1}}\right)}$$

(7) *dA/dD* pore area (dA/dD_i, m^2/g-A):

$$\frac{dA}{dD_i} = \frac{SA_i}{Dp_i - Dp_{i+1}}$$

(8) *dA/dlog(D)* pore area [$dA/dlog(D)_i$, m^2/g]:

$$\frac{dA}{d\log D_i} = \frac{SA_i}{\log\left(\frac{Dp_i}{Dp_{i+1}}\right)}$$

For fixed pore size tables (if selected), the following calculations are performed:

(1) Average Fixed Pore Size ($DF_{avg,j}$, *A*):

$$DF_{avg,j} = \frac{DP_{F_j} + Dp_{F_{j+1}}}{2}$$

calculated for all intervals in the fixed pore size table.

For the intervals with between the Minimum BJH diameter and the Maximum BJH diameter.

(2) Cumulative Pore volume ($VpF_{cum,i}$, cm^3/g):

$$VpF_{cum,i} = INTERP\left(DpF_{i+1}\right)$$

where *INTERP(x)* is the value interpolated from the function $X = Dp_{j+i}$ and

$Y = VP_{cum,i}$, using an AKIMA semi-spline interpolation.

(3) Incremental Pore Volume (VpF_i, cm^3/g):

$$VpF_i = VpF_{cum,i} - VpF_{cum,i-1}$$

where $VpF_{cum,0} = 0$.

(4) Cumulative Surface Area ($SAF_{cum,i}$, m^2/g):

$$SAF_{cum,i} = INTERP\left(DpF_{i+1}\right)$$

where *INTERP(x)* is the value interpolated from the function $X = Dp_{j+i}$ and $Y = SA_{cum,j}$.

(5) Incremental Surface Area (SAF_i, m^2/g):

$$SAF_i = SAF_{cum,i} - SAF_{cum_{i-1}}$$

where $SAF_{cum,0} = 0$.

(6) *dV/dD* pore volume ($dV/dDpF_i$, cm^3/g-A):

$$\frac{dV}{dDpF_i} = INTERP\left(DpF_{i+1}\right)$$

where *INTERP(x)* is the value interpolated from the function $X = D_{avg,j}$ and $Y = dV/dD_j$.

(7) *dV/dlog(D)* pore volume [$dV/dlog(DpF_i)$, cm^3/g]:

$$\frac{dV}{d\log(DpF_i)} = INTERP\left(DpF_{i+1}\right)$$

where *INTERP(x)* is the value interpolated from the function $X = D_{avg,j}$ and $Y = dV/dlog(D)_j$.

(8) *dA/dD* pore area ($dA/dDpF_i$, m^2/g-A):

$$\frac{dA}{dDpF1} = INTERP\left(DpF_{i+1}\right)$$

where *INTERP(x)* is the value interpolated from the function $X = D_{avg,j}$ and $Y = dA/dD_j$.

(9) *dA/dlog(D)* pore area [$dA/dlog(DpF_i)$, m^2/g]:

$$\frac{dA}{d\log(DpF_i)} = INTERP\left(DpF_{i+1}\right)$$

where *INTERP(x)* is the value interpolated from the function $X = D_{avg,j}$ and

$Y = dA/dlog(D)_j$.

COMPENDIUM OF VARIABLES

ΔTd	=	thickness of layer desorbed from walls of newly opened pores (Å)
ΔTw	=	thickness of adsorbed layer desorbed during interval (Å)
A	=	adsorbate property factor; from the *BJH Adsorptive Options* window
CSA	=	analysis gas molecular cross-sectional area (nm^2), user-entered on the *Adsorptive Properties* window
$CSAa$	=	annular cross-sectional area of the desorbed layer (cm^2)
$CSAc$	=	cross-sectional area of opening of newly opened pores (cm^2)
D_{avg}	=	average pore diameter (Å)
Dp	=	pore (or core) diameter (Å)
F	=	fraction of pores open at both ends; from the *BJH Adsorption Report Options* window or the *BJH Desorption Report Options* window
LP	=	length of pore (cm/g)
P	=	relative pressure
Q	=	quantity adsorbed expressed as a volume (cm^3/g STP)
Rc	=	Kelvin radius (Å) of core
SAw	=	total surface area of walls exposed (cm^2/g)
Tw	=	thickness of remaining adsorbed wall (Å)
Vc	=	volume desorbed from cores of newly opened pores (cm^3/g)
Vd	=	volume of gas desorbed from walls of previously opened pores (cm^3/g)
Vl	=	liquid equivalent volume of volume adsorbed (cm^3/g)
V_{mol}	=	liquid molar volume, from the fluid property information

CRYSTALLITE SIZE

$$d_{xtal} = \frac{1000k}{\overline{\rho} A_{metal}}$$

Where

k	=	shape factor; 6 for sphere, 5 for cube
$\overline{\rho}$	=	weighted average density of the active metals

DFT (DENSITY FUNCTIONAL THEORY)

The adsorption isotherm is known to convey a great deal of information about the energetic heterogeneity and geometric topology of the sample under study. The data of physical adsorption have been used for many years as the basis for methods to characterize the surface area and porosity of adsorbents. Real solid surfaces rarely approach ideal uniformity of structure. It is accepted that in general, the surface of even a nonporous material presents areas of greater or lesser attraction for adsorbed molecules.

This energetic heterogeneity greatly affects the shape of the adsorption isotherm with the result that simple theories such as the Langmuir and BET formulas can, at best, give only approximate estimates of surface area. Porous solids virtually are never characterized by a single pore dimension, but instead exhibit a more or less wide distribution of sizes. The observed adsorption isotherm for a typical material is therefore the convolution of an adsorption process with the distribution of one or more properties which affect that process. This was first stated mathematically by Ross and Olivier[1)] for the case of surface energy distribution and has become known as the integral equation of adsorption.

THE INTEGRAL EQUATION OF ADSORPTION

In a general form for a single component adsorptive, the integral equation of adsorption can be written as

$$Q(p) = \int da\, db\, dc \ldots q(p,\ a,\ b,\ c \ldots) f(a,\ b,\ c \ldots) \qquad (1)$$

where

$Q(p)$	=	the total quantity adsorbed per unit weight at pressure p,
$a,b,c,\ldots$	=	a set of distributed properties,
$f(a,b,c,\ldots)$	=	the distribution function of the properties, and
$q(p,a,b,c,\ldots)$	=	the kernel function describing the adsorption isotherm on unit surface of material with fixed properties $a,b,c,\ldots$

1) Ross and Olivier, J.P., “On Physical Adsorption,” J. Wiley and Sons, New York (1964).

Equation (1), a Fredholm integral of the first kind, is a member of a class of problems known as ill-posed, in that there are an infinite number of functional combinations inside the integral that will provide solutions. Even when the kernel function is known, experimental error in the data can make solving for even a single distribution function a difficult task. Solving for multiple distribution functions requires more data than provided by a single adsorption isotherm.

APPLICATION TO SURFACE ENERGY DISTRIBUTION

Under certain conditions, an energetically heterogeneous surface may be characterized by a distribution of adsorptive energies. The conditions are that the sample is not microporous, i.e., that adsorption is taking place on essentially a free surface with no pore filling processes at least to about 0.2 relative pressure. Secondly, that each energetically distinct patch contributes independently to the total adsorption isotherm in proportion to the fraction of the total surface that it represents. This condition is satisfied if the patches are relatively large compared to an adsorptive molecule, or if the energy gradient along the surface is not steep. In mathematical terms, this concept is expressed by the integral equation of adsorption in the following form.

$$Q(p) = \int d\epsilon \; q(p,\epsilon) f(\epsilon) \tag{2}$$

where

Q(p)	=	the experimental quantity adsorbed per gram at pressure p,
q(p,ε)	=	the quantity adsorbed per unit area at the same pressure, p, on an ideal free surface of energy ε, and
f(ε)	=	the total area of surface of energy ε in the sample.

The exact form of the energy-dependent term depends on the form of the model isotherms expressed in the kernel function and is provided in the model description.

APPLICATION TO PORE SIZE DISTRIBUTION

Similarly, a sample of porous material may be characterized by its distribution of pore sizes. It is assumed in this case that each pore acts independently. Each pore size present then contributes to the total adsorption isotherm in proportion to the fraction of the total area of the sample that it represents. Mathematically, this relation is expressed by

$$Q(p) = \int dH \; q(p,H) \; f(H) \tag{3}$$

where

$Q(p)$	=	the experimental quantity adsorbed at pressure p,
$q(p,H)$	=	the quantity adsorbed per unit area at the same pressure, p, in an ideal pore of size H, and
$f(H)$	=	the total area of pores of size H in the sample.

Numerical values for the kernel functions in the form of model isotherms can be derived from modern statistical mechanics such as density functional theory or molecular simulations, or can be calculated from one of various classical theories based on the Kelvin equation. Several types are found in the models library.

PERFORMING THE DECONVOLUTION

The integrations in equations (2) and (3) are carried out over all surface energies or pore sizes in the model. The functions $q(p,\varepsilon)$ and $q(p,H)$, which we call the kernel functions, are contained in numeric form as model isotherms. Because, in general, there is no analytic solution for equation (1), the problem is best solved in a discrete form; the integral equation for any distributed property Z becomes a summation

$$Q\left(p\right) = \sum_{i} q\left(p, Z_i\right) f\left(Z_i\right) \tag{4}$$

Given a set of model isotherms, $q(p,Z)$, from a model chosen from the models library and an experimental isotherm, $Q(p)$, contained in a sample information file, the software determines the set of positive values $f(Z)$ that most nearly, in a least squares sense, solves equation (4). The distributed property, surface energy or pore size, is then displayed on the *Report Options* window as a selection of tables or graphs.

REGULARIZATION

DFT allows a selectable regularization (also referred to as smoothing) constraint to be applied during the deconvolution process to avoid over-fitting in the case of noisy data or ill-fitting models. The method used is based on co-minimization of the second derivative of the distribution. The relative weight given to this term is determined by the value of the regularization parameter, which is set on the *DFT Pore Size* or *Surface Energy* window and also is shown in the header of reports. The value of the regularization parameter varies from zero (for no second derivative constraint) to ten (indicating a weight equal to minimizing the residuals), or even larger. When the distribution and residuals obtained change little with the value of the regularization parameter, it indicates that the chosen model provides a good representation of the data. Conversely, a large sensitivity to the regularization parameter might indicate inadequate data or a poor choice of model to represent the data.

DOLLIMORE-HEAL ADSORPTION

The calculations for the Dollimore-Heal reports are the same as those for BJH, except for the calculation of average pore diameter and pore length.

PORE DIAMETER

Pore diameter is determined from the Kelvin radius and thickness equation:

$$D_i = 2r_k P_i + tP_i$$

The average pore diameter is the arithmetic mean of the diameters that bound the interval.

$$\overline{D_i} = \left(\frac{D_i + D_{i+1}}{2}\right)$$

PORE LENGTH

$$l_i = \frac{A_{p,i} + 10^8}{\pi \overline{D}_i}$$

$$A_{p,i} = \frac{4 \times \left(10^8 \Delta V_p\right)}{\overline{D_i}}$$

$$\Delta V_p = Cv\left(D\left(Q_{i-1} - Q_i\right) - \Delta t \times 10^8\left(A_{p,cum} - 2\pi \bar{t} l_{i,cum}\right)\right)$$

$$Cv = \left(\frac{\overline{D_i}}{2(\overline{r_k} + t(P_i) - t(P_{i+1})}\right)^2$$

$$\bar{t} = \frac{\overline{D_i}}{2 - \overline{r_k}}$$

$$\overline{r_k} = \frac{\left(r_{k,i} + r_{k,i+1}\right)}{2}$$

where

ΔV_p = Change in pore volume

$A_{p,i}$	=	Pore surface area
$A_{p,i,cum}$, $l_{i,cum}$	=	Summations over the lengths and areas calculated so far
Cv	=	Volume correction factor
D	=	Density conversion factor
$\overline{r_k}$	=	Average Kelvin radius
$\bar{t}$	=	Average thickness

DUBININ-ASTAKHOV

The Dubinin-Astakhov equation is:

$$\log\left(Q\right) = \log\left(Q_0\right) - \left(\frac{RT}{\beta E_0}\right) \times \left(\log\frac{P_0}{P}\right)^N$$

where

β	=	the affinity coefficient of the analysis gas relative to the P_0 gas, from the *Dubinin Adsorptive Options* window
E_0	=	characteristic energy *(kj/mol)*
N	=	Astakhov exponent, may be optimized or user entered from the *Dubinin Report Options* window
P	=	equilibrium pressure
P_0	=	saturation vapor pressure of gas at temperature T
Q	=	quantity adsorbed at equilibrium pressure (cm^3/g STP)
Q_0	=	the micropore capacity (cm^3/g STP)
R	=	the gas constant (0.0083144 *kj/mol*)
T	=	analysis bath temperature (*K*)

For each point designated for Dubinin-Astakhov calculations, the following calculations are done:

$$LV = \log(Q)$$

$$LP = \log\left(\frac{P_0}{P}\right)^N$$

A least-squares fit is performed on the (*LP,LV*) designated pairs where *LP* is the independent variable and *LV* is the dependent variable. If the user selected *Yes* for the *Optimize Astakhov Exponent* prompt, a systematic search for the optimum value of *N* is conducted by recalculating the linear regression and selecting the value of *N* that gives the smallest standard error of the y-intercept. The exponent *N* is optimized to within 10^{-4}. If the optimum value for *N* is not found in this range, an exponent of 2 is used. The following are calculated:

a. Slope (*S* cm^3/g STP)
b. Y-intercept (*YI* cm^3/g STP)
c. Error of the slope (S_{err} cm^3/g STP)
d. Error of the y-intercept (YI_{err} cm^3/g STP)

e. Correlation coefficient

f. Optimized Astakhov exponent (N)

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

Monolayer Capacity (cm^3/g STP):

$$Q_0 = 10^{YI}$$

Micropore Volume (cm^3/g):

$$V_i = \frac{Q_i V_{mol}}{22414}$$

where

V_{mol} = liquid molar volume conversion factor from the fluid property information

Limiting Micropore Volume (cm^3/g):

$$V_0 = \frac{Q_0 V_{mol}}{22414 cm^3 STP}$$

where

V_{mol} = liquid molar volume from the fluid property information

Error of Limiting Micropore Volume (cm^3/g):

$$V_{0,\,err} = W_0\left(10^{YI_{err}} - 1.0\right)$$

Characteristic Energy (KJ/mol):

$$E = \frac{2.303(RT)}{\beta\left(2.303 \times S\right)^{1/N}}$$

Modal Equivalent Pore Diameter (Å):

$$D_{mode} = 2\left\{\left[\frac{3N}{3N+1}\right]^{1/N} \times \left[\frac{10^3 nm^3 / Å^3}{\beta \cdot E_0}\right]\right\}^{1/3}$$

where

β = affinity coefficient of the analysis gas relative to the P_0 gas from the *Dubinin Adsorptive Options* window

Maximum Differential Pore Volume (cm³/g-Å):

This value is also known as *frequency of the mode*[1] .

$$\frac{dV}{dD_{\text{mode}}}Max = 0.5\left(3N+1\right) W_0\left[\frac{3N+1}{3N}\right]^{1/3N}\left[\frac{\beta \cdot E_0}{\left(\left(10^3 nm^3\right)/\text{Å}^3\right)}\right]^{1/3} \exp\left(-\left[\frac{3N+1}{3N}\right]\right)$$

Mean Equivalent Pore Width (Å):

$$D_{mean} = 2 \times \frac{\left[\frac{\left(10^3 nm^3\right)/\text{Å}^3}{\beta \cdot E_0}\right]^{1/3}}{\Gamma\left(\frac{3N+1}{3N}\right)}$$

Micropore surface area (m²/g):

$$SDA = 1000 \times 2.0 \times W_0 \times \left[\frac{E_0}{k}\right]^{1/3} \times \Gamma\left(\frac{3N+1}{3N}\right)$$

Γ is calculated by a polynomial approximation over the domain $0 \le \times \le 1$:

$$\Gamma\left(x+1\right) = 1 + b_1 x + \quad b_2 x^2 + \ b_3 x^3 + b_4 x^4 + b_5 x^5 + b_6 x^6 + b_7 x^7 + b_8 x^8 + \epsilon x \, |\epsilon x| \le 3\left(10^{-7}\right)$$

where

b_1	=	-0.57719 1652
b_2	=	0.98820 5891
b_3	=	-0.89705 6937
b_4	=	0.91820 6857
b_5	=	-0.75670 4078
b_6	=	0.48219 9394

[1] Ross and Olivier, J.P., "On Physical Adsorption," J. Wiley and Sons, New York (1964).

b_7 = -0.19352 7818

b_8 = 0.03586 8343

and where

$$x + 1 = \left(\frac{3N+1}{3N}\right)$$

Equivalent Pore Diameter (Å):

$$D_i = 2\left[\frac{-\left(\frac{10^3 nm^3 / \text{Å}^3}{\beta \cdot E_0}\right)^N}{\ln(W_i) - \ln(W_0)}\right]^{1/3N}$$

dV/dD Pore Volume (cm^3/g-Å):

$$\frac{dV}{dD_i} = 0.5 \times W_0 \times 3N\left(\frac{10^3 nm^3 / \text{Å}^{3N}}{\beta \cdot E_0}\right)\left(\frac{D_i}{2}\right)^{-(3N+1)} \times \exp\left\{-\left(\frac{10^3 nm^3 / \text{Å}^3}{\beta \cdot E_0}\right)^N \left(\frac{D_i}{2}\right)^{-3N}\right\}$$

DUBININ-RADUSHKEVICH

The Dubinin-Radushkevich[1] equation is:

$$\log\left(Q\right) = \log\left(Q_0\right) - \frac{BT^2}{\beta} \times \left[\log\frac{P_0}{P}\right]^2$$

where

β	=	the affinity coefficient of analysis gas relative to P_0 gas (for this application β is taken to be 1)
B	=	a constant
P_0	=	saturation vapor pressure of gas at temperature T
P	=	equilibrium pressure
Q	=	quantity adsorbed at equilibrium pressure (cm^3/g STP)
Q_0	=	the micropore capacity (cm^3/g STP)
T	=	analysis bath temperature (K), from the P_0 *and Temperature Options* window

For each point designated for Dubinin-Radushkevich calculations, the following calculations are done:

$$LV = \log(Q)$$

$$LP = \log\left(\frac{P_0}{P}\right)^2$$

The intercept, *log(Vo)* can be found by performing a least-squares fit on the (*LP,LV*) designated pairs where *LP* is the independent variable and *LV* is the dependent variable. Assuming the adsorption of gas is restricted to a monolayer, *Vo* is the monolayer capacity. Based on this assumption, the following are calculated:

a. Slope (S cm^3/g STP)
b. Y-intercept (YI cm^3/g STP)
c. Error of the slope (S_{err} cm^3/g STP)

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[1] Dubinin, M., Carbon 21, 359 (1983); Dubinin, M., Progress in Surface and Membrane Science 9, 1, Academic Press, New York (1975); Dubinin, M. and Astakhov, V., Adv. Chem. Ser. 102, 69 (1971); Lamond, T. and Marsh, H., Carbon 1, 281, 293 (1964); Medek, J., Fuel 56, 131 (1977); Polanyi, M., Trans. Faraday Soc. 28, 316 (1932); Radushkevich, L., Zh. fiz. Kemi. 33, 2202 (1949); Stoeckli, H., et al, Carbon 27, 125 (1989).

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

e. Correlation coefficient

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

Monolayer Capacity (cm^3/g STP):

$$Q_0 = 10^{YI}$$

Error of Monolayer Capacity (cm^3/g STP):

$$Q_{0,\,err} = Q_0\left(10^{YI,\,err} - 1.0\right)$$

Micropore surface area (m^2/g):

$$SDP = \frac{\sigma\, Q_0\, N_A}{22414 cm^3\left(\frac{10^{18}\ \ nm^2}{m^2}\right)}$$

where

σ = molecular cross sectional area of gas (nm^2) from the *Adsorptive Properties* window

EQUATION OF STATE

The ideal gas law relates pressure, volume, temperature, and quantity of gas

where

P	=	pressure
R	=	a constant that depends on the units of n
		For n in cm^3, STP $R = \frac{P_{STD}}{T_{STD}}$
		For n in moles, $R = 8.3145\ J\ mol^{-1}\ K^{-1}$
T	=	temperature
V	=	volume
z	=	compressibility factor for the gas at the given pressure and temperature

The real gas equation of state

$$n = \frac{PV}{RTz(P,T)}$$

EQUILIBRATION

Equilibration is reached when the pressure change per equilibration time interval (first derivative) is less than 0.01% of the average pressure during the interval. Both the first derivative and average pressure are calculated using the Savitzky-Golay[1] convolution method for polynomial functions. The following equations are those used to compute weighted average and first derivative, respectively, for the 6th point of an 11-point window.

$$P_{avg} = \frac{-36(P_{11}+P_1)+9(P_{10}+P_2)+44(P_9+P_3)+69(P_8+P_4)+84(P_7+P_5)+89(P_6)}{429}$$

$$P_{chg} = \frac{5(P_{11}-P_1)+4(P_{10}-P_2)+3(P_9-P_3)+2(P_8-P_4)+(P_7-P_5)}{110}$$

$$P_{pcp,i} = 100\,\% \frac{P_{chg}}{P_{avg}}$$ pressure change per equilibration time interval

[1] Savitzky, A. and Golay, M.J.E., Anal. Chem. 36, 1627 (1964).

where the numerical constants are from the Savitzky-Golay convolution arrays, and

P_{avg}	=	average pressure
P_{chg}	=	change in pressure
$P_{pcp,i}$	=	percent change per interval
P_i	=	i^{th} pressure reading taken at equilibrium intervals

If a non-zero value that is too small is entered for the maximum equilibration time, the points are collected before equilibration is reached.

If P_{avg} is greater than 0.995 times the current P_0, equilibration will not take place until the *Minimum equilibration delay for* P/P_0 *0.995* has expired, in addition to the standard equilibration criteria.

F-RATIO METHOD

The *f*-Ratio is the quantity adsorbed divided by the quantity adsorbed in a reference isotherm at the same pressure.

$$f_i = \frac{Q_i}{Q_{ref} \quad P_i}$$

The reference quantity adsorbed is found by spline interpolation of the reference isotherm.

FREE SPACE

The free space is the physical volume below the sample valve. The different temperatures in the sample tube, stem, and port must be accounted for.

Free space volumes are calculated as:

$$n_p = \frac{P_s V_p}{z\left(P_s, T_p\right) T_p}$$

$$n_s = n_d - n_p$$

$$V_s = \frac{n_s z(P_s, T_s) T_s}{P_s}$$

The reported free space is

$$V_f = V_p + V_s$$

The quantity of gas in the free space for a given data point is:

$$n_p = P_s \left(\frac{V_p}{z\left(P_s, T_p\right) T_p} + \frac{V_s}{z(P_s, T_s,) T_s} \right)$$

where

n_d	=	quantity of gas dosed
n_p	=	quantity of gas in the port
n_s	=	quantity of gas in the sample tube
P_s	=	sample (and port) pressure
T_p	=	port temperature
T_s	=	sample temperature
V_p	=	volume of the sample port
V_s	=	volume of the sample tube
$z(P,T)$	=	gas compressibility factor P and temperature T for the gas used

FREUNDLICH ISOTHERM

The Freundlich isotherm has the form

$$\frac{Q}{Q_m} = CP^{\frac{1}{m}}$$

where

C	=	temperature-dependent constant
m	=	temperature-dependent constant
P	=	equilibrated collected pressure measured by gauge at temp T_{amb}
Q	=	quantity of gas adsorbed
Q_m	=	quantity of gas in a monolayer

The pressure is absolute; typically, m > 1. In terms of quantity adsorbed,

$$Q = Q_m CP^{\frac{1}{m}}$$

Taking the log of both sides yields

$$\log Q = \log Q_m C + \frac{1}{m}\log P$$

HORVATH-KAWAZOE

A relative pressure lower limit is determined such that L-d_0 never equals zero. All pressure points less than this limit are discarded. For each collected relative pressure point, values of L are chosen in an iterative manner, and the relative pressure (P/P_0) determined by solving one of the following equations:

- Slit Pore Geometry (original Horvath-Kawazoe)
- Cylinder Pore Geometry (Saito / Foley)
- Sphere Pore Geometry (Cheng / Yang)

SLIT PORE GEOMETRY (ORIGINAL HORVATH-KAWAZOE)

When using the original Horvath-Kawazoe[1)] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\frac{P}{P_0} = \frac{K}{RT}\frac{IP\times 10^{32}}{\sigma^4 L - 2d_0}\frac{\sigma^4}{3L - d_0{}^3} - \frac{\sigma^{10}}{9L - d_0{}^9} - \frac{\sigma^4}{3d_0{}^3} + \frac{\sigma^{10}}{9d_0{}^9}$$

where

10^{32} = the number of cm^4 that are equal to $Å^4$

α = gas solid nuclear separation at zero interaction energy (Å), $\frac{Z_S + Z_A}{2}$

d_0 = $\frac{D_A + D_S}{2}$

where:

D_A	=	molecular diameter (Å) from the *Horvath-Kawazoe Physical Properties* window
D_S	=	diameter of sample atom (Å) from the *Horvath-Kawazoe Physical Properties* window
IP	=	interaction parameter (erg-cm^4) from the *Horvath-Kawazoe Report Options* window
K	=	Avogadro Constant (N_A)
L	=	pore width (nucleus to nucleus) (Å)
P	=	equilibrium pressure
P_0	=	saturation pressure
R	=	gas constant (8.31441×10^7 erg/mol K)

1) Horvath, G. and Kawazoe, K., J. Chem. Eng. Japan 16(6), 470 (1983).

T = analysis bath temperature (*K*), from an entered or calculated value on the *P_0 and Temperature Options* window

where:

Z_s = sample equilibrium diameter at zero interaction energy (Å) from the *Horvath-Kawazoe Physical Properties* window

Z_A = zero interaction energy diameter from the *Horvath-Kawazoe Physical Properties* window

CYLINDER PORE GEOMETRY (SAITO/FOLEY)

When using the Saito/Foley[1)] method, the following equation is solved for each value of *P*. The value of *L* is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{P_0}\right) = \frac{3}{4}\frac{\pi K}{RT} \times \frac{IPx10^{32}}{d_0{}^4} \times \sum_{k=0}^{\infty}\left[\frac{1}{k+1}\left(1-\frac{d_0}{r_p}\right)^{2k} \times \left\{\frac{21}{32}\alpha_k\left(\frac{d_0}{r_p}\right)^{10} - \beta_k\left(\frac{d_0}{r_p}\right)^{4}\right\}\right]$$

where

10^{32} = the number of cm^4 that are equal to $Å^4$

β_k = $\left(\frac{-1.5-k}{k}\right)^2 \beta_{k-1}, \beta_0 = 1.0$

α_k = $\left(\frac{-4.5-k}{k}\right)^2 a_{k-1}, a_0 = 1.0$

d_0 = $\frac{D_A + D_S}{2}$

where

D_A = molecular diameter (Å) from the *Horvath-Kawazoe Physical Properties* window

D_S = diameter of sample atom (Å) from the *Horvath-Kawazoe Physical Properties* window

IP = interaction parameter (10^{-43} erg-cm^4) from the *Horvath-Kawazoe Report Options* window

K = Avogadro Constant (N_A)

1) Saito, A. and Foley, H. C., AlChE Journal 37(3), 429 (1991).

L	=	pore width (nucleus to nucleus) (Å)
P	=	equilibrium pressure
P_o	=	saturation pressure
R	=	gas constant (8.31441×10^7 erg/mol *K)*
r_p	=	radius of the cylindrical pore, $\frac{L}{2}$
T	=	analysis bath temperature (*K*), from an entered or calculated value on the *Po and Temperature Options* window

SPHERE PORE GEOMETRY (CHENG/YANG)

When using the Cheng / Yang[1)] method, the following equation is solved for each value of *P*. The value of *L* is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{P_0}\right) = \frac{6N_1\varepsilon^*_{12} + N_2\varepsilon^*_{22}\,L^3\times 10^{32}}{RTL - d_0{}^3}\left[-\left(\frac{d_0}{L}\right)^6\left(\frac{T_1}{12}+\frac{T_2}{8}\right)+\left(\frac{d_0}{L}\right)^{12}\left(\frac{T_3}{90}+\frac{T_4}{80}\right)\right]$$

where

10^{32}	=	the number of cm^4 that are equal to $Å^4$
ε^*_{12}	=	$\frac{Å_S}{4d_s{}^6}, where Å_s = \frac{6\left(mc^2\right)a_S a_A}{\frac{a_S}{X_S}+\frac{a_A}{X_A}}$
ε^*_{22}	=	$\frac{A_A}{4D_A{}^6}, where Å_A = \frac{3\left(mc^2\right)\left(a_A\right)\left(X_A\right)}{2}$
d_0	=	$\frac{D_A + D_S}{2}$

where

D_A	=	molecular diameter (Å) from the *Horvath-Kawazoe Physical Properties* window
D_S	=	diameter of sample atom (Å) from the *Horvath-Kawazoe Physical Properties* window

L	=	pore width (nucleus to nucleus) (Å)
N_1	=	$4\pi L_2 N_S$, where N_s = number of sample atoms/cm^2 at monolayer

1) Cheng, Linda S. and Yang, Ralph T., Chemical Engineering Science 49(16), 2599-2609 (1994).

N_2	=	$4\pi\ (L\text{-}d_0)^2 N_A$, where N_S = number of gas molecules/cm^2
P	=	equilibrium pressure
P_0	=	saturation pressure
R	=	gas constant (8.31441×10^7 erg/mol K)
T	=	analysis bath temperature (*K*), from an entered or calculated value on the *P_0 and Temperature Options* window

$$T_1 = \frac{1}{(1-S)^3} - \frac{1}{(1+S)^3}$$

$$T_2 = \frac{1}{(1+S)^2} - \frac{1}{(1-S)^2}$$

$$T_3 = \frac{1}{(1-S)^9} - \frac{1}{(1+S)^9}$$

$$T_4 = \frac{1}{(1+S)^8} - \frac{1}{(1-S)^8}$$

where $$S = \frac{L - d_0}{L}$$

CHENG/YANG CORRECTION

This factor corrects for the nonlinearity of the isotherm. It adds an additional term to the equations for the different geometrics:

$$\ln\left(\frac{P}{P_0}\right) = G(L) - \left[1 - \frac{1}{\theta}\ \ln\left(\frac{1}{1-\theta}\right)\right]$$

where

G(L)	=	one of the Horvath-Kawazoe equations given above
θ	=	degree of void filling; θ is estimated by first computing the monolayer capacity (Q_m) with the Langmuir equation over the range of data points from relative pressure 0.02 to 0.2 or the maximum relative pressure included in the Horvath-Kawazoe analysis. θ is computed as the quantity adsorbed over Q_m.

INTERACTION PARAMETER

The interaction parameter (IP) results from the following calculations:

The Kirkwood-Muller dispersion coefficients

$$A_S = \frac{6mc^2 \alpha_S \alpha_A}{\frac{\alpha_S}{X_S} + \frac{\alpha_A}{X_A}}$$

$$A_A = \frac{3mc^2 a_A x_A}{2}$$

where

α_A	=	polarizability of gas molecule (cm^3)
α_S	=	polarizability of sample atoms (cm^3)
mc^2	=	kinetic energy of electron (0.8183×10^{-6} erg)
X_A	=	diamagnetic susceptibility of gas molecule (cm^3)

$$IP = (N_A A_A) + (N_S A_S)$$

N_A	=	number of gas molecules/cm^2 at monolayer from the *Horvath-Kawazoe Physical Properties* window
N_S	=	number of sample atoms/cm^2 from the *Horvath-Kawazoe Physical Properties* window

X_s	=	diamagnetic susceptibility of sample atom (cm^3)

See *Interaction Parameter Components Table on page B - 33* for recommended values.

ADDITIONAL CALCULATIONS

Based on the previous calculations, the following can be calculated:

Adjusted Pore Width (Å):

(Shell to Shell)

$$AL_i = L_i - D_S$$

Cumulative Pore Volume (cm^3/g):

$$V_{cum,i} = \frac{Q_i V_{mol}}{22414 cm^3 STP}$$

where

V_{mol} = liquid molar volume from the fluid property information

dV/dD Pore Volume (cm^3/g-Å):

$$\frac{dV}{dD_i} = \frac{V_{cum,i} - V_{cum,i-1}}{AL_i - AL_{i-1}}$$

Median Pore Width (Å):

$$V_{half} = \frac{V_{cum,n}}{2}$$

$$D_{\text{med}} = \exp\left(\ln D_l + (\ln D_g - \ln D_l)\frac{\ln V_{\text{half}} - \ln V_l}{\ln V_g - \ln V_l}\right)$$

where

D_l	=	pore width (L_i) that corresponds to V_l
D_g	=	pore width (L_i) that corresponds to V_g
$V_{cum,n}$	=	total cumulative pore volume ($V_{cum,i}$) for points designated for Horvath-Kawazoe calculations
V_g	=	cumulative pore volume ($V_{cum,i}$) for first point greater than V_{half}
V_{half}	=	50% of total cumulative pore volume
V_l	=	cumulative pore volume ($V_{cum,i}$) for first point less than V_{half}

Interaction Parameter Components Table

Gas	Bath Temperature (K)	Sample Type	Interaction Parameter Calculated Value *
Argon	87.3	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.61 5.89 3.19
Carbon Dioxide	298.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.20 9.20 5.08
	273.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.34 9.35 5.22
	194.65	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.72 9.72 5.60
Nitrogen	77.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.84 6.53 3.49
* The interaction parameter is entered in the *Horvath-Kawazoe Report Options* window in the following field: Interaction parameter: (calculated value) × 10^{-43} erg-cm^4			

The following values were used to calculate the values in the previous table.

Carbon-Graphite

D_S = 3.40
N_S = 3.845×10^{15}
X_S = 1.05×10^{-29} (Ross/Olivier)
13.5×10^{-29} (Horvath/Kawazoe, implicit)
α_s = 1.02×10^{-24}

Zeolite

D_S = 3.04
N_S = 3.75×10^{15}
X_S = 1.94×10^{-29}
α_s = 0.85×10^{-24}

Nitrogen

D_A = 3.00
N_A = 6.71×10^{14}
X_A = 3.6×10^{-29}

Argon

D_A = 2.95
N_A = 7.608×10^{14}
X_A = 3.22×10^{-29}

α_A = 1.76×10^{-24}

α_A = 1.63×10^{-24}

Carbon Dioxide

D_A = 3.23

N_A = 4.567×10^{14} (25 ºC)

5.45×10^{14} (0 ºC)

7.697×10^{14} (-78 ºC)

X_A = 5.0×10^{-29}

α_A = 2.7×10^{-24}

D_A values are from van der Waal's constant.

N_A values are from liquid densities.

x and *a* values are derived from data found in Ross and Olivier[1] .

The physical parameters referenced in Saito/Foley are:

Aluminophosphate

D_S = 2.60

N_S = 1.48×10^{15}

X_S = 1.3×10^{-29}

α_s = 2.5×10^{-24}

Aluminosilicate

D_S = 2.76

N_S = 1.31×10^{15}

X_S = 1.3×10^{-29}

α_s = 2.5×10^{-24}

[1] Ross and Olivier, J.P., “On Physical Adsorption,” J. Wiley and Sons, New York (1964)

LANGMUIR SURFACE AREA FOR CHEMISORPTION

TRANSFORM FOR CHEMISORPTION

The Langmuir isotherm is

$$\frac{Q}{Q_m} = \frac{bP}{1+bP}$$

The isotherm is transformed so that *P/Q* is plotted as a function of pressure. The transformed data are fitted with a straight line. the slope (m) and intercept (y_0) of the fit line are used in the calculations below.

SURFACE AREA

$$A_{Lang} = \frac{\bar{A}_{atom}\bar{S}N_A}{V_{mol}m} \cdot 10^{-18}\frac{m^2}{nm^2}$$

MONOLAYER CAPACITY

$$Q_{m=\frac{1}{m}}$$

LANGMUIR b VALUE

$$b = \frac{1}{y_0 Q_m}$$

DISSOCIATIVE CHEMISORPTION

The Langmuir isotherm may be derived for dissociative chemisorption.

$$\frac{Q}{Q_m} = \frac{b\sqrt{P}}{1+b\sqrt{P}}$$

The calculations are performed with the slope and intercept of a fit of $\frac{\sqrt{P}}{Q}$ as a function of $\sqrt{P}$.

LANGMUIR SURFACE AREA FOR PHYSISORPTION

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

$$L = \frac{P_{rel}}{N_{ads}}$$

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

A least-squares fit is performed on the (P_{rel}, L) designated pairs where P_{rel} is the independent variable and L 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

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

Langmuir Surface Area (m^2/g):

$$SA_{Lan} = \frac{CSA \times N_A}{\left(22414 cm^3 \ STP\right)\left(\frac{10^{18} \ nm^2}{m^2}\right)S}$$

where

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

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

$$Q_m = \frac{1}{S}$$

Langmuir b Value:

$$b = Y_{\text{int}} V_m$$

[1] Langmuir, I., J. Am. Chem. Soc. 38, 2267 (1916); J. Am. Chem. Soc. 40, 1361 (1918); Phys. Rev 8, 149 (1916).

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

$$LAN_{err} = \frac{SA_{Lan} S_{err}}{S}$$

METAL DISPERSION

$$D = 100\% \cdot 100\% \frac{Q_o \overline{S}}{V_{mol} \Sigma \frac{P_i}{W_i}}$$

where

$V_{mol} \simeq 22414 cm^3 / mol$ the molar volume of an ideal gas at standard temperature and pressure

METALLIC SURFACE AREA

The metallic surface area is the total active metal surface area available for interaction with the adsorbate.

$$A_{metal} = \frac{N_A Q_o \overline{SA}_{atom}}{V_{mol}}$$

where

$N_A \simeq 6.023 \times 10^{23}$ the number of atoms per mole

MP-METHOD

With the (t_i,Q_i) data pairs[1] , the Akima semi-spline interpolation method is used to interpolate quantity adsorbed values based on thickness values that are evenly spaced 0.2 angstrom apart starting at the first outlier point. Outliers are defined as those points that have the maximum instantaneous slope within an iteratively shrinking subset of all points. The remaining pore surface area calculation result is the slope of the line defined by two consecutive interpolated points. The slopes of each pair of consecutive points from the origin to the last point must be monotonically decreasing and non-negative. With the interpolated points set the following can be calculated:

Average pore hydraulic radius (Å):

$$R_i = \frac{t_i + t_{i-1}}{2}$$

Remaining pore surface area for the ith point (m^2/g):

$$S_i = \frac{Q_i - Q_{i-1}}{t_i - t_{i-1}} \frac{V_{mol}}{22414 \ cm^3 STP} \times 10^4$$

where

10^4	=	unit conversions
V_{mol}	=	liquid molar volume from the fluid property information

Incremental pore surface area occluded for the ith point (m^2/g):

$$S_{inc,i} = S_{i-1} - S_i$$

Cumulative pore surface area occluded for the ith point (m^2/g):

$$S_{cum_i} = S_{inc,i} + S_{inc,i-1} + \ldots + S_{inc,i}$$

dA/dR pore surface area for the ith point (m^2/g-Å):

$$\frac{dA}{dR_i} = \frac{S_{inc_i}}{t_i - t_{i-1}}$$

Incremental pore volume occluded for the ith point (cm^3/g):

[1] Mikhail, R., Brunauer, S. and Bodor, E., J. Colloid and Interface Sci. 24, 45 (1968).

$$V_{inc,\,i} = S_{inc,\,i} R_i \times 10^{-4}$$

Cumulative pore volume occluded for the ith point (cm^3/g):

$$V_{cum,\,i} = V_{inc,\,i-1} + V + \ldots + V_{inc,\,i}$$

dV/dR pore volume for the ith point (cm^3/g-Å):

$$\frac{dV}{dR_i} = \frac{V_{inc,\,i}}{t_i - t_{i-1}}$$

QUANTITY ADSORBED FOR CHEMISORPTION

A portion of the dosing volume may be at a slightly elevated temperature due to heating of the sample ports. The manifold volume is partitioned into a volume at the temperature of the manifold block and a volume at the average temperature of the ports.

$$n_a = n_d - n_f$$

$$n_d = P_{1m}C\left(P_{1m}, T_{1m}, \overline{T}_{1p}\right) - P_{2m}C\left(P_{2m}, T_{2m}, \overline{T}_{2p}\right)$$

$$C\left(P, T_m, T_p\right) = V_m\left(\frac{\alpha}{z(P, T_m)T_m} + \frac{\beta}{z\left(P, T_p\right)T_p}\right)$$

where

α and β	=	constants that determine the relative weights of the manifold and port temperatures
n_a	=	quantity of gas adsorbed
n_d	=	quantity of gas dosed
n_f	=	quantity of gas in the free space
P_{1m}	=	manifold pressure before dosing onto the sample
P_{2m}	=	manifold pressure after dosing
T_{1m}	=	manifold temperature before dosing onto the sample
T_{1p}	=	average of all port temperatures before dosing onto the sample
T_{2m}	=	manifold temperature after dosing
T_{2m}	=	average of all port temperatures after dosing
V_m	=	volume of the dosing manifold

QUANTITY ADSORBED FOR PHYSISORPTION

For the ith dose, the quantity dosed is

$$n(i)_{dosed,i} = n_{dosed,i-1} + n\left(P_1, V_m, T_1\right) - n\left(P_2, V_m, T_2\right)$$

The pressure, volume, and temperature are those of the dosing manifold before and after expanding into the sample tube.

$$n_{ads,i} = n_{dosed,i} - n_{fs,i}$$

The quantity of gas in the free space is

$$n_{fs,i} = \frac{P_{s,i}}{T_{STD}}\left(\frac{V_{fc}}{z\left(P_{s,i},\ T_{bath}\right)} + \frac{V_{fw}}{z\left(P_{s,i},\ T_{amb}\right)}\right)$$

with the real gas equation of state. Here, P_s is the sample pressure.

where

T_{amb}	=	approximate room temperature (298 K)
T_{amb}	=	analysis bath temperature (*K*)
V_{fc}	=	volume of free space, cold (cm^3 at standard temperature)
V_{fw}	=	volume of free space, warm (cm^3 at standard temperature)

The specific quantity adsorbed is

$$Q_{ads,i} = \frac{n_{ads,i}}{m}$$

where *m* is the sample mass.

FREE SPACE - MEASURED

Measured free-space volumes are calculated using the following equations:

$$V_{fw} = \frac{V_{man}}{T_{man}}\left(\frac{P_1}{P_2} - 1\right)T_{STD}$$

$$V_{fc} = V_{fw}\left(\frac{P_2}{P_3}\right)$$

FREE SPACE - CALCULATED

The calculated free space is determined by subtracting the gas capacity of the volume occupied by the sample from the measured free space of the empty tube.

$$V_{fw} = V_{wb} - V_s\left(\frac{T_{STD}}{T_{amb}}\right)$$

REAL GAS EQUATION OF STATE

All chemisorption gas accounting calculations utilize the real gas equation of state and compressibility factor data traceable to NIST.

$$n = \frac{PV}{z(P,T)T}$$

where

n	=	quantity of gas
P	=	pressure
T	=	temperature
V	=	volume
$z(P,T)$	=	compressibility factor for the gas of interest at the given pressure and temperature

Quantity of gas in cm^3 STP is given by

$$Q = n\frac{T_{STD}}{P_{STD}}$$

RELATIVE PRESSURE

If P_0 is measured in the P_0 tube, the current pressure is measured in the P_0 tube when each point is taken, and used to calculate relative pressure for that point:

$$P_{rel} = \frac{P}{Po}$$

SATURATION PRESSURE

Saturation pressure (P_0) is selected on the *P_0 and Temperature Options* window. It may be entered or measured in the P_0 tube. The analyzer uses the following methods to get P_0:

1. P_0 is measured in the P_0 tube for each isotherm point.
2. The saturation pressure is measured in the sample tube after all adsorption data points have been collected. This pressure is used as P_0 for all data points.
3. P_0 is measured for all points as with #1. After all adsorption points have been taken P_0 is measured in the sample tube. The measured P_0 points are shifted so that the P_0 measured in the P_0 tube matches the P_0 measured in the sample tube. That is, $Po(i) = Po(i) + Po_s - Po_n$ where Po_n is the P_0 measured in the P_0 tube when P_0 in the sample tube (Po_s) was measured.
4. Determine P_0 from pressure measured over the dosing source. Note that the *Adsorptive Properties* must specify dosing from Psat tube, Sample port 3, or Vapor source.
5. The saturation pressure of a gas is measured in the P_0 tube for each data point. The bath temperature is found by looking up the temperature for the measured saturation pressure in the fluid properties. P_0 of the analysis gas is found from the bath temperature as in #6. If dosing is done from the Psat tube, P_0 is determined once at the beginning of the analysis and used for all data points. Otherwise, P_0 is measured for each data point.
6. P_0 is found by looking up the saturation pressure for the entered bath temperature in the fluid property information.

   Lookup of saturation pressure in the fluid properties is done by interpolating the Psat data using the Clausius-Clapeyron equation, $\ln\left(P\right) = \frac{a}{T} + b$. The constants *a* and *b* are determined from the pressures and temperatures that bound the bath temperature. Temperature lookup is done by solving for *T*, $T = \frac{a}{\ln(P) - b}$, where *a* and *b* are determined from the pressures that bound the given saturation pressure.

7. If entered, P_0 = user-entered value.

SPC REPORT VARIABLES

REGRESSION CHART VARIABLES

The line of best fit for the Regression Chart is calculated by the usual least squares method. [1] If there is only a single point or all *N* points have the same x-value, there can be no line of best fit in the standard form.

$$\bar{x} = \frac{\Sigma x_i}{N}$$

$$\bar{y} = \frac{\Sigma y_i}{N}$$

$$\text{Slope} = \frac{\Sigma\left(x_i - \bar{x}\right)\left(y_i - \bar{y}\right)}{\Sigma\left(x_i - \bar{x}\right)^2}$$

$$\text{Intercept} = \bar{y} - \text{Slope} \cdot \bar{x}$$

The coefficient of correlation for this line is also calculated in the usual way. [2]

$$\sigma_x = \sqrt{\frac{\Sigma\left(x_i - \bar{x}\right)^2}{N}}$$

$$\sigma_y = \sqrt{\frac{\Sigma\left(y_i - \bar{y}\right)^2}{N}}$$

$$\text{Cov}(x, y) = \frac{\Sigma\left(x_i - \bar{x}\right)\left(y_i - \bar{y}\right)}{N}$$

$$\text{Correlation Coeff} = \frac{\text{Cov}(x, y)}{\sigma_x \sigma_y}$$

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[1] BASIC Scientific Subroutines Vol II, by F.R. Ruckdeschel, Copyright 1981 BYTE Publications/McGraw Hill, p. 16.

[2] Mathematical Handbook for Scientists and Engineers, G.A. Korn and T.M. Korn, McGraw Hill, Sec. 18.4. (1968)

CONTROL CHART VARIABLES

$$\text{Mean} = \frac{\Sigma y_i}{N}$$

$$\text{Standard Deviation} = \sqrt{\frac{\Sigma\left(y_i - \text{Mean}\right)^2}{N-1}}$$

$$\text{C. V.} = \frac{\text{StdDev}}{\text{Mean}}$$

$$+ n\sigma = \text{Mean} + n \cdot \text{Standard Deviation}$$

$$- n\sigma = \text{Mean} - n \cdot \text{Standard Deviation}$$

SUMMARY REPORT

The following calculations and the results of previous calculations (as noted) are used to generate the summary report:

a. Single-point Surface Area (m^2/g)

$$S_{1PT} = \frac{\left[Q\left(1-P\right)\right] \times CSA\left(6.023 \times 10^{23}\right)}{22414cm^3 \times STP\left(\frac{10^{18}nm^2}{m^2}\right)}$$

where

P	=	pressure closest to 0.3 of the relative pressure points designated for surface area calculations.
Q	=	quantity adsorbed corresponding to P

b. Multi-point Surface Area. See *BET Surface Area on page B - 2*

c. Langmuir Surface Area. See *Langmuir Surface Area for Chemisorption on page B - 35* and *Langmuir Surface Area for Physisorption on page B - 36*

d. t-Plot Micropore Surface Area. See *t-Plot on page B - 52*

e. t-Plot External Surface Area. . See *t-Plot on page B - 52*

f. BJH Cumulative Adsorption

g. BJH Cumulative Desorption

h. Adsorption Total Pore Volume

i. Desorption Total Pore Volume

j. t-Plot Micropore Pore Volume.. See *t-Plot on page B - 52*

k. Freundlich. See *Freundlich Isotherm on page B - 26*

l. Temkin. See *Temkin Isotherm on page B - 48*

m. Alpha-S. See *Alpha-S Method on page B - 1Alpha-S Method on page B - 1*

n. DFT Pore Size and DFT Surface Energy. See *DFT (Density Functional Theory) on page B - 12*

o. Nanoparticle Size

$$d = \frac{6 \times 10^4}{A\rho}$$

where

ρ	=	sample density
A	=	BET surface area
d	=	side length (for cubic particles or diameter (for spherical particles)

p. Dubinin-Astakhov Micropore Surface Area. See ***Dubinin-Astakhov on page B - 17***

q. Dubinin-Astakhov Micropore Volume. See ***Dubinin-Astakhov on page B - 17***

r. Dubinin-Radushkevich Micropore Surface Area. See ***Dubinin-Radushkevich on page B - 21***

s. Dubinin-Radushkevich Monolayer Capacity. See ***Dubinin-Radushkevich on page B - 21***

t. MP-Method Cumulative Surface Area of Pores

S_{total}l = S_{cum}'i', See ***MP-Method on page B - 38*** for the last collected data point used in the MP-method Calculations, and the range of hydraulic pore radii over which the cumulative surface area was computed.

u. MP-Method Cumulative Pore Volume of Pores

$V_{total} = V_{cum,i'}$

See ***MP-Method on page B - 38*** for the last collected data point used in the MP-method calculations, and the range of hydraulic pore radii over which the cumulative pore volume was computed.

v. Average Pore Hydraulic Radius (Å)

$$\bar{r} = \frac{V_{total}}{S_{total}} \times 10^4$$

w. Horvath-Kawazoe. See ***Horvath-Kawazoe on page B - 27***

TEMKIN ISOTHERM

The Temkin isotherm has the form

$$\frac{Q}{Q_m} = \frac{RT}{q_0 \alpha} ln\left(A_o P\right)$$

where

A_0	=	adjustable constant
alpha		adjustable constant
P	=	equilibrium pressure measured by gauge at temp T_{amb}
q_0	=	the differential heat of adsorption at zero surface coverage
Q	=	quantity of gas adsorbed
Q_m	=	quantity of gas in a monolayer
R	=	molar gas constant $8.31441 \times 10^{-3} \frac{kJ}{molK}$
T	=	bath temperature

In terms of quantity adsorbed

$$Q = \frac{RTQ_s}{q_0 \alpha}\left[\ln A_0 + \ln\left(\frac{P}{P_0}\right)\right]$$

Thus, the plot of the natural log of absolute pressure vs. quantity adsorbed yields a straight line with slope $\frac{RTQ_m}{q_0}$ and intercept $\ln A_0 \frac{RTQ_m}{q_0 a}$.

THERMAL TRANSPIRATION CORRECTION

During data reduction, thermal transpiration correction is applied to the data if the user selected *Apply thermal transpiration correction* from the *Report Options* window. Starting with the first collected pressure, the following calculations are performed until the pressure ratio (PC/P) is greater than or equal to 0.99.

$$Y = \left(\frac{P \times SD \times MD^2}{2.33 \times T}\right)10^3$$

$$\mu = \frac{(1+G)Y}{(1+H)Y}$$

$$F = \frac{1}{\alpha Y^2 + \beta Y + \mu}$$

$$P = \left(1 - F\left(1 - \sqrt{\frac{T_{bath}}{T_{amb}}}\right)\right)$$

where

α	=	Weber's coefficient, 0.033
β	=	Weber's coefficient, 0.245
$F, Y\ \mu$	=	intermediate values for subsequent calculations
G	=	Weber's coefficient, 2.5
H	=	Weber's coefficient, 2
MD	=	thermal transpiration hard sphere diameter of gas (Å), from the *Adsorptive Properties* window
P	=	equilibrated collected pressure measured by gauge at temp T_{amb}
SD	=	inside diameter of sample tube (mm), from the *Report Options* window
T	=	average temperature $\frac{T_{amb}+T}{2}$
T_{amb}	=	room temperature (298 K)
T_{bath}	=	analysis bath temperature (K), from the P_0 *and Temperature Options* window

THICKNESS CURVE

For each point designated, the following parameters are used in thickness curve calculations:

C_1	=	parameter #1
C_2	=	parameter #2
C_3	=	parameter #3
$P_{rel,\ i}$	=	relative pressure for the ith point (mmHg)
t_i	=	thickness for ith point

REFERENCE

Interpolated from table.

KRUK-JARONIEC-SAYARI

$$t = \left(\frac{C_1}{C_2 = \log\left(P_{rel,\ i}\right)} \right)^{c3}$$

HALSEY

$$t_i = C_1 \left[\frac{C_2}{\ln\left(P_{rel,\ i}\right)} \right]^{C_3}$$

Halsey[1)]

HARKINS AND JURA

$$t_i = \left[\frac{C_1}{C_2 - \log\left(P_{rel,\ i}\right)} \right]^{C_3}$$

Harkins and Jura[2)]

1) Halsey, G., J. Chem. Phys. 16, 931-937 (1948).
2) Harkins, W.D. and Jura, G., J. Chem. Phys. 11, 431 (1943).

BROEKOFF-DE BOER

$$\log\left(P_{rel,i}\right) = \frac{C_1}{t^2{}_{,i}} + C_2 \exp\left(c_3 t_i\right)$$

CARBON BLACK STSA

$$t_i = C_1\left(P_{rel,i}\right)^2 + C_2\left(P_{rel,i}\right) + C_3$$

T-PLOT

A least-squares analysis fit is performed on the (t_i, $N_{ads,i}$) data pairs where t_i is the independent variable and $N_{ads,i}$ is the dependent variable. Only the values of t_i between t_{min} and t_{max}, the minimum and maximum thickness, are used. The following are calculated:

a. Slope (S cm^3/g-Å STP)
b. Y-intercept (Y_{int} cm^3/g STP)
c. Error of the slope (S_{err} cm^3/g-Å STP)
d. Error of the Y-intercept (YI_{err} cm^3/g STP)
e. Correlation coefficient

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

External Surface Area (m^2/g):

$$\frac{SV_{mol}}{F \times 22414 cm^3 STP} \times 10^4$$

where

10^4	=	unit conversions
F	=	surface area correction factor, user-entered on the *t*-Plot Report Options screen
V_{mol}	=	liquid molar volume, from the fluid property information

Micropore Surface Area (m^2/g):

$$SA_{\mu\rho} = SA_{total} + SA_{ext}$$

where SA_{total} is the BET surface area if the user enabled the BET report exclusively, or Langmuir surface area if the user enabled the Langmuir report exclusively. If neither report has been selected, SA_{total} is the BET surface area value calculated using a set of default parameters.

Micropore Volume (cm^3 liquid/g):

$$\frac{Y_{\text{int}} V_{mol}}{22414 cm^3 STP}$$

WEIGHTED METAL PARAMETERS

The stoichiometry factor, atomic weight, and density used in calculations are averages weighted by the number of moles of each active metal. For example, the average stoichiometry factor is

$$\overline{S} = \frac{\sum_i n_i S_i}{\sum_i n_i}$$

where

n_i = number of moles or metal

$$n_i = \frac{\alpha\beta X}{XW_m + YW_0}$$

where

a	=	fraction of sample mass
β	=	fraction reduced
X	=	number of metal atoms in the oxide
Y	=	number of oxygen atoms in the oxide
W_m	=	atomic weight of metal
W_o	=	atomic weight of Oxygen

Average density and atomic cross-sectional area are calculated similarly.

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C DFT MODELS

Theories are developed by scientists in an attempt to explain a class of observed behavior. In the experimental physical sciences, theories are often expressed in terms of a model that can be visualized and described mathematically. Early models of physical adsorption were quite simple, both conceptually and mathematically, for very practical reasons — hand computations were required. Today we can explore complex models that describe adsorption systems on the atomic scale of size and sub-picosecond time frame. This is not because scientists are smarter, but because of available tools. The DFT models are created by classical approaches to adsorption as well as models based on modern statistical thermodynamics.

MODELS BASED ON STATISTICAL THERMODYNAMICS

Included in this group are methods that model the adsorption system in terms of forces acting between individual molecules.

THEORETICAL BACKGROUND

Traditional adsorption theories attempt to describe experimental adsorption isotherms with an isotherm equation containing a small number of parameters. At a minimum, these parameters include the extent of the surface, such as the monolayer capacity (Q_m), and the molar intensity of the gas-surface interaction, such as the Langmuir “K” constant or the BET “C” constant. In some equations, additional parameters take into account the lateral interaction of adsorbed molecules with each other. Other theories, such as the Dubinin-Astakhov approach, also include parameters for the effect of adsorbent porosity.

Instead of this classical kinetic or phenomenological approach, we can use a molecular-based statistical thermodynamic theory that allows us to relate the adsorption isotherm to the microscopic properties of the system: the fluid-fluid and fluid-solid interaction energy parameters, the pore size, the pore geometry, and the temperature.

The following example is given so that you may understand how such a theory is constructed:

> A clean sample of a solid material containing slit-shaped pores of a single width is placed in an evacuated space. It is kept at a fixed temperature as a known quantity of pure argon gas is admitted into the space surrounding the sample. The pressure within the space is recorded over time. In this situation, the pressure falls rapidly from its initial value and gradually approaches a steady reading, called the equilibrium pressure. The amount adsorbed corresponds to the quantity of gas effectively removed from the gas phase by the solid surface. A graph that plots amount adsorbed versus equilibrium pressure is called an adsorption isotherm.

Under such conditions, the argon atoms that randomly enter the pore space feel the presence of the solid surface as the action of an external attractive force (the dispersion forces or Van der Waal's forces) and spend more time near the surface. As a result, the space near the surface acquires a greater average density of argon atoms than regions farther removed.

If the equilibrium distribution of the gas atoms near the surface could be described as a function of pressure and the molecular properties of the components of the system, then a model could be constructed for the adsorption isotherm for the system. Modern physical chemistry provides several ways to calculate this distribution. All these methods are based on the fundamental thermodynamic law that such a system adopts a configuration of minimum free energy at equilibrium. Also needed is a description of the pairwise interaction energy between atoms, U(s), commonly given by a Lennard-Jones potential:

$$U\left(s\right) = 4\epsilon\left(\frac{\sigma}{s}\right)^{12} - \left(\frac{\sigma}{s}\right)^{6}$$

where

ε = a characteristic energy of the adsorptive,
σ = the diameter of the adsorptive molecule, and
s = the separation distance.

MOLECULAR SIMULATION METHODS

Two simulation techniques are commonly used to determine the distribution of gas molecules in a system in equilibrium: the molecular dynamics method and the Monte Carlo method. Both of these are used as reference methods because their results are considered exact.

MOLECULAR DYNAMICS METHOD

In the molecular dynamics method, the position and velocity of individual gas particles are calculated over time at very short intervals. This method takes into account both the forces acting between the gas particles themselves and those acting between the gas particles and the atoms of the simulated surface. As the simulated particles collide with each other and with the surface, the average concentration of particles in the space near the surface is calculated; this calculation yields the amount of gas adsorbed.

This method can be thought of as a way to determine the chronological record of the movement of each particle in the system using time steps of 10-14 seconds. Although the mathematics are simple, the number of calculations required for a system of even a few hundred particles is astronomical and challenges even the fastest computers.

MONTE CARLO METHOD

In the Monte Carlo method, determination of the system equilibrium distribution begins with an assumption (which may be only approximate) about the initial configuration of particles in the system. The system is "equilibrated" through a process of randomly selecting one particle and conditionally moving it a random distance in a random direction.

If the move results in a configuration of *lower total energy*, then the move is completed and another particle is randomly selected to be moved.

If the move results in a configuration of *higher energy*, a probability for that event is calculated, and a random number between zero and one is generated. If the generated number is smaller than the probability of the event, then the move is accepted; otherwise, another particle is selected and the process is repeated. This process continues until the average total energy of the system no longer decreases; at this point, average configuration data are accumulated to yield the mean density distribution of particles in the system.

Monte Carlo simulations require considerably less computation time than molecular dynamic simulations and can yield the same results; however, neither method provides a really practical way to calculate complete isotherms.

DENSITY FUNCTIONAL FORMULATION

Density functional theory offers a practical alternative to both molecular dynamic and Monte Carlo simulations. When compared to reference methods based on molecular simulation, this theory provides an accurate method of describing inhomogeneous systems yet requires fewer calculations. Because the density functional theory provides accuracy and a reduced number of calculations, it is the basis embodied in the DFT models.

The system being modeled consists of a single pore represented by two parallel walls separated by a distance H. The pore is open and immersed in a single component fluid (adsorptive) at a fixed temperature and pressure. Under such conditions, the fluid responds to the walls and reaches an equilibrium distribution. In this condition (by the definition of equilibrium), the chemical potential at every point equals the chemical potential of the bulk fluid. The bulk fluid is a homogenous system of constant density; its chemical potential[1)] is determined by the pressure of the system using well-known equations. The fluid near the walls is not of constant density; its chemical potential is composed of several position-dependent contributions that must total at every point to the same value as the chemical potential of the bulk fluid.

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1) Chemical potential may be thought of as the energy change felt by a probe particle when it is inserted into the system from a reference point outside the system. It can also be defined as the partial derivative of the grand potential energy with respect to density (or concentration).

As noted previously, at equilibrium, the whole system has a minimum (Helmholtz) free energy, known thermodynamically as the grand potential energy (GPE). Density functional theory describes the thermodynamic grand potential as a functional of the single-particle density distribution; therefore, calculating the density profile that minimizes the GPE yields the equilibrium density profile. The calculation method requires the solution of a system of complex integral equations that are implicit functions of the density vector. Since analytic solutions are not possible, the problem must be solved using iterative numerical methods. Although calculation using these methods still requires supercomputing speed, the calculation of many isotherm pressure points for a wide range of pore sizes is a feasible task. The complete details of the theory and the mathematics can be found in the papers listed under ***DFT Model References on page C - 17***.

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

Figure 1 shows the density profile for argon at a carbon surface as calculated by density functional theory for a temperature of 87.3 K and a relative pressure of about 0.5.

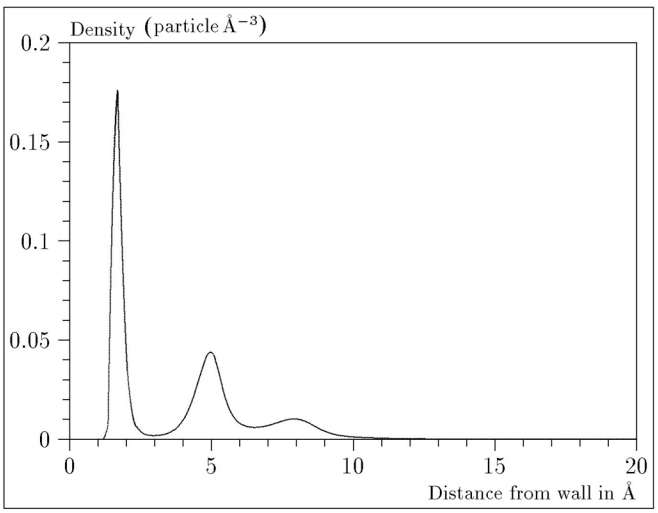

Figure 1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

This figure represents a cross-section of the region near the surface. Note the layerwise distribution of adsorbate; the first monolayer is sharply defined and a third layer can be distinguished. The area under the profile curve represents the amount adsorbed per unit area at this pressure. The positions of the maxima are separated by a distance determined by the size of the adsorptive atom.

Given the density profile, the amount adsorbed at the stated pressure can be easily calculated as the integral over the profile. Repeating this calculation over a range of pressures yields the adsorption isotherm for the model. If the value of H is very large, the isotherm obtained corresponds to that of an external, or *free*, surface. If H is smaller, a range of pressures is reached where two minima exist for the grand potential, showing the presence of two metastable phases having different density distributions but the same chemical potential. The phase with the lower GPE is the stable one. As the pressure is increased, a point is reached where the other phase becomes the stable one. This phase transition reflects condensation of adsorbate in the pore; the pressure at which it occurs is called the *critical pore-filling pressure*. This pressure is analogous to the condensation pressure predicted by the Kelvin equation in the classical model of pore filling.

Figure 2 shows how the profiles change with pressure for a model pore with H = 40 angstroms. The inset shows the density profiles for the corresponding points of the isotherm.

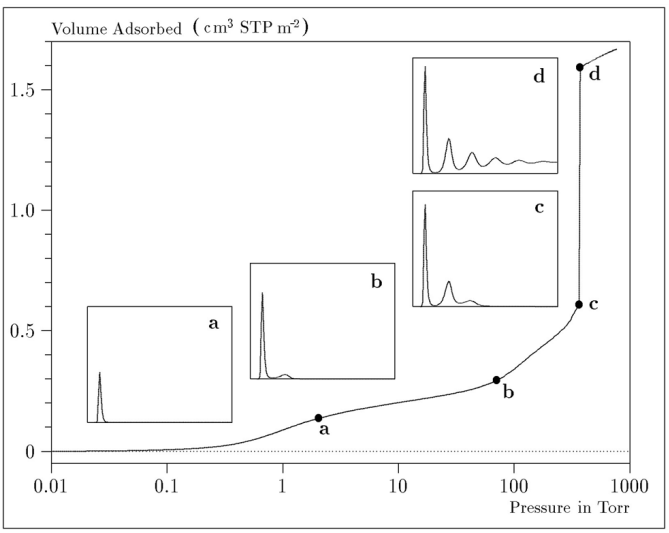

Figure 2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

The profiles show the density distribution from one wall to the center of the slit; the other half of the distribution is a mirror image of the profile shown.

As the pressure is first increased from zero, almost all the adsorbed atoms occupy a position close to the surface.

- Inset ***a*** shows the profile corresponding to point a on the isotherm where the surface is about half covered.
- At point ***b***, the first layer is so full that it is more favorable for atoms to start a new layer.
- At point ***c***, a third layer is forming. Point ***c***, for this size slit, is the critical pore-filling pressure. In inset ***c***, the profile shows the density decreasing to near zero (actually the bulk gas density) at 4 or 5 molecular diameters from the surface.

- Inset ***d*** shows the profile converging on a density similar to that of bulk liquid argon in the center of the pore, indicating a phase transition.

Note that the adsorption isotherms for pores larger than the one shown in the previous graph is identical up to point ***c***. The lower branch of the isotherm simply continues to a higher pressure for larger pores. This trend is illustrated in the Figure 3, where isotherms for some larger size pores are shown. It is clear that pore size is uniquely characterized by a corresponding critical pore-filling pressure. At large pore sizes, density functional theory produces results for the critical filling pressures that are in good agreement with those produced by the Kelvin equation.

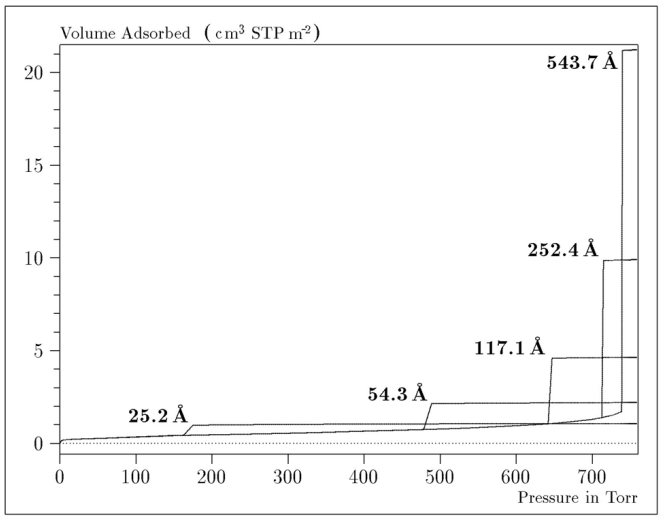

Figure 3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

Figure 4 shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.

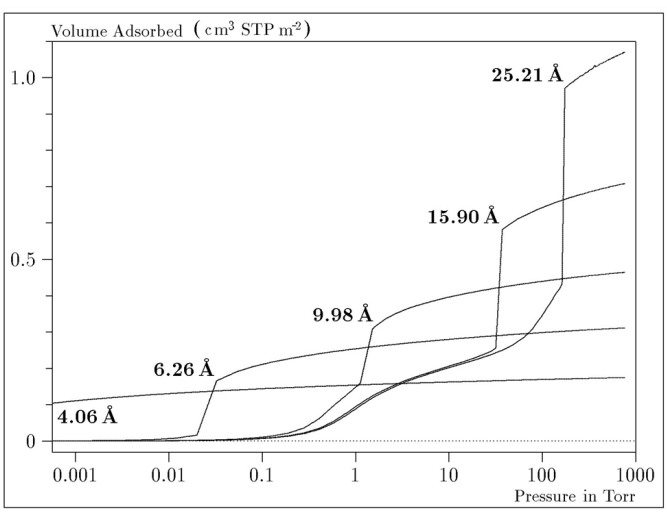

Figure 4. Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

Pores of 4 Å width, barely larger than the argon atom (3.38 Å), fill at pressures below 1 millitorr. Pores below 15 Å fill before a monolayer is completed on the surface of the larger pores. In the micropore size range, the pore volume fills more gradually with pressure and the total shape of the isotherm is important in characterizing the pore size.

Models Included

Non-Local Density Functional Theory with Density-Independent Weights

N2 - DFT Model
AR - DFT Model

Geometry:	Slit
Substrate:	Carbon (graphite)
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Using the methods of non-local density functional theory, two sets of isotherms have been calculated to serve as kernel functions for the characterization of porous solids from adsorption data. The model isotherms are stored in binary format files. These models assume a *slit-like pore geometry*. The pore size range from 4.0 to 4000 Å is covered in 91 classes in a geometric progression. The class intervals are rounded to the nearest 0.02 molecular diameters. A model for the free or external surface is included to account for unfilled pores. Each of the 92 model isotherms has been calculated at 181 pressure points from near 1×10^{-6} to near 1.00 relative pressure.

These models are identical to those supplied with the original DOS version of DFT software. Some slight difference from the DOS results may be noted when they are applied to the same data due to improvements in the deconvolution algorithm and better regularization of the current software.

Non-Local Density Functional Theory with Density-Dependent Weights

N2 - Modified Density Functional

Geometry:	Free surface
Substrate:	Surface energy
Method:	Nitrogen at 77K

Using the modified Tarazona prescription described by Olivier (see *DFT Model References on page C - 17* [items 3 and 4]), model isotherms were calculated for a wide range of adsorptive energies to a relative pressure of 0.6. The model makes no provision for pore filling in the micropore region. If the sample solid contains small mesopores, the isotherm data should be truncated (using the *Select Data Points* window) to a suitably low relative pressure to avoid trying to fit this region; mesopore filling reports as a large area of low energy in the calculated distribution of adsorptive potential.

The surface energy is reported in terms of the effective Lennard-Jones interaction parameter, ie, for the adsorptive / adsorbent pair divided by Boltzmann constant. The units are therefore Kelvin.

N2 - Cylindrical Pores - Oxide Surface
AR - Cylindrical Pores - Oxide Surface

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using a combination of statistical mechanical calculations and experimental observations for macroporous silicas and MCM-41 mesoporous silicas as well as zeolites. The pore-filling pressures were determined as a function of the pore size from adsorption isotherms on MCM-41 materials characterized by X-ray and other techniques. The variation of the pore fluid density with pressure and pore size has been accounted for by density functional theory calculations. The N2 model reports pore sizes ranging from 3.8 to 387 Å and the AR model from 3.8 to over 500 angstroms.

References: M. Jaroniec, M. Kruk, J.P. Olivier, and S. Koch, "A New Method for the Accurate Pore Size Analysis of MCM-41 and Other Silica-Based Mesoporous Materials," Proceedings of COPS-V, Heidelberg, Germany (1999).

N2 – Cylindrical Pores – Pillared Clay Surface (Montmorillionite)

Geometry: Cylinder
Substrate: Crystalline Silicate
Category: Porosity
Method: Nitrogen at 77K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFT) calculations and experimental isotherms for reference samples of montmorillionite.The construction method for the hybrid models was analogous to that described in the first reference below (Jaroniec et al,1999). The additional references add additional theoretical details as well as examples of the application of the model to pillared clay catalysts.This model reports pore widths from 3.8 to 387 angstroms.

References: Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V* , Unger, et al, Eds, Elsevier, Amsterdam, 2000.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of a Pillared Interlayered Clay (PILC) from a Hybrid Density Functional Theory (DFT) Method," *The Journal of Physical Chemistry B*; 2001, 105(3),

623-629.

M. L. Occelli, J. P. Olivier, J. A. Perdigon-Melon, and A. Auroux, "Surface Area, Pore Volume Distribution, and Acidity in Mesoporous Expanded Clay Catalysts from Hybrid Density Functional Theory (DFT) and Adsorption Microcalorimetry Methods," *Langmuir* 2002, 18, 9816-9823.9b.

James P. Olivier, "The Importance of Surface Heterogeneity in Developing Characterization Methods." *6th International Symposium on the Characterization of Porous Solids*, Studies in Surface Science and Catalysis 144, Elsevier, 2002.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of Pillared Rectorite Catalysts from a Hybrid Density Functional Theory Method," *Microporous and Mesoporous Materials* 2003, 57, 291-296.

C02 - DFT Model

Geometry: Slit
Substrate: Carbon
Category: Porosity
Method: Carbon dioxide at 273 K

Model isotherms were calculated using the non-local prescription of Tarazona, employing molecular parameters derived from the known bulk properties of carbon dioxide.

AR - Modified Density Functional Model

Geometry: Free surface
Substrate: Any
Category: Surface energy
Method: Argon at 87K

This model was produced in the same manner as the N2 Modified Density Functional model listed earlier, except applicable to argon adsorbed at 87.3 K.

N2 - Tarazona NLDFT, Esf = 30.0K

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a cylindrical pore geometry. The wall potential used is k = 30 K, typical for a silica or alumina surface.

This model file is particularly useful for sizing zeolites or zeolite containing materials that have substantial micropore volume. The reported pore size range is 3.8 to 387 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Slit Pores by NLDFT
Ar - Carbon Slit Pores by NLDFT

Geometry: Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a slit-like pore geometry. These models are slightly different from N2-DFT and Ar-DFT models that were calculated using NLDFT with density independent weighting functions.

The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Finite Pores, As=6, 2D-NLDFT
Ar - Carbon Finite Pores, As=6, 2D-NLDFT

Geometry: Finite Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions assuming 2D model of finite slit pores having a diameter-to-width aspect ratio of 6.

This model is particularly useful for microporous carbon materials. The reported pore size range is from 3.5 to 250 angstroms

References: Jacek Jagiello and James P. Olivier. “A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure

analysis.," The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N2 - Carbon Finite Pores, As=12, 2D-NLDFT
Ar - Carbon Finite Pores, As=12, 2D-NLDFT

Geometry: Finite Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the same methods and assumptions that were used in the model above except in this model, the aspect ratio is equal to 12.

These two finite pore models may be used as a research tool in conjunction with independent analytical techniques such as high-resolution transmission electron microscopy (HRTEM) and / or X-ray diffraction (XRD) to obtain comprehensive information about the structure of studied carbon material.

References: Jacek Jagiello and James P. Olivier. "A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.," The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N2 - Carbon Cylinder, single-wall nanotube by NLDFT
Ar - Argon Cylinder, single-wall nanotube by NLDFT

Geometry: Cylinder
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the graphitic surface of an infinitely long cylinder.

This model is particularly useful for characterizing carbon single-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Cylinder, multi-wall nanotube by NLDFT
Ar - Argon Cylinder, multi-wall nanotube by NLDFT

Geometry: Cylinder
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and multiple concentric graphitic surfaces of infinitely long cylinders.

This model is particularly useful for characterizing carbon multi-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987)

Ar - Zeolites H-Form by NLDFT

Geometry: Cylinder
Substrate: Zeolite
Category: Porosity
Method: Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is particularly useful for characterizing oxides and H+ and (NH4)+ exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

Ar - Zeolites Me-Form by NLDFT

Geometry: Cylinder
Substrate: Zeolite
Category: Porosity
Method: Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is similar to the model above, but it more appropriate is for characterizing alkali metal exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

MODELS BASED ON CLASSICAL THEORIES

Both surface energy distribution and pore size distribution may be evaluated using classical approaches to model kernel functions for use with equation (1) of the DFT Theory in *DFT (Density Functional Theory) on page B - 12*. Be aware that the deconvolution method only provides a fitting mechanism; it does not overcome any inherent shortcomings in the underlying theory.

SURFACE ENERGY

The use of classical theories to extract adsorptive potential distribution is mostly of historical interest. At a minimum, the equation must contain a parameter dependent on adsorption energy and another dependent on monolayer capacity or surface area. This is sufficient to permit the calculation of the set of model isotherms that is used to create a library model. The Langmuir equation has been used in the past, as have the Hill-de Boer equation and the Fowler-Guggenheim equation. All of these suffer from the fact that they only describe monolayer adsorption, whereas the data may include contributions from multilayer formation.

PORE SIZE

It is well established that the pore space of a mesoporous solid fills with condensed adsorbate at pressures somewhat below the prevailing saturated vapor pressure of the adsorptive. When combined with a correlating function that relates pore size with a critical condensation pressure, this knowledge can be used to characterize the mesopore size distribution of the adsorbent. The correlating function most commonly used is the Kelvin equation. Refinements make allowance for the reduction of the physical pore size by the thickness of the adsorbed film existing at the critical condensation pressure. Still further refinements adjust the film thickness for the curvature of the pore wall.

The commonly used practical methods of extracting mesopore distribution from isotherm data using Kelvin-based theories, such as the BJH method, were for the most part developed decades ago and were designed for hand computation using relatively few experimental points. In general, these methods visualize the incremental decomposition of an experimental isotherm, starting at the highest relative pressure or pore size. At each step, the quantity of adsorptive involved is divided between pore emptying and film thinning processes and exactly is accounted for. This computational algorithm frequently leads to inconsistencies when carried to small mesopore sizes. If the thickness curve used is too steep, it finally will predict a larger increment of adsorptive for a given pressure increment than is actually observed; since a negative pore volume is non-physical, the algorithm must stop. Conversely, if the thickness curve used underestimates film thinning, accumulated error results in the calculation of an overly large volume of (possibly nonexistent) small pores.

The use of equation (1) represents an improvement over the traditional algorithm. Kernel functions corresponding to various classical Kelvin-based methods have been calculated for differing geometries and included in the list of models.

MODELS INCLUDED

Kelvin Equation with Halsey Thickness Curve

N2 - Halsey Thickness Curve

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

The kernel function is calculated using the Halsey equation with standard parameters:

$$t = 3.54\left(\frac{-5.00}{\ln(P/P_0)}\right)^{1/3}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887 g cm^{-3}

N2 - Halsey Thickness Curve

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

Reference: G. Halsey, J. Chem. Phys 16, 931 (1948).

Kelvin Equation with Harkins and Jura Thickness Curve

N2 - Harkins and Jura Thickness Curve

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

The kernel function is calculated using the Harkins and Jura equation with standard parameters:

$$t = \left(\frac{13.99}{0.034 - \log(P / P_0)} \right)^{1/2}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887 g cm^{-3}

N2 - Harkins and Jura Thickness Curve

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

References: W. D. Harkins and G. Jura, J.A.C.S. 66, 1366 (1944).
J. H. DeBoer et al., J. Colloid and Interface Sci. 21, 405 (1966).

Kelvin Equation with Broekhoff-de Boer Thickness Curve

N2 - Broekhoff-de Boer Model

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

$$\log\left(p / p^0\right) = \frac{-16.11}{t^2} + 0.1682^{-0.1137t}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887g cm^{-3}

N2 - Broekhoff-de Boer Model

Geometry: Cylinder

Substrate: Average

Category: Porosity

Method: Nitrogen 77 K

The calculation is similar to the above except that cylindrical geometry is assumed, and the film thickness depends on pore size (see reference).

References: Specifically, equations 20 and 21 in: J.C.P. Broekhoff and J.H. de Boer, "The Surface Area in Intermediate Pores," Proceedings of the International Symposium on Surface Area Determination, D.H. Everett, R.H. Ottwill, eds., U.K. (1969).

DFT MODEL REFERENCES

The papers listed below provide additional information on DFT models:

1. "Determination of Pore Size Distribution from Density Functional Theoretic Models of Adsorption and Condensation within Porous Solids," J.P. Olivier and W.B. Conklin, Micromeritics Instrument Corp; presented at the International Symposium on the Effects of Surface Heterogeneity in Adsorption and Catalysts on Solids, Kazimierz Dolny, Poland (July 1992).
2. "Classification of Adsorption Behavior: Simple Fluids in Pores of Slit-shaped Geometry," Perla B. Balbuena and Keith E. Gubbins, *Fluid Phase Equilibria*, 76, 21-35, Elsevier Science Publishers, B.V., Amsterdam (1992).
3. "Modeling Physical Adsorption on Porous and Nonporous solids Using Density Functional Theory," J.P. Olivier, *Journal of Porous Materials*, 3, 9-17 (1995).
4. "The Determination of Surface Energetic Heterogeneity Using Model Isotherms Calculated by Density Functional Theory," J.P. Olivier; presented at the Fifth International Conference on the Fundamentals of Adsorption, Pacific Grove, CA (1995).

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D ERROR MESSAGES

If the *Action* response indicates to contact a Micromeritics service representative, record the error message, then make backup copies of any files involved in the operation.

2401 | FATAL ERROR.

Cause: An internal processing and / or hardware error has occurred during communication with the analyzer.
Action: Contact your Micromeritics service representative.

2430 | Error accessing file [*n*], error code = [*n*].

Cause A: Media may be damaged.
Action A: Clean the media drive. If this does not eliminate the problem, attempt operation using a backup copy of the file.
Cause B: Hard disk may be damaged.
Cause B: Contact your Micromeritics service representative.
Cause C: A software error occurred when the file was accessed.
Cause C: Contact your Micromeritics service representative.
Cause D: The file name specified contains one or more invalid characters.
Cause D: Enter a valid file name. Do not use characters such as * or ?. Refer to the operating system manual.

2431 | Error writing file [*n*], error code = [*n*].

Cause : Insufficient hard disk to perform the operation.
Action : Copy files not used regularly from the hard disk external media. Delete them from the hard disk, and then try the operation again.

2432 | Invalid response from MMI 'FILE_READ' request.

Cause: An internal processing and/or hardware error has occurred.
Action: Contact a Micromeritics service representative if this error message continues.

2433 | New entries have been found in this directory. Refresh the directory information?

Cause: Several analyzer files (sample information, analysis conditions, adsorptive properties, or report options) have been added to this directory by some function other than the analyzer program.
Action: Click **Yes** to update the directory information with data from each new file. This operation may take a minute.

Click **No** to locate the file manually. This option may be feasible if a large number of files have been copied into the directory and the file name is known.

2434 | File [*n*] — Subset [*n*] wrote wrong [*n*] of data, expected [*n*] bytes.

Cause: An internal processing and/or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2436 | Path specification [*n*] is invalid.

Cause: An invalid path name and / or extension was entered.
Action: Type a valid path name (including the proper extension), then press **Enter**.

2437 | Overlay file [*n*] does not exist.

Cause: The entered file specification does not exist.
Action: Enter an existing file specification, or select a file name from the list box.

2439 | Could not register file.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2440 | Subset not found.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2441 | Seek within file failed.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2442 | Bad header in subset file.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2443 | Subset owner denied access.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2444 | Not a valid file format.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2445 | Subset wrote the wrong amount of data.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2446 | Error reading data.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2447 | Error writing data.

Cause: An unexpected error occurred when you tried to access a data file.
Action: Contact your Micromeritics service representative.

2448 | Default parameter file directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.
Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2449 | This field does not contain a valid file specification.

Cause: An invalid file name was entered.
Action: See the description of file naming conventions in a Windows manual, then re-enter the name.

2450 | Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?

Cause: An automatic analysis (an analysis in which sample files are created using the defaults) was processing while editing the defaults.
Action: Finish the edit session of the defaults, close the window, then restart the automatic analysis.

2451 | Deleted entries have been found in this directory. Refreshing the directory information.

Cause: Informational message only indicating the system is looking for directory entries that cannot be found.
Action: Wait a few moments for the system to finish refreshing, then retry the operation again.

2452 | Attempt to write MICATTR.DIR in read only mode. [*n*]

Cause: The *Read-Only* attribute is turned on in the application's MICATTR.DIR file (this file exists in each folder containing sample or parameter files).
Action: Use Windows Explorer to access the folder containing the MICATTR.DIR file, then disable the *Read-Only* option.

2453 | Attempt to append MICATTR.DIR in read only mode. [file name]

Cause: The *Read-Only* attribute is turned on in the application's MICATTR.DIR file (this file exists in each folder containing sample or parameter files).
Action: Use Windows Explorer to access the folder containing the MICATTR.DIR file, then disable the *Read-Only* option.

2454 | Too many selections for a print-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

Cause: Too many files were selected for this operation.
Action: Select only the number of files specified in the message.

2455 | Too many selections for an export-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

Cause: Too many files were selected for this operation.
Action: Select only the number of files specified in the message.

2456 | Insufficient file handles available. Application cannot continue.

Cause: More than 50 files are open at the same time.
Action: Refer to an operating system manual then set the limit for open files to 50 or greater.

2457 | Results cannot be displayed. More than [*n*] windows are currently displaying or printing results.

Cause: Too many windows are open in the application.
Action: Close some of the open windows.

2458 | An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause: An attempt was made to exit the application while the instrument was performing a critical operation. This operation must be completed before the application can be exited.
Action: Wait a short time and attempt to exit the application again.

2459 | An instrument is busy. A delay in restarting this application could result in loss of new data. Continue with program Exit?

Cause: An attempt was made to exit the application while an analysis was in progress. While this is possible, the data collected when the application is inactive will not be permanently recorded until the application is re-started. A power failure to the instrument could cause some data to be lost.
Action: If not concerned with the potential for loss of data should a power failure occur, click **Yes** to continue; otherwise, click **No**.

2460 | Fatal Communications error on [*n*].

Cause: There was a fatal error in communication between the application and the software in the instrument. All displays for that instrument will be closed.
Action: Ensure that the analyzer is connected to the computer on the communications port configured in the *Setup* program. Stop and restart the analyzer software. Contact your Micromeritics service representative.

2461 | No instruments are in operation. This application will unconditionally terminate.

Cause: At least one instrument must be active for the application to operate. The initialization of all of the instruments configured with the Setup program has failed. The application stops.

Action A: Usually this message is preceded by another message giving the reason for the instrument's failure to initialize. Refer to the instructions for that message.

Action B: Ensure that the instrument is attached to the computer on the communications port configured with the *Setup* program. Verify that the instrument's power switch is in the ON position and that the light on the front panel is illuminated. If the application continues to fail in its attempts to initialize the instrument, contact your Micromeritics service representative.

2477 | [*n*] did not properly initialize.

Cause: The software was unable to initialize this instrument. This is usually caused by one of the conditions listed in the previous error messages.

Action A: Run the *Setup* program and ensure that a valid port is specified; if not, specify a valid one when prompted.

Action B: Reinstall the software, then restart application.

Action C: Contact a Micromeritics service representative if this error message continues.

2478 | Error copying sequential data segment.

Cause: An internal processing and / or hardware error occurred while accessing a portion of a sample file.

Action: Confirm that the media being accessed does not contain errors.. Contact your Micromeritics service representative.

2479 | The instrument is busy performing an operation of which this application is unaware. Do you want to cancel? [Yes, No]

Cause: During initialization of the application, the status of the analyzer was found to be in a different state than expected.

Action: Click **Yes** to cancel the operation in process and allow the analyzer to reset, then continue with initialization. Click **No**to cancel the initialization process.

If this error message continues, verify that files in the application directory structure are not being changed or removed.

2480 | File [*n*] cannot be analyzed. It is currently being edited.

Cause: An attempt was made to start an analysis using a file that is open for editing.

Action: Finish editing the file, save and close it, then start the analysis.

2481 | Error accessing the sample information file [*n*].

Cause: An unexplained error prevented access to this file.
Action: The hard disk drive may be corrupt. Run diagnostics.

2482 | File cannot be opened for writing.

Cause: An attempt was made to open a file currently being used.
Action: Locate the application using the file (in the Micromeritics application, use the Windows menu item to get a list of all open windows, one of which may contain this file).

2483 | An analysis cannot be performed on [*n*]. It is open for editing and contains errors.

Cause: An attempt was made to use a sample file containing errors that is currently open.
Action: Go to the window containing the file, correct the errors, then save it.

2484 | The edit session for [*n*] must be saved before the analysis. Save changes and continue with the analysis?

Cause: An attempt was made to start an analysis using a file that contains unsaved changes and is open for editing.
Action: Click **Yes** to save the changes, then proceed with the analysis. Click **No** to cancel the analysis, then continue editing the Sample Information file.

2485 | The service test file has an invalid status and cannot be used for this analysis.

Cause: The selected file has a status other than *No Analysis*.
Action: Select a different service test file, or create a new one and use **Replace All** to copy parameters from the file originally selected.

2486 | Could not construct [*n*] report type. Program will terminate.

Cause A: Full rights to the application's folders and files is required.
Action A: Contact a system administrator to have full rights granted.
Cause B: An internal processing and / or hardware error has occurred.
Action B: Contact your Micromeritics service representative.

2487 | Could not start report generator. Error code [*n*]. Program will terminate.

Cause A: Full rights to the application's folders and files is required.
Action A: Contact a system administrator to have full rights granted.
Cause B: An internal processing and / or hardware error has occurred.
Action B: Contact your Micromeritics service representative.

2488 | File [*n*] cannot be opened for editing.

Cause: The specified file is being used in another edit operation.
Action: Check the Windows list to locate the other edit session.

2489 | File [*n*] cannot be opened for writing.

Cause: The specified file in a *Save As* operation is already open for edit.
Action: Select a different file for the *Save As* operation.

2490 | No '.INI' file present. Application will terminate.

Cause: The ASCII .INI file containing initialization information and system options information used during program startup does not exist.
Action: Run the analyzer *Setup* program (located on the applications CD), select *Change analyzer setup* and enter the pertinent information.

2491 | Highlighted fields contain errors. Please correct the errors before dialog box.

Cause: The highlighted fields contain invalid entries. The window cannot be closed until all errors are corrected.
Action: Check the entries, correct the errors, then close the window.

2492 | This field's entry is invalid.

Cause: The highlighted field contains an invalid entry.
Action: Check the entry and correct the error.

2493 | An entry is required for this field.

Cause: This field requires a valid entry to proceed.
Action: Enter or select an appropriate value.

2494 | Value is out of the valid range.

Cause: The entered value in the highlighted field is outside the valid range of values.
Action: Check the entry, then either enter or select an appropriate value.

2495 | Value is out of the valid range. Enter a value between [*n*] and [*n*].

Cause: The entered value in the highlighted field is outside the valid range of values.
Action: Check the entry, then either enter or select an appropriate value.

2496 | Invalid number.

Cause: An invalid number was entered in the highlighted field.
Action: Check the entry, then either enter or select a valid number.

2497 | This field contains an invalid character.

Cause: An invalid character was entered in the highlighted field.
Action: Check the entry, then enter valid characters.

2498 | The requested change to the Sample's status is invalid at this time.

Cause: A request to change the file's status, for example, from *automatically collected* to *manually entered* could not be done.

Action: Contact your Micromeritics service representative. Record the name of the sample file in which the problem occurred.

2499 | Sequence number must contain at least 3 digits.

Cause: An attempt was made to enter a sequence number that did not contain at least three digits.

Action: Enter a sequence number that contains at least three digits.

2500 | All sample file names that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause: No more sample information files can be created using the currently entered file name sequence number.

Action: Go to ***Options > Default Method***, then enter another sequence number.

2501 | System resources have reached a dangerously low level. Please close some windows to avoid the loss of data.

Cause: A large number of windows are open and consuming the system resources available to all applications.

Action: Close one or more windows. Contact your Micromeritics service representative.

2505 | Error logger cannot be initialized. Error code [*n*]. Program will exit.

Cause: An internal processing error has occurred.

Action: Contact your Micromeritics service representative.

2506 | Sample file [*n*] has a *No Analysis* status and cannot be used for this operation.

Cause: The selected sample file does not have collected data and cannot be used for operations, for example, reporting.

Action: Enter the name of a file with a status of *Complete*, *Analyzing*, or *Entered*. Alternatively, select a sample file from the list box.

2507 | The sample has an invalid status and cannot be used for degassing.

Cause: A sample file has been selected which does not have a *No Analysis* or *Prepared* status.

Action: Select a different file with a status of *No Analysis* or *Prepared*.

2508 | Overlay [*n*] was not found. It will not be included in the reports.

Cause: The specified overlay file could not be found.

Action: Ensure the file specified as an overlay exists.

2509 | Error opening file [*n*]. Reports cannot be produced.

Cause: An error occurred while the program was opening a file necessary to the report operation.
Action: Use the name given in the error message to investigate. Contact your Micromeritics service representative.

2510 | Error parsing reports from file [*n*]. Reports cannot be produced.

Cause A: One or more data entry fields in the sample file may contain an invalid character (such as a single quote or double quotes).
Action A: Review the data entry fields (for example, the *Sample* field), then remove the invalid character.
Cause B: The system was unable to create the usual temporary files during the report, possibly due to insufficient disk space.
Action B: Check the space available on the hard disk.
Cause C: An internal processing error occurred.
Action C: Contact your Micromeritics service representative.

2511 | Print job [*n*] has been canceled due to insufficient disk space. Delete unnecessary files and restart the report.

Cause: The disk drive does not have required space for the temporary file.
Action: Delete unnecessary files from the disk. At least five megabytes of free space is required for normal operation.

2512 | Print job [*n*] canceled.

Cause: The print job was canceled by the operator.
Action: None required.

2513 | Unable to read the calibration file [*n*].

Cause: An invalid calibration file was selected or cannot be read.
Action: Ensure the media containing the calibration file has no problems.

2514 | Unable to write the calibration file [*n*].

Cause: An attempt to save calibration data has failed due to possible media problems.
Action A: Ensure the destination location has no problems.
Action B: Choose an alternate media to save the calibration data.

2515 | Warning: Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?

Cause: The process of performing a calibration operation was started.
Action: Calibration operations should only be done by or under the direction of qualified service personnel.

2516 | Warning: Keeping a backup copy of the calibration data is recommended by Micromeritics. Would you like to do so now?

Cause: A calibration operation was performed and a backup copy is recommended.
Action: Go to ***Unit [n] > Calibration > Save to File*** to perform a calibration save operation.

2517 | Canceling this dialog will reset the calibration state to what it was when this dialog was first opened. Are you sure you want to cancel?

Cause: The calibration has not been accepted.
Action: If the calibration operation was successful, click **Accept**.

2520 | No data points available for reporting.

Cause: The selected sample file does not have collected data and cannot be used for reporting.
Action: Select a different sample file.

2521 | Unable to program controller.

Cause: A hardware malfunction has occurred.
Action: Contact your Micromeritics service representative.

2522 | Invalid controller application file.

Cause: The application's control file has been corrupted or deleted.
Action: Reinstall the analysis program.

2523 | Programming controller failed.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2524 | CRC check failed on programming controller.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2525 | Unknown error programming controller.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2526 | Controller download was not successful.

Cause: An internal processing and / or hardware error has occurred.
Action A: Contact your Micromeritics service representative.

2527 | Controller CRC error on boot block.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2528 | Controller DRAM error.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2529 | Controller Com 1: error.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2530 | Controller Com 2: error.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2531 | Controller debug port error.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2532 | The instrument contains a different software version. Do you want to reset it?

Cause: The application has discovered a different version of software operating in the analyzer.
Action: If there are no analyzers other than the one connected to the computer, click **Yes**, then allow the updated software to load.

2533 | Analyzer initialization failed.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2534 | Unable to establish the TCP connection with [*n*].

Cause: There was a problem establishing communication with the analyzer.
Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection.

Ensure that no other Micromeritics application is initializing another instrument. If there is another Micromeritics application open and initializing an instrument, wait until the instrument initialization completes or is canceled.

Contact a Micromeritics service representative if this error message continues.

2548 | System status 1 [*n*].

Cause: There was a problem establishing communication with the analyzer.
Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2549 | Error accessing online manual file [*n*].

Cause: The operator's manual file could not be located.
Action A: Reinstall the application.
Action B: Copy the contents of the manual folder from the setup CD to the application directory.

2550 | Attempts to acquire the instrument's status timed out.

Cause: There was a problem establishing communication with the analyzer.
Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2551 | Cannot access web page [*n*].

Cause: The Micromeritics web page for DFT models cannot be accessed. This could be caused by an ISP problem of high internet traffic.
Action: Try the operation later.

2552 | Configured serial number does not match instrument.

Cause: An instrument was substituted without properly changing the instrument serial number.
Action: Use the installation program to add or move devices as necessary.

2553 | Dialog ID [*n*] can not be created!

Cause: A required window could not be found by the software.
Action: Re-install the software.

2556 | Directory database [*n*] error [*n*].

Cause: The sample file is currently selected and is undergoing a critical operation.
Action: Open the sample file after the critical operation has completed.

2557 | Cannot access web page.

Cause: The Micromeritics web page for DFT models cannot be accessed. This could be caused by an ISP problem of high internet traffic.
Action: Try the operation later.

2558 | The instrument is busy. The requested operation cannot be executed.

Cause: The instrument is analyzing and cannot be interrupted.
Action: Try the operation later.

2576 | The instrument [*n*] is not calibrated.

Cause: The analyzer application is in the process of initializing the instrument and is unable to locate the calibration files.
Action A: Click **OK**. Go to ***Unit [n] > Calibration > Load from File***, then select a file containing calibration data.
Action B: Click **OK**. Close the application, then use the *Setup* program to reinstall calibration files.

2585 | The following libraries are missing: [*n*]

Cause: This message is triggered on application start up if any of the library files used by an application, do not exist on disk.
Action: Add the library into the libraries.

2599 | The selected file has an extension that is not supported by this operation

Cause: The selected file does not have a supported file extension.
Action: Open the adsorptive properties file. Open the FPI file selector and select another file with a supported file extension.

4012 | Psat gas in sample file does not match any gas in the unit.

Cause: If using *Measure psat of a gas in* P_o *and T* options in *Analysis Conditions*, the selected gas is not one of the selected gases in *Unit Configuration*.
Action A: If the incorrect psat was selected, change the psat gas.
Action B: If the gas was recently connected to the instrument, update the *Unit Configuration*.

4014 | File [*n*] is not a valid file for conversion.

Cause: The file selected for conversion is not a valid file.
Action: Select only files that have been created by the proper program.

4015 | Error creating export file for sample [*n*].

Cause: A file error occurred during creation of an export output file.
Action: The output file name may be invalid. Ensure that the target directory exists and is not full or write protected. The target disk drive may be damaged or inoperative. Verify that other files may be created on the same drive. Contact your Micromeritics service representative.

4016 | Sample [*n*] has no data for export.

Cause: The file selected for export has a status of *No Analysis*. No export file will be

created.

Action: Select a file which contains analysis data.

4017 | Damage to the instrument will result if the sample [*n*] has not been manually evacuated. Have you evacuated the sample?

Cause: *Backfill sample at start of analysis* was not selected on the *Sample Backfill Options* window. The sample tube is normally at atmospheric pressure when an analysis is started, and it must be backfilled before the analysis begins to prevent sample material from being drawn into the manifold.

Action: If the sample tube has been manually evacuated, click **Yes**. If not, click **No**, either perform a manual evacuation or go to the *Sample Backfill Options* window, then select *Backfill sample at start of analysis*.

4020 | Disabling this option may damage the instrument. Are you sure that the sample should not be backfilled?

Cause: *Backfill sample at start of analysis* was not selected on the *Sample Backfill Options* window. The sample tube is normally at atmospheric pressure when an analysis is started; it must be backfilled before the analysis begins to prevent sample material from being drawn into the manifold.

Action: To manually evacuate the sample prior to the start of the analysis, click **Yes**. Otherwise, click **No**, go to the *Sample Backfill Options* window, then select *Backfill sample* at start of analysis.

4021 | The entered [*n*] value ([*n*] and Temperature Option of the Analysis Conditions*)* is outside the range of the pressures listed in the Psat vs Temperature Table (Adsorptive Properties).

Cause: The entered P_0 value is not within the range of pressures selected for analysis.

Action A: Enter a new P_0 value.

Action B: Add more pressures and corresponding temperatures to the *Analysis Conditions* pressure table to include the presently selected P_0 value.

4022 | The entered bath temperature value ([*n*] and Temperature Options of the Analysis Conditions) is outside the range of the temperatures listed in the Psat vs Temperature Table (Adsorptive Properties).

Cause: The entered bath temperature is outside of the range of temperatures specified in the *Adsorptive Properties*.

Action A: Change the entered temperature.

Action B: Change the adsorptive.

Action C: Add more temperatures and corresponding pressures to *Adsorptive Properties*.

4023 | The file [*n*] cannot be prepared for analysis. It is open for editing and contains errors.

Cause: An attempt was made to start an analysis using a file that contains errors and is open for editing.
Action: Finish editing this file, save and close it, then start the analysis.

4024 | Backfill gas in sample file does not match any gas in the unit.

Cause: The backfill gas specified in the sample information file does not match the analysis gas entered in the *Unit Configuration.*
Action A: If the wrong backfill gas was selected in the sample information file, change the backfill gas in the file.
Action B: If necessary, attach the appropriate gas cylinder, then enter the gas in the *Unit Configuration*.

4026 | Cannot calculate Dubinin-Astkahov: bad least squares data.

Cause: Less than two selected data points are within the fitted pressure range.
Action: Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4027 | Fewer than two sample files have data suitable for heat of adsorption reports.

Cause: Less than two of the selected sample files for heat of adsorption reports contain appropriate data.
Action: Edit the *Quantity Adsorbed* table, or select other sample files.

4028 | Dubinin calculations cannot be performed because the affinity coefficient of the analysis gas is zero.

Cause: Dubinin values could not be calculated because the affinity coefficient of the analysis gas is zero.
Action: Access the *Dubinin Report Adsorptive* options in the sample file, then enter an appropriate value for the analysis gas.

4029 | At least two fitted data points are needed for Alpha-S calculations.

Cause: Fewer than two data points fall within the selected Alpha-s range.
Action: Edit either the calculation pressure in the fitted Alpha-s range, or use a different reference curve.

4030 | Preparations failed in primary data.

Cause: Appropriate data were not available to generate the report.
Action: This message was preceded by a different error message. Refer to the cause/action of the preceding message.

4031 | Not enough points with a relative pressure in the range [*n,n*].

Cause: Fewer than two data points selected for the Dubinin report fall within the selected relative pressure range.

Action: Edit the calculation pressure range or the fitted relative pressure range.

4032 | Some summary reports could not be produced because they require the Micropore option.

Cause: Some of the summary reports requested were not produced because the micropore option was not installed.

Action: Edit the summary report and deselect the micropore options.

4033 | Not enough points to generate Dubinin Tabular Report.

Cause: There are fewer than two valid data points available for the Dubinin tabular reports.

Action: At least two micropore pressures must be selected for inclusion in the Dubinin report. Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4034 | Fewer than 2 points available for Dubinin calculations.

Cause: There are fewer than two valid data points available for Dubinin reports in one of the sample files selected for overlaying.

Action: At least two micropore pressures must be selected for inclusion in the Dubinin report. Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4035 | Cannot calculate optimized Astakhov exponent.

Cause: There are fewer than two valid data points in the relative pressure range specified. Astakhov reports will not be produced.

Action: At least two pressures must be selected for inclusion in the Astakhov report. Edit the selection of data points on the Astakhov interactive editor or on the *Astakhov Pressures* window.

4036 | Fewer than 2 points available for Horvath-Kawazoe calculations.

Cause: At least two data points must be selected for inclusion in the Horvath-Kawazoe analysis. No report will be produced.

Action: Edit the selection of points on the Horvath-Kawazoe interactive editor or on the *Horvath-Kawazoe* window.

4037 | Computations failed while processing the primary data set. No reports will be produced.

Cause: The preparation of data for reporting could not be successfully completed. No Horvath-Kawazoe reports will be produced. This message will always be preceded

with another one containing additional information.

Action: Refer to the error message number which preceded this one for an explanation.

4038 | Fewer than 2 points available for the Langmuir Qm computation.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm must include at least two points above 0.02 relative pressure for the Langmuir equation to be applied.

Action: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the *Horvath-Kawazoe Report Options* window to prevent this message from appearing on future reports.

4039 | The isotherm does not meet the constraints of the Cheng/Yang assumption.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm does not correlate to the Langmuir assumption with a coefficient of 0.98 or more. The correction is not applicable to this isotherm or to the range of the data points selected.

Action A: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the *Horvath-Kawazoe Report Options* window to prevent this message from appearing on future reports.

Action B: Generate the Langmuir report for the same data points selected for the Horvath-Kawazoe report. If the Langmuir correlation coefficient can be brought above 0.98 by removing some points at high relative pressure, remove them, then reproduce the Horvath-Kawazoe reports.

4040 | The value of Qm computed from the Langmuir equation is too low.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The computed value is less than the volume adsorbed at the largest relative pressure included in the analysis. The correction is not applicable to this isotherm or to the range of the data points selected.

Action: The analysis will be performed and the Cheng/Yang correction will be applied to all points with a volume adsorbed less than the value of Vm. The pore size will not be calculated for data points with an invalid volume adsorbed. Deselect Apply Cheng/Yang correction on the *Horvath-Kawazoe Report Options* window to clear this message.

4041 | Cheng/Yang correction is inappropriate for some [*n*].

Cause: The Cheng/Yang correction is usually inappropriate for any P/P_0 above the isotherm knee. In some instances, the computed pore sizes may decrease above the knee. While it is possible to include these relative pressures (usually above 0.1

P/P_0) in the analysis, the computed pore sizes for these pressures are usually meaningless.

Action: Change the data points selected for the Horvath-Kawazoe report to include only relative pressures at or below the knee of the isotherm, or change the Horvath-Kawazoe report options so that the Cheng/Yang correction is not applied.

4042 | 0.0 cannot be a starting or ending pressure for a geometric progression from low pressure.

Cause: An attempt was made to generate a pressure table from a geometrically progressing range.

Action: Change the 0.0 entered value.

4043 | 1.0 cannot be a starting or ending pressure for a geometric progression toward saturation.

Cause: An attempt was made to generate a pressure table from a geometrically progressing range.

Action: Change the 1.0 entered value.

4044 | Points in the Langmuir report pressure table lie outside the collected data.

Cause: Calculation pressure range is not being used. More than one of the report pressure table points is above the range of the collected data and more than one is below.

Action: Change the report pressure table to be more consistent with the collected data.

4045 | Points in the report pressure table lie outside the collected data.

Cause: Calculation range is not being used. More than one of the report pressure table points is above the range of the collected data and more than one is below.

Action: Change the report pressure table to be more consistent with the collected data.

4046 | [*n*] could not be opened for reading.

Cause: A thickness curve file could not be opened.

Action: If the problem persists, restart the computer, then optionally perform a media integrity check.

4047 | Warning: An error occurred while reading [*n*].

Cause: An error happened during a read operation of a thickness curve file.

Action: If the problem persists, restart the computer, then optionally perform a media integrity check.

4048 | Warning: An error occurred while restoring the heat of adsorption report editor.

Cause: The state of the heat of adsorption report editor could not be restored. Default

settings will be used.

Action: No action.

4049 | The sample [*n*] does not have enough data. A minimum of two adsorption points is required.

Cause: A sample file has been included in the Heat of Adsorption report that does not have enough data.

Action: Remove the file from the selected file list.

4050 | None of the requested quantities adsorbed is within the range of the collected data of more than one sample file.

Cause: The *Heat of Adsorption* report failed because the specified quantities are not within the range of the collected data.

Action: Edit the quantities adsorbed so that they are within the range of the collected data, or select other sample files.

4051 | The sample [*n*] does not have any data in the range of the requested quantities adsorbed.

Cause: The sample's data cannot be interpolated to any of the quantities adsorbed.

Action: Edit the quantities adsorbed so that they are within the range of the collected data.

4052 | Fewer than two points are selected for this report.

Cause: At least two points are required for the BET calculations.

Action: Edit the calculation range in the BET report.

4053 | At least two data points must be selected for t-Plot calculations.

Cause: At least two points are required for the t-Plot calculations.

Action: Edit the calculation range for the t-Plot report.

4054 | Fewer than two data points are inside the fitted thickness range.

Cause: At least two points must be within the fitted thickness range for the *t*-Plot calculations.

Action A: Edit the calculation range for the *t*-Plot report.

Action B: Edit the fitted thickness range in the *t*-Plot report editor.

4055 | A positive BET surface area was not calculated. Please check your BET Report.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.

Action A: Assign more points to the surface area calculation.

Action B: Select a different surface area in the *t*-Plot report editor.

4056 | A positive Langmuir surface area was not calculated. Please check your Langmuir report.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.
Action A: Assign more points to the surface area calculation.
Action B: Select a different surface area in the t-Plot report editor.

4057 | At least two data points are needed for Freundlich calculations.

Cause: Less than two data points have been selected for the Freundlich report; at least two are required.
Action: Edit the selection of points on the Freundlich interactive editor or on the *Freundlich Pressures* window.

4058 | At least two data points are needed for Temkin calculations.

Cause: Less than two data points have been selected for the Temkin report; at least two are required.
Action: Edit the selection of points on the Temkin interactive editor or on the *Temkin Pressures* window.

4059 | Fewer than 2 points available for MP-Method calculations.

Cause: At least two points are required for the MP-Method calculations.
Action: Edit the calculation range for the MP-Method report.

4060 | Sample [*n*] contains no data points.

Cause: An attempt was made to save a sample without collected data as a t-curve or alpha-S curve.
Action: Repeat the *Save As t-curve* or *Save As alpha-S* operation after opening a sample that has collected data.

4061 | The t-curve must contain at least 2 points.

Cause: At least two points are required in a thickness curve definition.
Action: Edit the thickness curve.

4062 | Error during report preparation.

Cause: An internal processing error has occurred.
Action: Contact your Micromeritics service representative.

4063 | The data requested on this report are not available. No subreports selected.

Cause: There is no information in the sample log to report.
Action: A sample file was selected of which no instrument operations were used. Select a

sample file with a status of *Prepared*, *Preparing*, *Analyzing*, or *Complete* to obtain a valid sample log report.

4067 | No data points are within the range of pressures in the reference isotherm.

Cause: There are no collected data points within the range of pressures in the reference isotherm.

Action: Select data points in the range of the reference isotherm, or select a more appropriate reference isotherm.

4068 | No points were selected for the *f*-Ratio report.

Cause: The *f*-Ratio report does not have any points selected.

Action: Edit the selection of data points on the *f-Ratio* window.

4070 | Unable to load deconvolution model [*n*].

Cause: The list of available models was corrupted; therefore, the model selected could not be loaded for the deconvolution.

Action: Exit the application. Reinstall the software, then try again.

4071 | The selected pressures points do not form a valid set for deconvolution.

Cause: The data points selected for analysis do not contain enough information to allow a DFT data reduction.

Action: At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction. Edit the selection of data points on the DFT interactive editor or on the *DFT Pressures* window.

4072 | The range of pressures selected is too small to deconvolute using this model.

Cause: A null result was found using the selected model.

Action: At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction. Edit the selection of data points on the DFT interactive editor or on the *DFT Pressures* window.

4073 | The analysis gas [*n*] does not match the model gas [*n*].

Cause: The model assumes a specific gas, and the sample file uses a different one.

Action: Select a model that assumes the same gas.

4074 | The analysis temperature [*n*] does not match the model temperature [*n*].

Cause: The temperature for the selected model did not match the analysis temperature.

Action: Select a different model.

4075 | The models cannot be located in the models folder. Reinstall the software.

Cause: The models could not be located. They may have been inadvertently deleted or moved.
Action: Reinstall the software.

4077 | Cannot get surface area for: [*n*]

Cause: The Isotherm report for the named overlay file has Per gram selected for the *Volume Adsorbed*, and the Isotherm report for the primary file has a surface area option selected for the *Volume Adsorbed*.
Action A: Edit the Isotherm report for the named overlay file, then select a surface area option for *Volume Adsorbed*.
Action B: Click **Overlays** on the *Report Options* window of the primary file, then remove the named overlay file from the list.

4078 | Slope and Y-Intercept cannot be determined from the selected points.

Cause: The Langmuir report cannot be generated from the selected points.
Action: Edit the calculation pressure range in the Langmuir report pressure window.

4112 | Hard-sphere diameter, molecular weight, and mass flow constant have been updated from the fluid property information.

Cause: A new fluid property information file was loaded. The indicated fields have been updated with values from the file.
Action: This message is informational; no action is required.

4135 | HOA file [*n*] does not exist.

Cause: The sample file in the *Heat of Adsorption* report list does not exist.
Action: Go to ***Report > Heat of Adsorption***. Click **Add Samples**, then select the sample file.

4136 | HOA file [*n*] is corrupt.

Cause: The sample file in the *Heat of Adsorption* report list is corrupt.
Action: Go to ***Report > Heat of Adsorption***. Select the corrupt sample file, then click **Remove Sample**. Rerun the *Heat of Adsorption* report.

4407 | Error searching for installed Smart VacPreps. The registry could not be read.

Cause: The application was not installed properly.
Action: Reinstall the application. If the problem persists, contact your Micromeritics service representative.

4408 | The [*n*] in [*n*] already has Smart VacPrep S/N [*n*] . The Smart VacPrep must be removed from the [*n*] before it can be added.

Cause: The Smart VacPrep was already installed for another application / unit.

Action: Remove the Smart VacPrep from the installed unit before adding it to the preferred unit.

4409 | A free IP address on the same subnet as [*n*] could not be found.

Cause: All IP addresses on the network for the Ethernet card specified during installation are in use by other Micromeritics applications on this computer.
Action A: Uninstall unused Micromeritics applications.
Action B: Configure a different Ethernet card for use by the application using the application installer.

4410 | The .INI file could not be updated with configuration for Smart VacPrep S/N [*n*].

Cause: The application .INI file is opened by another application and could not be updated.
Action: Close all open applications and add the Smart VacPrep again using the Smart VacPrep menu.

4411 | Error dosing.

Cause: The backfill timed out.
Action: Ensure there is gas available and the pressure regulator is set to the appropriate pressure. Also ensure that the gas supply regulator shutoff valve is open.

4412 | Error calibrating the servo.

Cause: Calibration results are out of range.
Action: Follow standard calibration procedures and try again. If the problem persists, contact your Micromeritics service representative.

4413 | Error overheating on port [*n*] . Current = [*n,n*], Target = [*n,n*], Limit = [*n.n*].

Cause: The temperature of the indicated mantle exceeded the maximum allowed value.
Action: Ensure the power and thermocouple connectors for the mantle are properly installed. If they are, the mantle may be defective; contact your Micromeritics service representative.

4414 | Error thermocouple unplugged on port [*n*]. Target = [*n,n*].

Cause: The thermocouple is unplugged or has malfunctioned.
Action: Ensure the thermocouple is plugged in. If the problem persists, contact your Micromeritics service representative.

4415 | Degas transducer zero calibration failed. Current Offset = [*n*] counts, Current Pressure = [*n,n*], New Offset = [*n*] counts, Nominal = [*n*] counts.

Cause: The pressure transducer offset exceeds the recommended limit.
Action: Ensure that the vacuum pump is on. Repeat the pressure offset calibration..Contact a Micromeritics service representative if this error message continues.

4416 | Degas transducer scale calibration failed. Reference = [*n,n*], Current = [*n,n*], New

Scale = [*n,n*] / count, Nominal = [*n,n*] / count.

Cause: The transducer scale calibration was rejected.
Action: Contact your Micromeritics service representative.

4417 | Degas vacuum gauge low point calibration failed. Reference = [*n,n*] / count, Current = [0] counts.

Cause: The transducer offset calibration was rejected.
Action: Contact your Micromeritics service representative.

4418 | Degas vacuum gauge high point calibration failed. Reference = [*n,n*], Current = [0] counts.

Cause: The vacuum gauge calibration was rejected.
Action: Contact your Micromeritics service representative.

4419 | Error reading servo DAC.

Cause: There is a problem with the servo DAC timing out.
Action: Contact your Micromeritics service representative.

4420 | Communications error.

Cause: The application failed to connect to the Smart VacPrep.
Action: Ensure the unit is powered and properly connected to the network specified during installation. Check the power cable, power switch, and Ethernet cable, then reconnect to the Smart VacPrep either through the Smart VacPrep menu for this instrument or by restarting the application. If the problem persists, contact your Micromeritics service representative.

4421 | Smart VacPrep S/N [*n*] is busy and could not be removed.

Cause: The Smart VacPrep cannot be removed because it is currently performing an operation.
Action: Wait until the Smart VacPrep completes the current operation and try again.

4422 | The file [*n*] does not exist.

Cause: The selected file does not exist on the disk drive.
Action: Select an existing file. Ensure that the file has been created before use.

4423 | The sample [*n*] is already selected on port [*n*].

Cause: The selected sample file is already selected for use on a different port.
Action: Select another sample file for this port.

4424 | The file [*n*] on port [*n*] could not be opened. Check if the sample file is already in use for editing or analysis.

Cause A: The selected sample file is already open by this or another application.
Cause B: The selected sample file is damaged.
Action: Select another sample file.

4425 | The sample [*n*] on port [*n*] has an invalid status and cannot be used for degassing.

Cause: The status of the file is not consistent with the current operation.
Action: Select a sample file that has not been used for an analysis. Only sample files with a status of *No Analysis* or *Prepared* may be selected.

4426 | Port [*n*] is currently in use. Operation cannot be started.

Cause: The current operation cannot be completed because the port is already in use.
Action: Wait for port to terminate operation or perform the desired operation on an unused port.

4430 | Pressure level is out of range.

Cause: The reference gauge reading is not valid for scaling the manifold pressure transducer.
Action: Adjust the manifold pressure until the reference gauge reading is in the recommended scale calibration range.

4431 | Pressure is out of range.

Cause: The reference gauge reading is not valid for scaling the manifold pressure transducer.
Action: Adjust the manifold pressure until the reference gauge reading is in the recommended scale calibration range.

6000 | An error occurred while loading the application control information. Data entry cannot be performed. (Code [*n*]).

Cause: An error occurred accessing the control information disk file required by this application.
Action: The disk drive may have failed or be corrupt. Run diagnostics on the disk drive.

6002 | File cannot be opened for writing.

Cause: An attempt to save a file marked as "read-only" was made. Files can be marked automatically as read-only when they are transferred from a CD to the application directory.
Action: Use Windows Explorer to access the folder containing the file, then disable the Read-Only option.
Action: Enter a different name to save the file.

6003 | Unable to read the calibration file [*n*].

Cause: An attempt to load a previously saved calibration file was unsuccessful.
Action: Ensure the file exists and the file name is entered correctly, then try again.

6004 | Unable to write the calibration file [*n*].

Cause: An attempt to save the calibration to a separate file was unsuccessful.
Action: Ensure the file exists and the file name is entered correctly, then try again.

6005 |The sample has an invalid status and cannot be used for this analysis.

Cause: A sample file with a status other than *No Analysis* or *Prepared* was selected.
Action: Select a sample file with a status of *No Analysis* or *Prepared*.

6008 | At least one sample must be selected to proceed.

Cause: An attempt was made to start an analysis without selecting any sample files.
Action: Select at least one file, then start the analysis.

6010 |This sample requires a different adsorptive and cannot be analyzed at the same time as the other samples.

Cause: A sample file for analysis was selected that requires a different adsorptive gas than the sample files selected for the other ports.
Action: Select only sample files to be analyzed with the same adsorptive, then perform the analysis. Next, perform the analysis with the other adsorptive.

6011 |The adsorptive required by this analysis is not available on this instrument.

Cause: An attempt was made to start an analysis with an adsorptive that is not connected to the instrument or has not been designated in the software.
Action A: Ensure that the adsorptive is connected to the instrument, then select ***Unit [n] > Unit Configuration*** to tell the application that the gas is connected.
Action B: Select only sample files for which the analysis gas is available.

6012 | Cannot read the analysis conditions parameter file.

Cause: The parameter file is either corrupt or has been deleted.
Action A: If this is a file created in a lab, recreate the file.
Action B: If this is a default file created during application installation, re-install the software.

6013 | Cannot read the adsorptive properties parameter file.

Cause: The parameter file is either corrupt or has been deleted.
Action A: If this is a file created in a lab, recreate the file.
Action B: If this is a default file created during application installation, re-install the software.

6014 | Cannot read the report options parameter file.

Cause: The parameter file is either corrupt or has been deleted.
Action A: If this is a file created in a lab, recreate the file.
Action B: If this is a default file created during application installation, re-install the software.

6015 | Cannot read the sample tube properties parameter file.

Cause: The file selected as a sample tube properties file is not valid.
Action: Select a different file.

6016 | Dosing manifold from valve [*n*] failed.

Cause A: The maximum time was exceeded before the target pressure point was reached. The nitrogen regulator may be set too low or turned off.
Action A: Set the analysis gas regulator to at least 10 psig (0.7 bar), then resume the analysis.
Cause B: The analysis gas cylinder is empty.
Action B: Connect a new analysis gas cylinder, then resume the analysis.

6017 | Leak test failed on port [*n*].

Cause: With the sample port valve closed, the sample pressure increased by 0.15 mmHg before the leak test duration was completed.
Action: Check sample tube fitting and ensure that it is securely attached to the port, then restart the analysis.

6018 | Volume dosed exceeded [*n,n*]. Analysis is canceled.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6019 | Elevator failed to reach lower limit switch.

Cause A: There is an obstruction in the elevator path.
Action A: Clear all obstructions and restart the analysis.
Cause B: Ice is present in either the bottom or the neck of the dewar, preventing the elevator from rising completely.
Action B: Check the dewar, remove ice, then restart the analysis.
Cause C: If results for Actions A and B failed, contact a Micromeritics service representative.
Action C: There is an obstruction in the elevator path.

6020 | Warning, servo valve performance is out of specification.

Cause: The servo valve tried to dose to a pressure but was unable to reach it within specification. The analysis will continue.

Action: At the next appropriate time, calibrate the servo valve to bring it back within specification.

6021 | Servo calibration failed.

Cause A: The maximum time was exceeded before the target pressure point was reached. The nitrogen regulator may be set too low or turned off.
Action A: Set the analysis gas regulator to at least 10 psig (0.7 bar), then resume the analysis.
Cause B: The analysis gas cylinder is empty.
Action B: Connect a new analysis gas cylinder, then resume the analysis.

6022 | This file already selected for the analysis.

Cause: An attempt was made to choose a file for the analysis on this port, which has been selected for another port.
Action: Choose a different file.

6023 | Desorption points in this sample file cannot be processed by this instrument. Continue with adsorption points?

Cause: The selected sample file in this analysis has desorption points selected in the pressure table. The analyzer (Gemini 2390a or 2390p) cannot report desorption data.
Action A: Click **Yes** to continue with the analysis and report adsorption data only. Click **No** either to choose a different file or to edit the current file.
Action B: If using a Gemini 2390t, select *Start Analysis* for that unit.

6024 | Evacuation failed.

Cause: While attempting to zero the pressure transducers, the instrument was unable to evacuate to a pressure of less than 1 mmHg. This may be due to a leak or a bad calibration.
Action A: Check the sample tube fitting and ensure that it is securely attached to the port.
Action B: Use the Setup program to reinstall the calibration files.

6025 | Target pressure [*n,n*] exceeded maximum manifold pressure of [*n,n*] Analysis is canceled.

Cause: An absolute pressure greater than (pressure) units was attained that exceeded the specified maximum manifold pressure.
Action: The analysis was canceled. All previously collected data were stored. Change the maximum manifold pressure value in the *Adsorptive Properties* file.

6026 | Psat gas is not condensing.

Cause A: The working dewar does not contain enough bath liquid.
Action A: Refill the dewar, then try the operation again.

Cause B: The Psat gas is contaminated.
Action B: Replace the Psat gas supply.
Cause C: The Psat tubing from the regulator to the instrument is contaminated.
Action C: Pump out the tubing.

6027 | There is no Nitrogen attached to the unit.

Cause: A calibration requiring nitrogen was attempted but the software does not recognize that nitrogen is attached.
Action: Ensure that a nitrogen gas cylinder is installed at one of the analysis ports, select ***Unit > Unit Configuration***, then enter *N2* for the appropriate valve.

6028 | The backfill gas in sample file does not match any gas in the unit.

Cause: An attempt was made to start an analysis with a gas that is not connected to the instrument or has not been designated in the software.
Action: Ensure the gas is connected to the instrument, then select ***Unit [n] > Unit Configuration*** to tell the application that the gas is connected.

6029 | The Po in the sample file does not match any gas in the unit.

Cause: An attempt was made to start an analysis with a gas that is not connected to the instrument or has not been designated in the software.
Action: Ensure the gas is connected to the instrument, then select ***Unit [n] > Unit Configuration*** to tell the application that the gas is connected.

6030 | Dosing method choice is invalid. The Krypton analysis requires that Adsorptive Properties *Dosing Method* is set to *From Psat tube*.

Cause: A file was selected for a krypton analysis that has *Normal* selected for the *Dosing Method*. *Normal* is for standard analyses only.
Action: Open the sample file and change the *Dosing Method* to *From Psat tube*. Otherwise, select a different file for the analysis.

6031 | Dosing method choice is invalid. The analysis requires that Adsorptive Properties *Dosing Method* is set to *Normal*.

Cause: A file was selected for a standard analysis that has *From Psat tube* selected for the *Dosing Method*. *From Psat tube* is for krypton analyses only.
Action: Open the sample file and change the *Dosing Method* to *Normal* . Otherwise, select a different file for the analysis.

6032 | Template file [*n*] for the selected analysis type does not exist. Select another analysis type.

Cause: A program piece required to run the PCP analysis is missing. Applies when in *Service Test Mode*.

Action: Re-install the software.

6033 | Krypton gas is not condensing in the Psat tube [*n,n*].

Cause A: The krypton gas may be contaminated.
Action A: Evacuate the krypton gas inlet line.
Action A: Manually verify the saturation pressure of the krypton gas:

1. Evacuate the psat tube.
2. Backfill with krypton gas to 20 mmHg.
3. Raise the dewar.

These steps should condense the krypton gas to a pressure below 3 mmHg.
Cause B: The dewar does not contain enough cryogen.
Action B: Refill the dewar.

6034 | Zeroing of a transducer failed. Analysis Canceled.

Cause: The transducer did not respond correctly.
Action: Contact your Micromeritics service representative.

6035 | Purification of krypton in the Psat tube failed.

Cause A: Krypton pressures in the psat tube did not stabilize after the purification steps.
Action A: Evacuate the krypton gas inlet line.
Action A: Manually verify the saturation pressure of the krypton gas:

1. Evacuate the psat tube.
2. Backfill with krypton gas to 20 mmHg.
3. Raise the dewar.

These steps should condense the krypton gas to a pressure below 3 mmHg.
Cause B: The dewar does not contain enough cryogen.
Action B: Refill the dewar.

6036 | Pressure is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Select ***Unit > Calibration > Load from File***, then select a file containing calibration data. Contact a Micromeritics service representative if this error message continues.

6037 | Sample pressure underrange.

Cause: A test of the sample pressure transducer showed a low reading. The current operation will continue.
Action: Contact a Micromeritics service representative if this error message continues.

6038 | Sample pressure overrange.

Cause A: Adsorbate gas pressure is too high. The current operation will continue.
Action A: Verify that the gas inlet pressure regulator setting does not exceed 15 to 18 psi (103.4 to 124.1 kPa). Contact your Micromeritics service representative.
Cause B: An error occurred in the pressure measurement electronics.
Action B: Contact your Micromeritics service representative.

6039 | Po pressure underrange.

Cause: A test of the Po pressure transducer showed a low reading. The current operation will continue.
Action: Contact your Micromeritics service representative.

6040 | Failed to reach pressure dosing through servo. Calibration canceled.

Cause A: There was insufficient gas pressure to calibrate for matching transducers. The gas is not connected or the tank is almost empty.
Action A: Ensure the gas is connected properly and assigned correctly in the *Unit Configuration* window. Replace the tank if necessary.
Cause B: The gas valve is not working properly.
Action B: Contact your Micromeritics service representative.

6041 | Servo Calibration failed.

Cause: There is a problem with the analyzer’s calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6042 | Master pressure transducer calibration failed. Offset is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6043 | Port 1 pressure transducer calibration failed. Offset is out of range.

Cause: There is a problem with the analyzer’s calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6044 | Port 2 pressure transducer calibration failed. Offset is out of range.

Cause: There is a problem with the analyzer’s calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6045 | Port 3 pressure transducer calibration failed. Offset is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6046 | Po pressure transducer calibration failed. Offset is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6047 | 10 torr pressure transducer calibration failed. Offset is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6048 | Master pressure transducer calibration failed. Scale is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6049 | Port 1 pressure transducer calibration failed. Scale is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6050 | Port 2 pressure transducer calibration failed. Scale is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6051 | Port 3 pressure transducer calibration failed. Scale is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6052 | Po pressure transducer calibration failed. Scale is out of range.

Cause: There is a problem with the analyzer's calibration.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6053 | 10 torr transducer failed. Scale is out of range.

Cause: There is a problem with the analyzer's calibrations.
Action: Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6557 | File [*n*] already selected for the analysis.

Cause: The same sample file is already assigned to a different port for this analysis.
Action: Select a different sample file.

6558 | Gas [*n*] in sample file [*n*] does not match any gas in the unit.

Cause: The analysis gas specified in the sample information file does not match the analysis gas entered in the *Unit Configuration.*
Action: If the wrong adsorptive was selected in the sample information file, change the adsorptive in the file.

10050 | Pirani offset calibration is invalid.

Cause: There is a problem with the Pirani offset calibration.
Action: Contact your Micromeritics service representative.

10070 | Cold cathode offset calibration is invalid.

Cause: There is a problem with the cold cathode offset calibration.
Action: Contact your Micromeritics service representative.

10080 | Cold cathode scale calibration is invalid.

Cause: There is a problem with the cold cathode scale calibration.
Action: Contact your Micromeritics service representative.

10100 | Vacuum gauge (Pirani) error detected.

Cause: There is a problem with the vacuum gauge (Pirani).
Action: Contact your Micromeritics service representative.

10110 | Vacuum gauge (cold cathode) error detected.

Cause: There is a problem with the vacuum gauge (cold cathode).
Action: Contact your Micromeritics service representative.

10120 | Vacuum gauge communications error

Cause: There is problem with communication between the instrument and the vacuum gauge.
Action: Contact your Micromeritics service representative.

10180 | [XDCR] Transducer offset calibration rejected (current: [*n,n*], new: [*n*], nominal: [*n*], max: [*n*]**).**

Cause: There is problem with communication between the instrument and the vacuum gauge.
Action: Contact your Micromeritics service representative.

10190 | Transducer scale calibration rejected (current: [*n,n*], new: [*n*], nominal: [*n*], min: [*n*], max: [*n*]).

Cause: The transducer scale calibration was rejected.
Action: Contact your Micromeritics service representative.

10200 | Transducer underrange detected.

Cause: There is a problem with the transducer underrange.
Action: Contact your Micromeritics service representative.

10200 | Transducer overrange detected.

Cause: A manifold pressure over 1000 mmHg was detected.
Action: Observe caution when operating the analyzer manually. Contact a Micromeritics service representative if this error message continues.

10240 | Temperature offset calibration rejected.

Cause: There is a problem with the temperature offset calibration.
Action: Contact your Micromeritics service representative.

10250 | Temperature scale calibration rejected.

Cause: There is a problem with the temperature scale calibration.
Action: Contact your Micromeritics service representative.

10270 | Invalid servo calibration error.

Cause: There is a problem with the servo calibration.
Action: Contact your Micromeritics service representative.

10280 | Servo DAC timeout detected.

Cause: There is a problem with the servo DAC timing out.
Action: Contact your Micromeritics service representative.

10300 | Turbo pump failure detected.

Cause: There is a problem with the turbo pump.
Action: Contact your Micromeritics service representative.

10310 | Turbo pump temperature error detected.

Cause: There is a problem with the turbo pump temperature.
Action: Contact your Micromeritics service representative.

10320 | Turbo pump communications error detected.

Cause: There is a problem with the turbo pump communication.
Action: Contact your Micromeritics service representative.

10360 | Time limit exceeded while waiting for the elevator to rise into position (elapsed: [*n*] s, max allowed: [*n*] s, status: [*n*], alarm code: [*n*], inputs: [*n*], position: [*n*]).

Cause: There is a problem with the elevator.
Action: Contact your Micromeritics service representative.

10370 | Time limit exceeded while waiting for the elevator to lower into position (elapsed: [*n*] s, max allowed: [*n*] s, status: [*n*], alarm code: [*n*], inputs: [*n*], position: [*n*]).

Cause A:: There is a problem with the elevator.
Action A: Check the dewar. Remove ice if necessary, then restart the analysis. Contact your Micromeritics service representative.
Cause B: The Psat tube is interfering with elevator movement.
Action B: Ensure the Psat tube is close to the sample tube and the dewar lid is over both the sample and Psat tubes, then restart the analysis. Contact your Micromeritics service representative if necessary.

10380 | Elevator error detected (code: [*n*])

Cause: There is a problem with the elevator.
Action: Contact your Micromeritics service representative.

10390 | Homing of the elevator failed (position: [*n*], home sensor: [*n*]).

Cause: There is a problem with the elevator.
Action: Contact your Micromeritics service representative.

10410 | Elevator communications error detected.

Cause: There is a problem with the elevator.
Action: Contact your Micromeritics service representative.

10420 | [*n*] **over-pressure detected (pressure: [*n,n*], max allowed: [*n*]).**

Cause A: A pressure greater than 1000 mmHg was detected in the instrument. The instrument has automatically canceled any operations in progress and taken action to relieve the pressure.
Action A: If the instrument was being operated manually, review recent activity to identify the cause of the overpressure and avoid a repetition. Contact a Micromeritics

service representative if this error message continues.

Cause B: If the error occurred when the dewar was lowered, excessive adsorption of condensation of gas may have occurred during analysis and returned to gas phase too rapidly when the dewar was lowered.

Action B: Revise the analysis conditions or sample quantity to prevent recurrence. Contact a Micromeritics service representative if this error message continues.

Cause C: If the error occurred during dosing from a gas inlet, the gas inlet pressure might be excessive.

Action C: Adjust the gas inlet pressure to recommended range. Contact a Micromeritics service representative if this error message continues.

10480 | Operation canceled by operator.

Cause: The operator canceled the operation.

Action: None.

10490 | Operation canceled by instrument.

Cause: The instrument canceled the operation.

Action: An accompanying message will display explaining why the operation was canceled. Correct the indicated problem and restart the operation.

10500 | Operation skipped by operator.

Cause: The operator skipped the operation.

Action: None.

10510 | Operation suspended by operator

Cause: The operator suspended the operation.

Action: None. Click **Play** to resume the operation when on the *Analysis* window.

10520 | Operation suspended by instrument.

Cause: The instrument suspended the operation.

Action: An accompanying message will display explaining why the operation was suspended. Correct the indicated problem. Click **Play** to resume the operation when on the *Analysis* window.

10530 | Operation resumed by operator.

Cause: The operator resumed the operation.

Action: None.

10560 | Instrument communications error detected.

Cause: There was a fatal error in communication between the application and the software

in the instrument. All displays for that instrument will be closed.

Action: Ensure that the analyzer is connected to the computer on the communications port configured in the *Setup* program. Stop and restart the analyzer software. Contact your Micromeritics service representative.

10710 | Manifold temperature error detected (manifold: [*n,n*], port: [*n,n*], heater: [*n,n*], heater target: [*n,n*], power: [*n*]).

Cause: An error was detected during manifold temperature control.

Action: Ensure the cover to the upper cabinet of the instrument is securely fastened. If the source of the problem has been identified and corrected, close the application program, cycle power to the instrument off for a few seconds, then turn the power *ON*. Restart the application program. Contact a Micromeritics service representative if this error message continues. Contact the local fire department, if necessary..

10720 | Manifold heater breaker open (manifold: [*n,n*], port: [*n,n*], heater: [*n,n*], heater target: [*n,n*], power: [*n*]).

Cause: The circuit breaker to the manifold heater is open.

Action: Contact your Micromeritics service representative.

10730 | Mantle temperature error detected (type: [*n*], actual: [*n,n*], max allowed: *[n,n]*, target: [*n,n*], power: [*n*])

Cause: An error was detected with the mantle temperature.

Action: Contact your Micromeritics service representative.

10740 | Mantle breaker open (type: [*n*], actual: [*n,n*], max allowed: *[n,n]*, target: [*n,n*], power: [*n*])

Cause: The circuit breaker to the mantle is open.

Action: Contact your Micromeritics service representative.

10750 | Time limit exceeded during evacuation (target: [*n,n*], pressure: [*n,n*], elapsed: [*n*] s).

Cause: The maximum time for evacuating the sample was exceeded. Possible causes are a leak in the sample tube fitting, a crack in the sample tube, or a poorly degassed sample.

Action: Check the sample tube and the sample tube fitting. Ensure that the tube is securely attached to the port. Ensure that the sample is properly degassed, then restart the analysis.

10760 | Time limit exceeded while dosing (gas: [*n*], valve: [*n*], target: [*n,n*], pressure: [*n,n*], elapsed: [*n*] s).

Cause A: The specified pressure was not attained. The gas regulator may be set too low or turned off.
Action A: Set the gas regulator to 10 psig (0.7 bar), then restart the analysis.
Cause B: The gas cylinder is empty.
Action B: Connect a new gas cylinder, then restart the analysis.

10770 | Attempts to dose failed on sample port [*n*]. (qty required: [*n,n*], qty dosed: [*n,n*], sample pressure: [*n,n*], gas: [*n*]).

Cause: There was a problem dosing the sample to target pressure. The instrument was unable to dose the required quantity of gas after several attempts.
Action: Check that the outlet stage of the gas regulator is within specification. Review the analysis parameters.

10780 | Leak test failed (sample port: [*n*], interval: [*n*] s, leak rate: [*n,n*]/min, max allowed: [*n,n*]/min).

Cause: With the sample port valve closed, the sample pressure increased by 0.15 mmHg before the leak test duration was completed.
Action: Check sample tube fitting and ensure that it is securely attached to the port, then restart the analysis.

10790 | Quantity dosed on sample port [*n* (*n,n*)] has exceeded the maximum of [*n,n*] (qty dosed this point [*n,n*], pressure: *[n,n]*, *[n]:* [*n,n*], gas: *[n*]).

Cause: An excessive quantity of gas has been dosed into the sample port due to an excessive quantity of a sample with high pore volume, condensation of gas due to a lower than expected saturation pressure, or a leak.
Action: Review the analysis conditions, the sample quantity, and the sample tube connection to identify, then correct the problem before repeating the analysis.

10801 | P0-over-sample failed on sample port [*n*] (pressure: [*n,n*], last pressure: [*n,n,n*], [*n*]: [*n,n*], rel pressure: [*n*], qty ads: [*n,n*], doses: [*n*])

Cause: Attempts to condense the adsorptive gas in the sample tube have failed due to an excessive quantity of a sample with high pore volume, adsorptive gas contamination, a higher than expected saturation pressure, or a leak.
Action: Review the analysis conditions, the sample quantity, the gas supply, and the sample tube connection to identify, then correct the problem before repeating the analysis.

10830 | Warm free-space measurement failed on sample port [*n*] (qty in free-space: [*n,n*], qty in port: [*n,n*], pressure: [*n,n*], port vol: [*n,n*], port temp: [*n,n*]).

Cause: There is a problem with the warm free-space measurement on the sample port.
Action: Ensure that no problem exists with the sample tube or gas connection.

10840 | Cold free-space measurement failed on sample port [*n*] (qty in free-space: [*n,n*],

qty in port: [*n,n*], pressure: [*n,n*], port vol: [*n,n*], port temp: [*n,n*]**, warm free-space:** [*n,n*]**).**

Cause: There is a problem with the cold free-space measurement on the sample port.
Action: Ensure there is no problem with the sample tube or analysis bath.

10850 | Maximum target pressure exceeded in sample port [*n*] (target pressure: [*n,n*]**,** [*n*]**:** [*n,n*]**, max instrument manifold pressure: [*n,n*], *gas:*** [*n*]**, max gas manifold pressure: [*n,n*], max transducer pressure:** [*n,n*]**).**

Cause: A target pressure was requested that exceeds the maximum allowed. The maximum pressure may be based on saturation pressure of the gas at the temperature of the gas source, the manifold, the sample, or ambient temperature.
Action: Review the maximum allowable pressures in the error message and the analysis conditions to identify and correct the problem before repeating the analysis.

10860 | Psat gas [*n*] is not condensing. (pressure: [*n,n*] maximum manifold pressure: [*n,n*]).

Cause: The Psat gas is not condensing.
Action: Review the analysis parameters, gas connections, and analysis bath.

10870 | Adsorptive [*n*] is not condensing. (pressure: [*n,n*] maximum manifold pressure: [*n,n*]).

Cause: The adsorptive gas is not condensing.
Action: Review the analysis parameters, gas connections, and analysis bath.

10880 | Zeroing of the transducers failed.

Cause: The transducers failed to zero out.
Action: Contact your Micromeritics service representative.

10890 | Purification of the adsorptive [*n*] failed at [*n,n*] (charge pressure: [*n,n*]**, minimum allowed: [*n,n*]).**

Cause: The adsorptive gas failed to purify at the specified pressure.
Action: Check the gas connection.

10902 | Sample pressure on sample port [*n*] **([*n,n*]) is below the minimum desorption pressure (**[*n,n*]**).**

Cause: A target pressure for desorption was requested that is below the minimum desorption pressure allowed.
Action: Review the analysis conditions to remove the disallowed pressure.

10950 | Power supply voltage error detected (type: [*n*]**, voltage: [*n*] volts, nominal: [*n*] volts).**

Cause: There is a problem with the power supply voltage.
Action: Contact your Micromeritics service representative.

11002 | Manifold heater temperature error (measurements: [*n*], mean: [*n*], std dev:[*n*], min: [*n*], **max:** [*n*], **since: [*n*]).**

Cause: An error was detected with the manifold heater temperature.
Action: Check the top cover of the instrument. Ensure it is installed and sealed properly.

E FREE SPACE CORRECTION

Free space is that volume of the sample tube which is unoccupied by the sample. The quantity of gas dosed into the sample tube is calculated from the difference in pressures in the manifold before and after the dose is delivered. The quantity of gas adsorbed by the sample is calculated by subtracting the quantity of gas remaining in the free space of the sample tube after equilibrium is established from the quantity of gas originally dosed into the sample tube. Free space must be determined accurately to obtain a precise value for quantity adsorbed.

Static-volumetric systems consist basically of a gas manifold joined to a sample tube by an isolation valve. The manifold section has connections for an absolute pressure transducer, a temperature gauge, and a vacuum system. It also has inlets for the adsorptive gas and helium. A dewar flask containing a cryogenic liquid (usually LN_2 at approximately 77 K) is situated so that it can be raised to immerse most of the sample tube. Two temperature zones exist within the sample tube when immersed in the cryogenic bath: a warm zone (the volume above the liquid level and near ambient temperature) and a cold zone (the volume below the liquid level at cryogenic temperature). Not only must the total free space volume be determined, but it also is necessary to determine the quantity of gas residing within the “cold” zone since a nonideality correction must be applied to only that quantity of gas.

The total quantity of gas in the partly immersed sample holder cannot simply be determined using n = PV/RT because temperature is not constant over the total volume, but instead is distributed as two temperature zones with a steep temperature gradient between them. A convenient method for resolving this problem is to derive two factors which, for the existing temperature profile, can be multiplied by the prevailing pressure to reveal the molar volume of gas contained in the cold zone and the total quantity residing in the free volume of the immersed sample holder (the cold free space).

The analyzer provides the following methods for free space determination:

- Measure
- Calculate
- Enter

MEASURE

Generally, this method, although requiring a little more time (approximately 10 minutes), is the most preferred one for determining free space. It is simple, automatic, requires very little information, and essentially is error-proof. With this method, the instrument first evacuates the manifold and sample tube (containing sample), then isolates the sample tube by closing the valve. Then the manifold is charged with helium, the pressure measured, and the valve opened allowing the helium to expand into the sample tube at ambient temperature. Again the pressure is measured.

The dewar is raised and the sample tube is cooled to cryogenic temperature. Again pressure drops; when pressure has equilibrated, the value is recorded. Warm and cold free spaces are calculated from (1) system volume, (2) system, ambient, and bath temperatures, and (3) measured pressures. From these, the value of the portion of cold free space at cryogenic temperature which requires correction for nonideality can be determined.

This method may be undesirable if:

- Helium is unavailable; free space determination by the analyzer requires the use of helium
- Analysis speed is a major factor; a helium free space measurement of 10 to 15 minutes is required
- The sample tends to absorb and retain helium for a prolonged period of time or if it adsorbs helium

CALCULATE

This method is the most rapid and efficient way of compensating for free space. Ensure the following is accomplished:

- Perform a blank analysis on the sample tube
- Load the blank analysis file data into the sample tube file
- Enter the analysis bath temperature (found on the *p° and Temperature* window)
- Enter the sample mass and density (found on the *Sample Description* tab)

ENTER

This method allows for entering predetermined values for the warm and cold free spaces. The values to enter may be obtained in one of two ways:

- A pre-analysis free space calibration of the sample tube containing sample
- The total free space of an empty sample tube is measured and the displacement of the sample calculated from its mass and density and subtracted from the total free space

In either procedure, ensure that the level (or, in cases where the Isothermal Jacket is used, the effective level) of the cryogen bath on the sample tube is the same when the analysis is performed as it was when gathering data for free space calculations.

F GAS CONVERSION CONSTANTS

The ChemiSorb analyzer uses Mass Flow Controllers (MFCs) to control the flow of gases. These MFCs require a conversion constant for each gas or gas mixture, to compensate for variations in gas flows resulting from variations in the properties of gases. A default gas table containing MFC conversion constants is included on the *Options* menu. The following table provides a more complete list of gases and their conversion constants.

To determine a new conversion constant when using a unique gas mixture. See ***Gas Mixtures Worksheet on page L - 1***.

Gas Conversion Constants for the MFCs

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Acetylene	C_2H_2	0.6535
Air (mixture)		0.9901
Allene	C_3H_4	0.4752
Ammonia	NH3	0.7822
Argon	Ar	1.3861
Arsine	A_5H_3	0.7525
Boron Trichloride	BCl_3	0.4356
Boron Trifluoride	BF_3	0.5743
Bromine Pentafluoride	BrF_5	0.2871
Bromine Trifluoride	BrF_3	0.4356
Butane	C_4H_{10}	0.2871
Butene	C_4H_8	0.3267
Carbon Dioxide	CO_2	0.7723
Carbon Monoxide	CO	0.9802
Carbon Tetracloride	CCl_4	0.3465
Carbon Tetrafluoride	CF_4	0.4752
Carbonyl Fluoride	COF_2	0.2673
Carbonyl Sulfide	COS	0.6733
Chlorine	Cl_2	0.8218
Chloroform	$CHCl_3$	0.4356

Gas Conversion Constants for the MFCs (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Chlorine Trifluoride	ClF_3	0.4257
Cyanogen	C_2N_2	0.4950
Cyclopropane	C_3H_6	0.5050
Deuterium	D_2	0.9901
Diborane	B_2H_6	0.5446
Dichlorosilane	SiH_2Cl_2	0.4356
Dimethylamine	$(CH_3)_2NH$	0.6634
Dimethylether	$(CH_3)_2O$	0.5842
Ethane	C_2H_6	0.5446
Ethyl Chloride	C_2H_5Cl	0.2871
Ethylene	C_2H_4	0.6139
Ethylene Oxide	C_2H_4O	0.5842
Fluorine	F_2	0.9208
Fluroform	CHF_3	0.5644
Freon 11	CCl_3F	0.3762
Freon 12	CCl_3F_2	0.3861
Freon 13	$CClF_3$	0.4257
Freon 13 B1	$CBrF_3$	0.4059
Freon 14	CF_4	0.4703
Freon 21	$CHCl_2F$	0.4554
Freon 22	$CHClF_2$	0.5050
Freon 23	CHF_3	0.5644
Freon 113	$CCl_2F\text{-}CClF_2$	0.2277
Freon 114	$CCl_2F_4\text{-}CClF_2$	0.2554
Freon 115	$CClF_2\text{-}CF_3$	0.2713
Freon 116	$CF_3\text{-}CF_3$	0.2277

Gas Conversion Constants for the MFCs (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Germane	GeH_4	0.6436
Helium	He	1.3762
Hexamethyldisizane	HMDS	0.1386
Hydrogen	H_2	1.0000
Hydrogen Bromide	HBr	0.9703
Hydrogen Chloride (Dry)	HCl	0.9802
Hydrogen Fluoride	HF	0.9901
Hydrogen Iodide	HI	0.9505
Hydrogen Selenide	H_2Se	0.8317
Hydrogen Sulfide	H_2S	0.8416
Isobutane	C_4H_{10}	0.3069
Isobutylene	C_4H_8	0.3366
Krypton	Kr	1.3762
Methane	CH_4	0.8020
Methylamine	CH_3NH_2	0.5644
Methyl Bromide	CH_3Br	0.6436
Methyl Chloride	CH_3Cl	0.6832
Methyl Fluoride	CH_3F	0.7525
Methyl Mercaptan	CH_4S	0.5842
Neon	Ne	1.3861
Nitric Oxide	NO	0.9901
Nitrogen	N_2	0.9950
Nitrogen Dioxide	NO_2	0.7525
Nitrogen Trioxide	N_2O3	0.4356
Nitrogen Trifluoride	NF_3	0.5446

Gas Conversion Constants for the MFCs (continued)

Gas	Symbol	MFC Conversion Constant (H_2 = 1.000)
Nitrous Oxide	N_2O	0.7426
Oxygen	O_2	0.9802
Ozone	O_3	0.7327
Pentaborane	B_5Hg	0.2871
n Pentane	C_5H_{12}	0.2376
Perchloryl Fluoride	ClO_3F	0.4455
Phosgene	$COCl_2$	0.5050
Phosphine	PH_3	0.7822
Phosphorous Pentafluoride	PF_5	0.3465
Propane	C_3H_8	0.3861
Propylene (Propene)	C_3H_6	0.4653
Silane	SiH_4	0.6733
Silicon Tetrachloride	$SiCl_4$	0.3168
Silicon Tetrafluoride	SiF_4	0.3960
Sulfur Dioxide	SO_2	0.7228
Sulfur Hexafluoride	SF_6	0.2970
Trichlorosilane	Cl_3HSi	0.3267
Trimethylamine	$(CH_3)_3N$	0.3168
Tungsten Hexafluoride	WF_6	0.2871
Uranium Hexafluoride	UF_6	0.2178
Vinyl Bromide	C_2H_3Br	0.5248
Vinyl Chloride	C_2H_3Cl	0.5347
Vinyl Fluoride	C_2H_3F	0.5743
Xenon	Xe	1.3762

G MAINTAINING HIGH PURITY GASES

The analysis system was designed to accurately measure the surface area of all types of materials. It is important that the gases (especially krypton) used for these measurements be of highest purity, especially when analyzing low surface area samples. Three ways to ensure high purity gases are to always maintain:

- thoroughly purged gas pressure regulators
- non-permeable gas lines
- leak-free connections

Impure gas is strongly indicated, for example, if a series of measurements on a low surface area material yields decreasing specific surface areas with decreasing quantities of sample. The analyzer uses very small amounts of helium; therefore any residual air in the regulator can distort results of subsequent analyses for quite some time.

Micromeritics offers the following suggestions to assist in maintaining high purity gases (particularly helium):

- Use metal gas lines only
- Remove trapped air from the regulator and gas lines

USING METAL GAS LINES

Always use metal gas lines which have been carefully cleaned of any oils and greases used in the manufacturing process. Do not use plastic or rubber gas lines. When these types of permeable, nonmetallic gas lines are used with helium, contaminants accumulate at a much faster rate. This causes errors in analysis results and can also contaminate a clean sample.

REMOVING TRAPPED AIR

When connecting the regulator to the gas cylinder, air is unavoidably trapped on the high- and low-pressure sides of the regulator, as well as in the gas lines. Remove as much of this air as is possible ***before*** opening the gas cylinder valve. If this air is allowed to remain in the regulator, it will mix with the helium and cause inaccurate results in subsequent analyses. Or if the valve is open for any length of time, the air trapped on the high pressure side may diffuse back into the gas cylinder and contaminate its entire contents.

There are two methods for removing trapped air from the regulator lines: the Purge Method and the Evacuation Method.

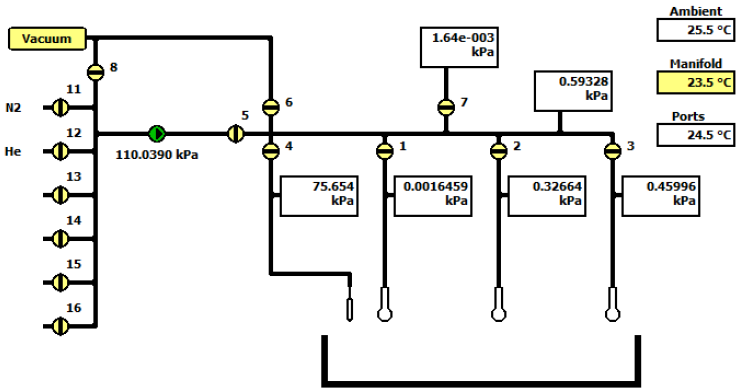
PURGE METHOD

This is the preferred method for removing trapped air.

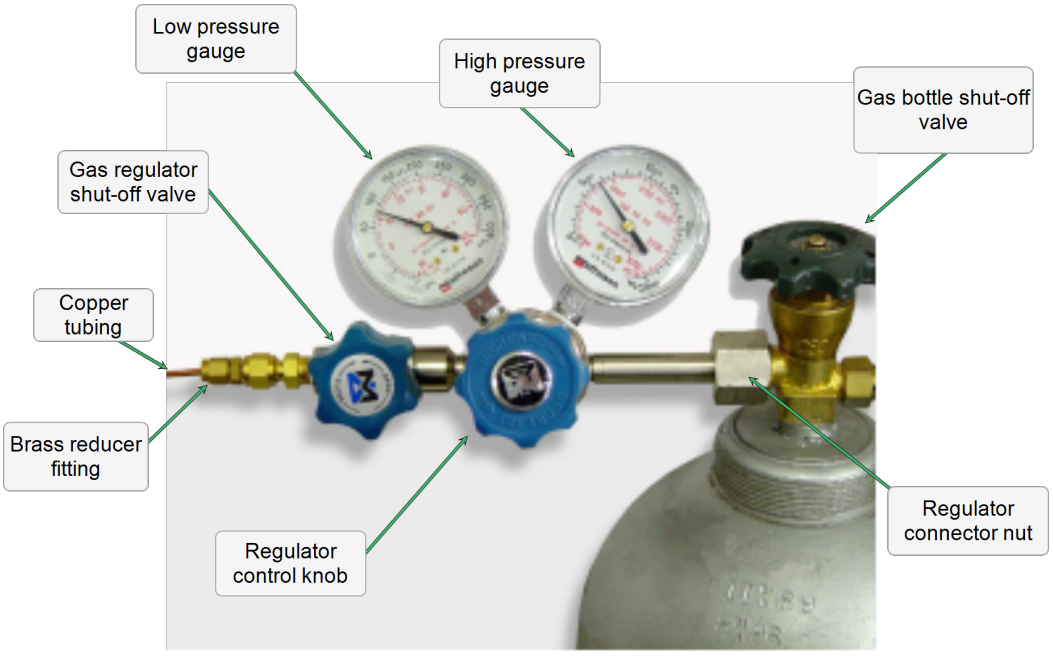
1. Go to ***Unit > Enable Manual Control*** (if the analyzer schematic is not displayed, go to ***Unit > Show Instrument Schematic***).

If multiple instruments are installed, choose the correct *Unit* menu.

2. Close all valves by right clicking on each valve, then click **Close**.



3. Open the regulator shutoff valve.
4. Open the gas cylinder valve ***briefly*** and allow the regulator to be charged with gas until the high-pressure gauge reads just over half the tank pressure, then quickly close the valve.



5. Using the Pressure Control knob, set the output pressure (gas cylinder pressure gauge) to 15 psig.
6. Loosen the fitting at the helium inlet (on the rear panel of the analyzer) until the low pressure side drops to approximately 3 psig (0.02 MPa), then tighten the fitting.
7. Repeat steps 4, 5, and 6 three times.
8. Briefly open the gas cylinder valve, then, using the Pressure Control knob, reset the regulator output pressure to 15 psig.
9. After the pressure has stabilized (indicating there are no leaks), open the gas cylinder valve.

EVACUATION METHOD

To use this method, the gas tank must be within 10 feet of the instrument.

1. Do one of the following:

If...	Then...
The regulator has not been filled with gas and the gas line is attached to the instrument:	Close the gas cylinder valve.
	Open the regulator shutoff valve.
The regulator is filled with gas:	Close the gas cylinder valve.
	Open the regulator shutoff valve.
	Loosen the helium inlet fitting (or nut) on the rear panel of the instrument.
	Allow all of the gas in the regulator to expel from the line (pressure reading will be zero).
	Retighten the helium inlet fitting (or nut).

2. Go to ***Unit > Enable manual control*** (if the instrument schematic is not displayed, go to ***Unit > Show instrument schematic***).

If multiple instruments are installed, make sure to choose the correct Unit menu.

3. Close all valves, then open valves 6, 7, and 10.
4. Allow evacuation to continue for 20 minutes. This pulls a vacuum on the helium line to the gas cylinder. The manifold pressure transducer should fall close to zero.

Allow evacuation for a full 20 minutes. If evacuation time is too short, trapped air may remain in the lines.

5. Close valves 6, 7, and 10.

H PYTHON MODULE - ADVANCED REPORTS

The mic Python module is automatically imported when running a user supplied script. The module provides access to primary and overlay isotherm data and provides support for summary, tabular, and graphical reports.

- **Summary reports.** Consist of summary sections, each containing a two-column table of label and value pairs. Summary reports are created with the *mic.summary* call.
- **Tabular reports.** Consist of one or more tables each containing one or more labeled columns of data.Tabular reports are created with the *mic.table* call.
- **Graphical reports**. Consist of a single graph with one or more curves on one or two y-axes. Graphical reports are created with the *mic.graph* call.

Calls for accessing the sample file data can be found in the *Mic Module Python Calls* section of this appendix. More advanced example python scripts are included in the analyzer software. Application specific discussions can be found on http://www.micro-report.com

The examples in this topic are also included as a part of the Micromeritics installation process and are located in the *Scripts* sub-directory.

RUN A SCRIPT

1. Open a sample file with a *Complete* file status.
2. Select *Advanced* in the drop-down list at the bottom of the window.
3. Select the *Report Options* tab.
4. Highlight *Advanced* in the *Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add**. Locate and select one or more python scripts then click **Select**. The selected scripts become a part of the drop-down list in the *Available Scripts* section of the *Advanced Report Options* window.
6. In the *Selected Reports* section, select up to five Advanced reports in the drop-down lists. Use the **Pressures** button to include or exclude available pressures in the report.
7. Click **OK** to close the window.
8. Click **Preview** on the *Report Options* tab to view all reports selected in the previous window.

EDIT A SCRIPT

When a script is added, the code is stored within the application. If the script changes outside of the application, the script file will have to be re-added to the Advanced Report Options window for the changes to take affect.

Field or Button	Description
Add	Adds one or more scripts to the *Available Scripts* box. The added scripts then become available as options in the *Selected Reports* section.
Edit	Edits the script stored within the application but does not affect the original .py text file.
Overlay samples	Select to enable the overlay sample files process.
Pressures	Select to include or exclude pressures from the report. • **Calculation pressure range.**Enter the minimum and maximum pressures to be used in the pressure table. • **Cancel**. Discards any changes or cancels the current process. • **Exclude All.**Select to exclude all pressure points in the table. • **Include All.**Select to include all pressure points in the table. • **OK.**Saves and closes the active window.
Remove	Removes the script from the *Available Scripts* box but does not affect original .py text file
Replace	Replaces the contents of the selected script however, the script name remains the same.

REMOVE A SCRIPT

Select the script in the *Available Scripts* box then click **Remove**. The script is removed from the application however, the original .py text file is not affected.

SUMMARY REPORT

This script produces a summary report with two summaries:

```
import mic
mic.summary( "My Summaries" )

mic.summary.add( "Summary A",
                 ["label 1:", "label 2:", "label 3:"],
                 ["val1", "val2", "val3"] )


mic.summary.add( "Summary B",
                 ["label 4:", "label 5:", "label 6:"],
                 ["val4", "val5", "val6"] )
```

The result is:

Summary A
label 1: val1
label 2: val2
label 3: val3

Summary B
label 4: val4
label 5: val5
label 6: val6

TABULAR REPORT

If more than one column is required, the call *mic.table* is employed. This script produces a tabular report consisting of two tables. **NOTE**: This script uses the Python package "numpy" and c-style formatting of the numerical values.

```
import mic
import numpy as np

mic.table("My Tables")

mic.table.addtable( "My set A" )
mic.table.addcolumn( "x", ["1.0", "2.0", "3.0"] )
mic.table.addcolumn( "y", ["0.5", "1.0", "1.5"] )

x1 = 0.2
x2 = 0.5
x3 = 3.14159/2
mic.table.addtable( "My set B" )
mic.table.addcolumn( "x", ["%8.3f" % x1,
                           "%8.3f" % x2,
                           "%8.3f" % x3 ] )

mic.table.addcolumn( "sin(x)", ["%8.3f" % np.sin(x1),
                                "%8.3f" % np.sin(x2),
                                "%8.3f" % np.sin(x3)] )


mic.table.addcolumn( "cos(x)", ["%8.3f" % np.cos(x1),
                                "%8.3f" % np.cos(x2),
                                "%8.3f" % np.cos(x3)] )
```

The result is:

My set A

x	y
1.0	0.5
2.0	1.0
3.0	1.5

My set B

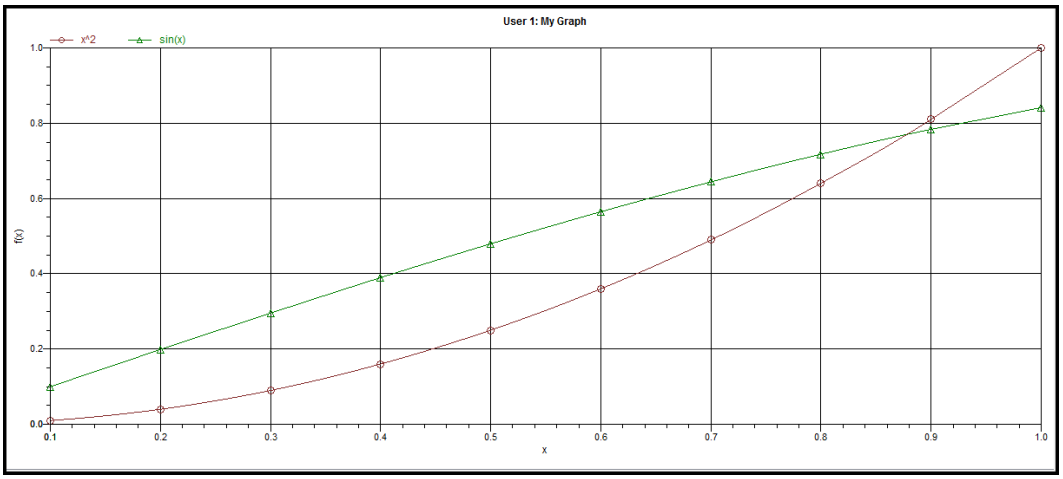
x	sin(x)	cos(x)
0.200	0.199	0.980
0.500	0.479	0.878
1.571	1.000	0.000

GRAPHIC REPORT

This script is an example of the mic module producing a graph with two curves:

```
import mic
import numpy as np

mic.graph( 'My Graph', 'x', 'f(x)' )

myx = np.array( [0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0 ] )
mic.graph.add( 'x^2', myx, myx*myx, marker='o' )
mic.graph.add( 'sin(x)', myx, np.sin(myx), marker='^' )
```

The results are:



ACQUIRE BASIC INFORMATION FOR PHYSISORPTION

This script produces a graph of the adsorption and desorption isotherms for both relative and absolute pressure, and prints summaries of the sample information and the adsorptive properties.

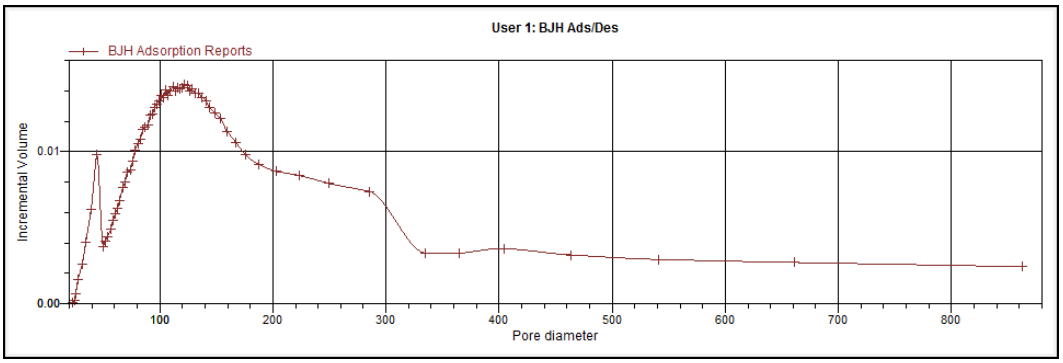
To acquire the adsorption isotherm and other basic information about the sample being edited, the calls *mic.isotherm*, *mic.sample_information* and *mic.adsorptive_data* are applied.

Note the calls to *mic.isotherm* and *mic.adsorptive_data* above are each returning results as a list with elements of varying return type.

```
import mic

prel, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('rel')
mic.graph( 'Graphical Report 1', 'Rel. Press', 'Quantity Adsorbed' )
mic.graph.add( 'Sample isotherm', prel, qads )

pabs, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('abs')

mic.graph( 'Graphical Report 2' 'Abs. Press', 'Quantity Adsorbed')
mic.graph.add('Sample Isotherm', pabs, qads)

mass = mic.sample_information('sample mass' )
Tanl = mic.sample_information('analysis temperature' )
dens = mic.sample_information('sample density')

mic.summary( "Sample Information" )
mic.summary.add( "Sample Information:",
                 [ "Number of adsorption points:",
                   "Warm Free space:",
                   "Cold Free space:" ,
                    "Sample mass (g):",
                    "Description:",
                    "Analysis Temp:",
                    "Sample Density (g/cm^3):" ],
                 [ "%8.3f" % n_ads,
                   "%8.3f" % warm_fs,
                   "%8.3f" % cold_fs,
                   "%8.3f" % mass,
                   desc,
                   "%8.3f" % Tanl,
                   "%8.3f" % dens ] )

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()

mic.summary.add( "Adsorptive Data",
```

```
                    [ "Cross Sectional Area",
                      "Hard Sphere Diameter",
                      "Density Conversion Factor",
                      "Molecular Weight",
                      "Analysis gas"],
                    [ "%8.3f" % csa,
                      "%8.3f" % hsd,
                      "%8.3f" % dcf,
                      "%8.3f" % mol_weight,
                      analysis_gas  ]  )
```

The result is:



ACQUIRE BASIC INFORMATION FOR CHEMISORPTION

This script produces a graph of the primary, repeat, and difference isotherms; and prints summaries of the sample information and the adsorptive properties.

To acquire the adsorption isotherm and other basic information about the sample being edited, the calls *mic_chem.isotherm*, *mic.sample_information* and *mic.adsorptive_data* are applied.

Note the calls to *mic_chem.isotherm* and *mic.adsorptive_data* above are each returning results as a list with elements of varying return type.

```
import mic

p_primary,    q_primary    = mic.chem_isotherm('primary')
p_repeat,     q_repeat     = mic.chem_isotherm('repeat')
p_difference, q_difference = mic.chem_isotherm('difference')
mic.graph( 'Graphical Report 1', 'Abs. Press', 'Quantity Adsorbed')
mic.graph.add('Primary', p_primary   , q_primary)
mic.graph.add('Repeat', p_repeat    , q_repeat)
mic.graph.add('Difference', p_difference, q_difference)

mic.summary( "Sample Information" )
mic.summary.add( "Sample Information:",
                 [ "Ambient Free space (cm^3):",
                   "Analysis Free space (cm^3):" ,
                    "Sample mass (g):",
                    "Description:",
                    "Analysis Temp (K):",
                    "Sample Density (g/cm^3):" ],
                 [ "%8.3f" % mic.sample_information('ambient freespace'),
                   "%8.3f" % mic.sample_information('analysis freespace'),
                   "%8.3f" % mic.sample_information('sample mass'),
                   mic.sample_information('sample description'),
                   "%8.3f" % mic.sample_information('analysis temperature'),
                   "%8.3f" % mic.sample_information('sample density') ] )

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()

mic.summary.add( "Adsorptive Data",
                 [ "Cross Sectional Area",
                   "Hard Sphere Diameter",
                   "Density Conversion Factor",
                   "Molecular Weight",
                   "Analysis gas"],
                 [ "%8.3f" % csa,
                   "%8.3f" % hsd,
```

```
                    "%8.3f" % dcf,
                    "%8.3f" % mol_weight,
                    analysis_gas  ]  )
```

ACQUIRE REPORT RESULTS

Sample file report results may be accessed using the *mic.report* call. This script prints a summary of the results of the *t*-plot and BET reports.

```
import mic

sa   = mic.report("bet",  "surface area")
c    = mic.report("bet",  "bet constant")
vm   = mic.report("bet",  "monolayer capacity")
esa  = mic.report("tplot", "external surface area")
vol  = mic.report("tplot", "micropore volume")

mic.summary( "BET and T-plot Results" )

mic.summary.add( "Report Results",
                 [ "bet surface area",
                   "bet constant",
                   "bety 6" ,
                    "tplot external surface area",
                    "tplot micropore volume"],
                 [ "%10.5f" % sa,
                   "%10.5f" % c,
                   "%10.5f" % vm,
                   "%10.5f" % esa,
                   "%10.5f" % vol ] )
```

Acquiring the results from a pore-distribution report such as the BJH method is done in a similar way as in the previous script except the return values from the *mic.report* call are slightly different since they involve lists of data. For example,

```
import mic
xdat, ydat, desc = mic.report('bjhads' ,'incremental distribution' )
mic.graph( 'BJH Ads/Des', 'Pore diameter', 'Incremental Volume' )
mic.graph.add( desc, xdat, ydat )
```

See the *Mic Module Python Calls* section for a more complete description of the usage and scope of the *mic.report* call.

The result is:

Report Results

bet surface area	796.36286
bet constant	137786.85871
bet monolayer capacity	182.96348
tplot external surface area	416.38843
tplot micropore volume	0.17931

ACQUIRE OVERLAY SAMPLE DATA

The call to obtain overlay sample data is similar to the calls for the primary sample. This script involves two overlay sample files.

The calls to obtain adsorptive data and report results for an overlay sample file using *mic.report* and *mic.adsorptive_data* have a very similar interface as the *mic.overlay call*, and a summary of their usage is shown in the example in this topic.

```
import mic

p, q, n, fsw, fsc, mass, desc = mic.isotherm('rel')
p1, q1, n1, fsw1, fsc1, mass1, desc1 = mic.overlay( 1, 'rel')
p2, q2, n2, fsw2, fsc2, mass2, desc2 = mic.overlay( 2, 'rel')

mic.graph( 'Three Sample Isotherms',
           'Rel. Press',
           'Quantity Adsorbed (cm^3/g)' )

mic.graph.add( 'Primary Isotherm ', p, q )
mic.graph.add( 'Overlay Isotherm 1', p1, q1 )
mic.graph.add( 'Overlay Isotherm 2', p2, q2 )

mic.summary( "A summary report" )

mic.summary.add( "Two samples",
                 [ "Primary Sample:",
                   "Overlay Sample 1:",
                   "Overlay Sample 2:" ],
                 [ desc,
                   desc1,
                   desc2] )
```

To enable the use of overlay data in the Advanced reports, the following two actions must be taken prior to running the script. Instructions for each of the following actions are provided below.

- Sample files to overlay must be selected, and
- The *Overlay samples* checkbox on the *Advanced Report Options* window must be selected

ACQUIRE OVERLAY SAMPLE DATA FOR CHEMISORPTION

The call to obtain overlay sample data is similar to the calls for the primary sample. This script involves two overlay sample files.

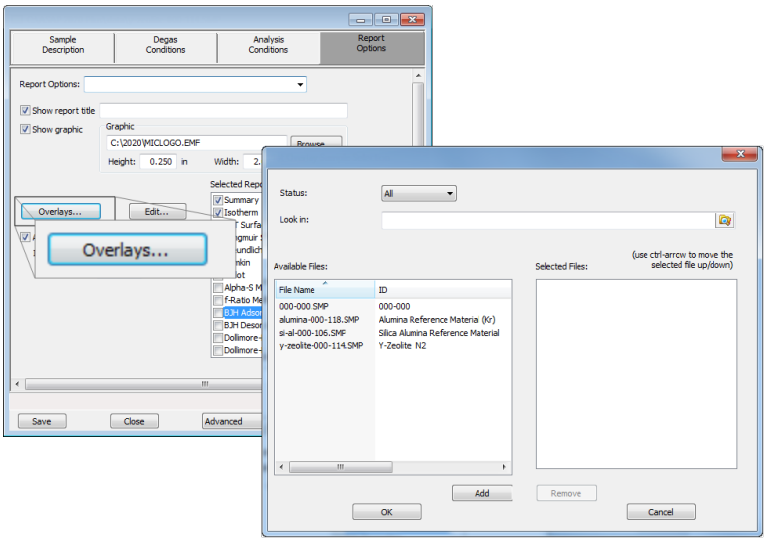
The calls to obtain adsorptive data and report results for an overlay sample file using *mic.report* and *mic.adsorptive_data* have a very similar interface as the *mic.chem.overlay call*, and a summary of their usage is shown in the example in this topic.

```
import mic


p0, q0    = mic.chem_isotherm('primary')
p0r,q0r = mic.chem_isotherm('repeat')
p1, q1 = mic.chem_overlay(1,'primary')
p1r, q1r = mic.chem_overlay(1,'repeat')
p2, q2 = mic.chem_overlay(2,'primary')
p2r, q2r = mic.chem_overlay(2,'repeat')
mic.graph( 'Graphical Report 1', 'Abs. Press', 'Quantity Adsorbed')
mic.graph.add('prim 0',p0,q0)
mic.graph.add('rep 0',p0r, q0r)
mic.graph.add('prim 1',p1,q1)
mic.graph.add('rep 1',p1r, q1r)
mic.graph.add('prim 2',p2,q2)
mic.graph.add('rep 2',p2r, q2r)

mic.summary( "A summary report" )

mic.summary.add( "Sample and Two Overlays",
                 [ "Primary Sample:",
                   "Overlay Sample 1:",
                   "Overlay Sample 2:" ],
                 [  mic.sample_information('sample description'),
                    mic.sample_information('sample description',1),
                    mic.sample_information('sample description',2)  ] )
```

ENABLE THE USE OF OVERLAY DATA

1. On the *Report Options* tab, click **Overlays**.

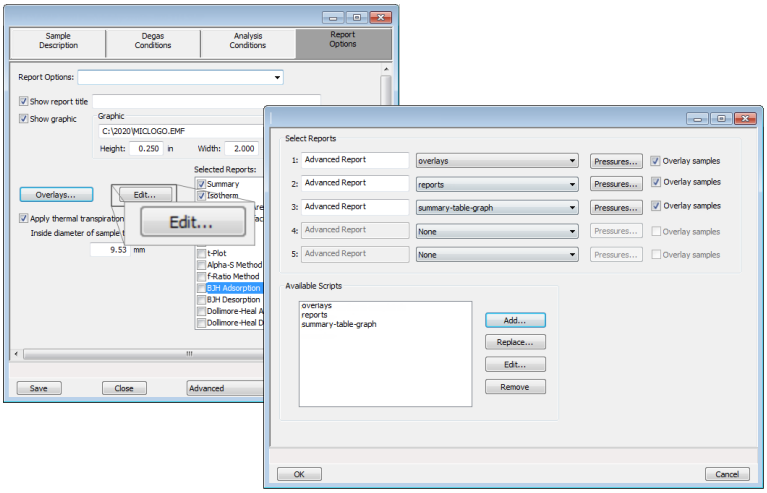
2. On the *Plot Overlay Sample Selection* window, use one of the following options to move up to 25 files from the *Available Files* box to the *Selected Files* box:



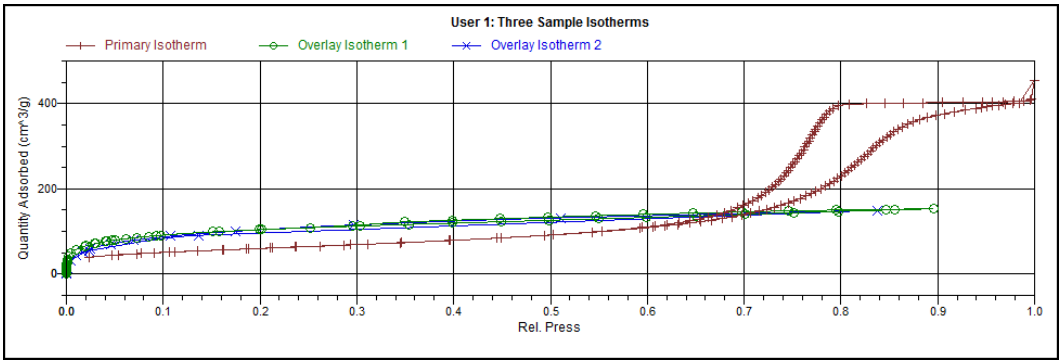
   - Double click a file name in the *Available Files* box to move the file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, double click the file name in the *Selected Files* box, or
   - Select a file name in the *Available Files* box. Click **Add** to move the selected file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, select a file name in the *Selected Files* box, then click **Remove**. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.

3. Click **OK**.

4. On the *Report Options* tab, highlight *Advanced* in the *Selected Reports* list box.
5. Click **Edit** to the left of the *Selected Reports* list box.
6. Select the *Overlay samples* checkbox to the right of the selected report.
7. Click **OK**.
8. Run the script using the instructions found in ***Run a Script on page H - 1***.



The results are:



Two samples

Primary Sample: 12 mm Tube N2 Silica-Alumina ADS-DES with FS
Overlay Sample 1: Activated Carbon Hexane Dosed from Port 3 - 2
Overlay Sample 2: Activated Carbon Tube C4 Butane Port 3

ACQUIRE METAL COMPOSITION DATA FOR CHEMISORPTION

The call to obtain information about active metals in a chemisorption sample is *mic.metal_composition*. Specifically, this call provides access to the data shown in the table of the *Active Metals* window. With no arguments specified, the call returns a list of all the active metals in the sample. When called with a metal specified, the method returns an associative array (python dictionary) of the metal's properties. With both the metal and property specified, the call returns the value for the specified metal property. The following example script illustrates these three usage patterns.

```
import mic
import pprint as pp

mnames = mic.metal_composition()
mic.summary( "Metal Composition:" + pp.pformat( mnames ) )

mprops = sorted( mic.metal_composition( mnames[0] ).items() )
mkeys = []
mvals = []
for k, v in mprops :
    if ( 'cross sectional area' in k ) :
        mkeys.append( k + ' (nm^2)' )
    elif ( 'atomic weight' in k ) :
        mkeys.append( k + ' (amu)' )
    elif ( 'density' in k ) :
        mkeys.append( k + ' (g/cm^3)' )
    else :
        mkeys.append( k )
    mvals.append("%8.3f" % v )
mic.summary.add( "Properties for " + mnames[0], mkeys, mvals)

mweights = []
for mname in mnames :
    mweights.append( "%8.3f" % mic.metal_composition(mname, 'atomic weight') )
mic.summary.add("Active Metals and Atomic Weight (amu)", mnames, mweights)
```

MIC MODULE PYTHON CALLS

TABLES

Available Mic Python calls for tables:

- Create a new tabular report
- Add a column
- Add a table

Create a New Tabular Report

```
mic.table( title='User Table' )

Keyword arguments:

  title --- the tabular report title (default = 'User Table')
```

Add a Table

This script adds a table to the last created tabular report:

```
mic.table.addtable( name )

Keyword arguments:

   name --- the table name
```

Add a Column

This script adds a column to the last created table:

```
mic.table.addcolumn( header, values )

Keyword arguments:

   header --- column header; must be a string (or convertible)
   values --- column values; must be a list of strings (or convertible)
```

SUMMARY REPORTS

Available Mic Python calls for summary reports:

- Add a summary section to the last created summary report
- Create a new summary report

Create a New Summary Report

```
mic.summary( title='User Summary' )

Keyword arguments:

   title --- the summary title
```

Add a Summary Section

This script adds a summary section to the last created summary report:

```
mic.summary.add( name, labels, values )

Keyword arguments:

   name   --- summary section name
   labels --- column of labels; must be a list of strings
              (or convertible) and the same length as values
   values --- column of values; must be a list of strings
              (or convertible) and the same length as labels
```

GRAPHIC REPORTS

Available Mic Python calls for graphic reports:

- Add a curve
- Add a curve using the second Y-axis
- Create a new graphic report

Create a New Graphical Report

```
mic.graph( title='User Graph', xlabel='X axis', ylabel='Y axis', yylabel='YY
axis', xlinear=True, ylinear=True, yylinear=True )

Keyword arguments:

   title    --- the graphical report title (default = 'User Graph')
   xlabel   --- x-axis label (default = 'X axis')
   ylabel   --- y-axis label (default = 'Y axis')
   yylabel  --- yy-axis label (default = 'YY axis')
   xlinear  --- x-axis linear scale; if false, use log scale
                (default = True)
   ylinear  --- y-axis linear scale; if false, use log scale
                (default = True)
   yylinear --- yy-axis linear scale; if false, use log scale
                (default = True)
```

Add a Curve

This script adds a curve to the last created graphical report:

```
mic.graph.add( name, x, y, yyaxis=False, color=None, linestyle='-', mark-
er='a', graphtype='both' )

Keyword arguments:

   name      --- the curve name
   x         --- list of x values; must be a list of floats
                 (or convertible) and the same length as y
   y         --- list of y values; must be a list of floats
                 (or convertible) and the same length as x
   yyaxis    --- place this curve on the yy-axis if True
                 otherwise place on the  y-axis (default = False)
   color     --- RGB color as an HTML hex string (e.g., '#4169e1')
                 or a three-element list or tuple (e.g., [65,105,225]);
                 if None, color is automatically selected (default = None)
   linestyle --- line style; (default = '-')
                    '-'        : solid
```

```
                    '--'       : dash
                    '.'        : dot
                    '-.'       : dash dot
                    '-..'      : dash dot dot
marker    --- marker shape; (default = 'a')
                 '+'        : plus
                 'o' or '0' : circle
                 'x'        : cross
                 '^'        : up triangle
                 'v'        : down triangle
                 's'        : square
                 'd'        : diamond
                 '8'        : hourglass
                 '~'        : horizontal hourglass
                 '' or None : no marker
                 'a'        : automatically selected
graphtype --- graph type; (default = 'both')
                 'curve'  or 'c' : curve
                 'points' or 'p' : points
                 'both'   or 'b' : curve-and-points
                 'hist'   or 'h' : histogram
```

Add a Curve Using the Second Y-Axis

This script adds a curve to the last created graphical report using the second y-axis:

```
mic.graph.addyy( name, xx, yy )

    Add a curve to the last created graphical report using the second
    y-axis.  The arguments to this call are the same as to mic.graph.add
    with the argument
```

GET PRIMARY ISOTHERM DATA FOR CHEMISORPTION

```
mic.chem_isotherm( branch='primary' ) :
Get primary, repeat and difference isotherm data.

Keyword arguments:
  branch --- Specifies which analysis to get isotherm data;
             use 'primary' for the first analysis,
                 'repeat' for the repeat analysis
             and 'difference' for the difference of these two

Usage:

   p, q = mic.chem_isotherm('primary')
   p, q = mic.chem_isotherm('repeat')
   p, q = mic.chem_isotherm('difference')
```

```
   p    --- array of absolute pressures
   q    --- array of cumulative quantity adsorbed
```

GET PRIMARY ISOTHERM DATA

```
mic.overlay( overlay_number = 1, press_type='rel' )

Keyword arguments:

  overlay_number --- the overlay number (1 through 8; default = 1)
  press_type     --- the pressure basis; use 'rel' for relative pressure,
                     'abs' for absolute (default = 'rel')

Usage:

  p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.overlay(1, 'rel')


  p       --- array of pressure (relative or absolute);
              empty-array if overlay is unavailable
  qads    --- array of cumulative quantity adsorbed;
              empty-array if overlay is unavailable
  num_ads --- number of points in the adsorption curve;
              0 if overlay is unavailable
  warm_fs --- warm free-space; 0.0 if overlay is unavailable
  cold_fs --- cold free-space; 0.0 if overlay is unavailable
  mass    --- sample mass; 0.0 if overlay is unavailable
  desc    --- sample description; empty-string if
              overlay is unavailable
```

GET OVERLAY ISOTHERM DATA FOR CHEMISORPTION

```
mic chem_overlay( overlay_number = 1, branch='primary' ) :

Get overlay isotherm data.

Keyword arguments:

  overlay_number --- the overlay number (1 through 8; default = 1)

   branch --- Specifies which analysis to get isotherm data;
              use 'primary' for the first analysis,
                  'repeat' for the repeat analysis
              and 'difference' for the difference of these two

Usage:

  p, q = mic.chem_overlay(1, 'primary')
```

```
  p, q = mic.chem_overlay(1, 'repeat')
  p, q = mic.chem_overlay(1, 'difference')
  p    --- array of absolute pressures
  q    --- array of cumulative quantity adsorbed
```

GET OVERLAY ISOTHERM DATA

```
mic.overlay( overlay_number = 1, press_type='rel' )

Keyword arguments:

  overlay_number --- the overlay number (1 through 8; default = 1)
  press_type     --- the pressure basis; use 'rel' for relative pressure,
                     'abs' for absolute (default = 'rel')

Usage:

  p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.overlay(1, 'rel')


  p       --- array of pressure (relative or absolute);
              empty-array if overlay is unavailable
  qads    --- array of cumulative quantity adsorbed;
              empty-array if overlay is unavailable
  num_ads --- number of points in the adsorption curve;
              0 if overlay is unavailable
  warm_fs --- warm free-space; 0.0 if overlay is unavailable
  cold_fs --- cold free-space; 0.0 if overlay is unavailable
  mass    --- sample mass; 0.0 if overlay is unavailable
  desc    --- sample description; empty-string if
              overlay is unavailable
```

GET ADSORPTIVE DATA FOR EACH SAMPLE

```
mic.adsorptive_data( sample_number = 0 )

Keyword arguments:

  sample_number --- Identifier for the adsorptive data to retrieve
                    0           : the current sample file
                    1 through 8 : the corresponding overlay sample file

Usage:

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()
csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data(0)

  csa          --- cross sectional area (nm^2)
```

```
  hsd          --- hard sphere diameter (angstroms)
  dcf          --- density conversion factor (dimensionless)
  mol_weight   --- molecular weight
  analysis_gas --- mnemonic for the analysis gas species
                  (e.g., 'CO', 'H2')
```

GET SAMPLE INFORMATION ITEM

```
mic.sample_information( item, sample_number = 0 )

Keyword arguments:

  item          --- string identifying the item to be returned.
                    Accepted identifiers are

                    'sample mass'
                    'sample description'
                    'analysis temperature'  (degrees Kelvin)
                    'sample density'        ( g/cm^3 )

  sample_number --- Sample to retrieve (default = 0).
                    0           : the current sample file
                    1 through 8 : the corresponding overlay sample file

Usage:

  mass = sample_information('sample mass')
  mass = sample_information('sample mass',0)
```

GET REPORT RESULTS

This script gets report results for the indicted report and sample.

```
mic.report( report_name, result, sample_number = 0 )


Keyword arguments:

  sample_number --- Identifier for the sample data to retrieve
                    0           : the current sample file
                    1 through 8 : the corresponding overlay sample file

Usage:

  sa                      = mic.report( 'bet' , 'surface area' )
  porewidth, incvol, desc = mic.report( 'bjhads' ,
                                        'incremental distribution'  )
```

```
The available report keywords, result keywords and a corresponding
description of the result is listed in the table below:

Report keyword  Result keyword              Description
--------------  --------------              -----------
    bet         surface area               Surface area ( m^2/g )
    bet         bet constant               BET constant ( dimensionless )
   bet          monolayer capacity         Monolayer capacity ( cm^3/g )
   tplot        external surface area      External surface area (m^2/g)
   tplot        micropore volume           Micropore volume (cm^3/g)
   bjhads       incremental distribution   Incremental Distribution
   bjhdes       incremental distribution   Incremental Distribution
   dhads        incremental distribution   Incremental Distribution
   hk           incremental distribution   Incremental Distribution
   dft          incremental distribution   Incremental Distribution
   nldft        incremental distribution   Incremental Distribution

where the incremental pore distribution result above (for those
reports which return this) is a list with three components being,

porewidth --- array of pore dimension boundaries (angstroms);
              empty-array if unavailable.
incvol    --- array of incremental pore volumes (cm^3/g);
              empty-array if unavailable.
desc      --- Name of data set; empty-string if unavailable.
```

GET IMPORTED PORE DATA

```
mic.imported_pore_data( import_number = 1 )

Keyword arguments:

  import_number --- the import number (1 through 8)

Usage:

  xdat, ydat, desc = mic.imported_pore_data(1)


  xdat --- array of pore dimension boundaries (angstroms);
           empty-array if unavailable.
  ydat --- array of incremental pore volumes (cm^3/g);
           empty-array if unavailable.
  desc --- Name of data set; empty-string if unavailable.
```

GET METAL COMPOSITION FOR CHEMISORPTION

```
mic metal_composition( metal='' , metal_property='' , sample_number = 0 ) :

Get information about the active metals in this sample

Keyword arguments:

  metal           --- the metal to return information about
                      if '' or None, then return a list of the
                      active metals

  metal_property  --- the specific property to return information on
                      if '' or None, then return all the properties
                      for the specified metal (requires metal to be
                      specified)

  sample_number --- Identifier for the metal data to retrieve
                    0           : current sample file (default)
                    1 through 8 : corresponding overlay sample file

Usage:

  metal_list  = mic.metal_composition()
  copper_prop = mic.metal_composition( 'copper')
  copper_perc = mic.metal_composition( 'copper',
                                        'percent of sample mass' )

In the above first usage case, the list of active metals is returned.
In the above second usage case, a python dictionary type
is returned which includes all the properties of the metal
available and their corresponding values.  The last case returns
a single value (int, float, or string) for the specified property.

The metal_properity keywords which one can use are

  atomic weight
  oxygen atoms
  density
  percent of sample mass
  metal atoms
  cross sectional area
  percent reduced
  stoichiometry H2
  stoichiometry O2
  stoichiometry He

Or as just mentioned, one can make the call
```

```
metal_composition( metalname ) without any metal_property
keyword provided to see the whole dictionary.
```

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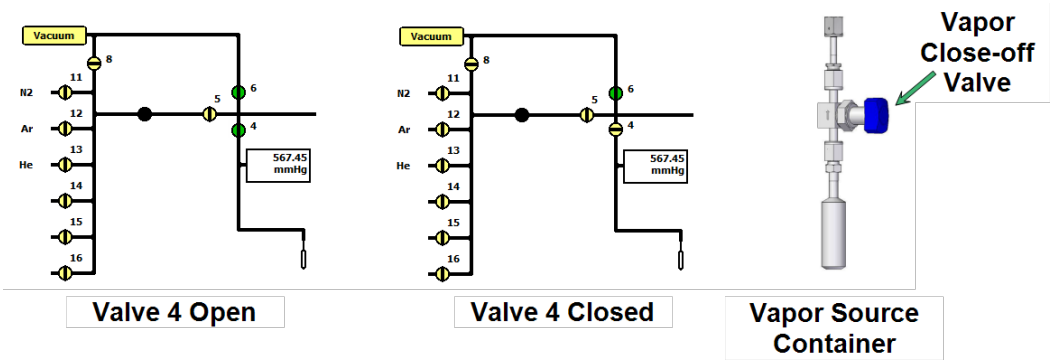
I VAPOR PURIFICATION

Physical adsorption of gases is generally used to calculate pore size and surface area of solid materials. Using vapor adsorptives allows the sorption capacity and isosteric heat of adsorption, the energy released as molecules adsorb on the sample surface, to be calculated by using isotherms collected at different temperatures with the same vapor and adsorbent. Heats of adsorption data are very useful in research applications. In the case of competitive adsorption, the vapor with the highest heat of adsorption will adsorb first and have the strongest interaction with the surface. However, if the heat of adsorption is too high, the molecule will be so strongly adsorbed that desorption or regeneration of the material may be difficult. In order to properly collect vapor isotherms, a purified vapor must be used. A liquid-filled reservoir is used as the source of the vapor. This procedure describes a freeze-thaw method to remove dissolved gases and air within the reservoir so the vapor purity is suitable for analysis on the 3Flex The general principles of this method could also apply to vapor purification on other gas adsorption instruments. The basic principle is to evacuate non-condensed species while the vapor reservoir is immersed in a cryogenic bath. At the pressures achieved during the purification process, nitrogen and oxygen are not condensed. The use of liquid nitrogen is limited to vapors that will not sublimate at cryogenic temperatures.



1. Fill a clean vapor reservoir with liquid. Fill halfway (10 ml) if using water. Fill with approximately 20 ml for other liquids.
2. Attach vapor reservoir to the 3Flex. See *Install a Vapor Source Container on page 11-14* in the 3Flex Operator Manual.
3. Firmly close the blue vapor close-off valve above the liquid-filled reservoir (blue valve on the vapor container).
4. On the instrument schematic, open valve 4 to evacuate the space above the vapor close-off valve.
5. Close instrument valve 4 on the instrument schematic, then open the vapor close-off valve on the reservoir. Allow the pressure to equilibrate in the vapor container.
6. Submerge the vapor reservoir in a cryogen bath. The use of liquid nitrogen as the cryogen should be limited to vapors with no sublimation pressure at cryogenic temperatures. Wait for the pressure in the vapor container to drop as low as possible. This may only be 150 torr for the first cycle but

should be near zero after two or three cycles. A lab jack is useful to hold and adjust the dewar filled with liquid nitrogen.

7. Once the vapor has condensed and has frozen, open valve 4 on the instrument schematic with the cryogen bath still in place and evacuate the vapor container (the vapor close-off valve should still be open). Pressures in the range of 10^{-4}-10^{-5} torr should be achievable.
8. Close valve 4 on the instrument schematic and remove the cryogen bath. Let the vapor container thaw. The vapor close-off valve should remain open during this step. To expedite the process, a warm water bath may be used temporarily to raise the temperature of the vapor container closer to the ambient temperature. However, a layer of ice formed on the reservoir could also create a barrier for heat transfer, so it is best to not introduce the water bath immediately. If pressure stabilizes near the calculated saturation pressure at room temperature, the vapor is free of impurities.
9. If the pressure does not stabilize around the calculated saturation pressure, repeat steps 3 through 8 until the pressure at the last step stabilizes near the calculated saturation pressure of the vapor. When the vapor is pure, the pressure should stabilize around the same value after each thawing cycle. Typically, three total purification cycles (two repeats) is sufficient.

J WETTED MATERIALS

Contact Micromeritics for assistance before running a gas such as ammonia or pyridine that is incompatible with some of the system components.

Wetted Materials

Material	Location
304 stainless steel , 403 stainless steel , Ceramic (Al2O3), Silicon, SiO2, Si3N4, Gold, Viton, Low out gassing epoxy resin	Vacuum gauge
316 Stainless Steel, Hastelloy C-22, 17-7 PH, 430SS, Nickel, Kalrez (FFKM)	MFC (chemi only)
Aluminum	NW/KF ring in vacuum line
Aluminum alloys, stainless steels, fluoroelastomer and nitrile O-rings, hydrocarbon lubricant, felt, rare earth magnets, silicon nitride, phenolic resin, carbon-fiber reinforced epoxy resin, fire retardant polypropylene, polyamide and PVC.	Turbo pump
Borosilicate Glass	Physisorption sample tubes, filler rods
Buna-N	• Gas inlet manifold, valve plungers, and O-rings • NW/KF ring in vacuum line
Buna-N, FKM (Viton), Stainless steel	TranSeal (optional)
Copper	• Gaskets in vacuum pump and vacuum gauge • Gas inlet lines
FFKM (Kalrez)	• Optional sample port O-rings • Servo valve
Gold plated copper	Turbo vacuum pump gasket and vacuum gauge
Inconel	Transducers
Kel-F (PCTFE)	• Valve seats analysis manifold • Valve seat exhaust valve (chemi only)
PCTFE (Kel-f), Buna-N, FKM (Viton), Stainless steel, Borosilicate Glass	Check seal (optional)
PTFE, FPM (fluroelastomer), Aluminum	Diaphragm roughing pump
Quartz	Chemisorption sample tubes, filler rods, wool filter discs
Stainless steel	• Manifolds, valve bodies, plumbing, VCR gaskets,

Wetted Materials (continued)

Material	Location
	port transducers, filler rods. • Gas inlet lines(optional) • Ferrules in exhaust line (chemi only)
Teflon (PTFE)	Ferrules in gas inlet manifold, P-zero tube
Viton type A (FKM)	Sample port O-rings

K WORKSHEETS

Worksheets in this section may be copied as needed.

- Input Gas Worksheet
- Sample Data Worksheet
- Valve Test Worksheet

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GAS MIXTURES WORKSHEET

A conversion constant for a mixture of gases can be determined easily using the conversion constants for each gas in the mixture.

1. Record the names and constants for each gas in the mixture.

Gases in the Mixture

Gas Name	Conversion Constant
1.	
2.	
3.	
4.	
5.	
6.	
7.	
8.	
9.	
10.	

2. Use the following formula to calculate the conversion constant for the gas mixture:

$$M = \frac{1}{\left[\frac{P_1}{F_1 \times 100}\right] + \left[\frac{P_2}{F_2 \times 100}\right] + \ldots + \left[\frac{P_n}{F_n \times 100}\right]}$$

- M = the mixture conversion constant
- P = the percentage of gas n in the mixture, expressed as a whole number (example: for 15%, use 15, not .15)
- F = the conversion constant (factor) for gas n

3. Enter the gas mixture in the *Gas Defaults* table; use M as the conversion constant.
   - Mixture name:_____
   - Constant name: _____

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SAMPLE DATA WORKSHEET FOR GAS ADSORPTION

Sample Tube Identification:_____

Sample Data (g)			
Record all values in grams			
	Before Degas	After Degas	After Analysis
A. Mass for empty sample tube set:			
B. Sample tube set plus sample mass:			
C. Sample mass (B - A):			

Degas Information	
Degas apparatus:	
Temperature (ºC):	
Time (hours):	
Actual time started:	
Actual time finished:	

Degas notes: _____

_____

_____

_____

_____

_____

_____

Copy and use this form as needed

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SMART VACPREP OPERATOR MANUAL

The Smart VacPrep Operator Manual is applicable only if the optional Smart VacPrep degasser is installed.

SMART VACPREP

INTELLIGENT VACUUM SAMPLE PREPARATION SYSTEM

MICROMERITICS

OPERATOR MANUAL

067-42800-011

Dec 2013

ABOUT THIS MANUAL

The following icons and format may be used throughout this manual to identify notes of importance, warnings, and cautions.

NOTE - Notes contain important information pertinent to the subject matter.

CAUTION - Cautions contain information to help prevent actions that may damage the analyzer or components.

WARNING - Warnings contain information to help prevent actions that may cause personal injury.

For Chemisorption - Indicates the marked item is applicable to chemisorption analyzers only.

For Physisorption - Indicates the marked item is applicable to physisorption analyzers only.

Field Labels and Screen Titles

Labels and Buttons	Description
Buttons (in the application)	Buttons in the application are represented as bold font weight and blue letters. Some examples are: **Save**, **Edit**, **Replace All**, etc.
Buttons (on the equipment)	Buttons on the equipment are represented as bold font and black letters. some examples are: **Start** or **Cancel**.
Field Labels	Field Labels are represented as italicized words. Some examples are: *Sample, Automatically Collected, Analysis Conditions*, etc
Keyboard Commands	Keyboard commands are represented as bold font weight and black letters. Some examples are: **F2**, **Alt+F4**, etc.
Menu Instructions	Menu instructions are represented as bold and italicized words. Some examples are: ***File > New Sample*** ***Reports > Start Report*** ***Unit [n] > Unit Configuration***
Screen Tabs	Screen Tabs are represented as italicized words. Some examples are*:* *Sample Description*, *Degas Conditions*, *Analysis Conditions*, and *Report Options*.
Screen Titles	Screen Titles are represented as italicized words. Some examples are: *Analysis Adsorptive Properties*, *Free Space*, *Sample Tube*, etc

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Table of Contents

1 About the Smart VacPrep

The Smart VacPrep is an advanced six-port degassing system that utilizes vacuum to degas samples by heating and evacuation. Each of the ports may be operated independently of one another. Samples may be added or removed from degas ports without disturbing the treatment of other samples undergoing preparation. When starting a new sample, ports already degassing samples are automatically suspended unless they are in the slow evacuation stage. When unrestricted evacuation is attained by the new samples, the previously suspended samples will automatically resume their evacuation. Degassing will automatically terminate when each sample has completed all programmed steps.

The Smart VacPrep remembers the previously selected set of degas conditions and allows them to be reused. However, the option of programming each port with a different set of parameters is available. A record of the sample preparation time and temperature can be recorded as part of the sample data file.

The degas program may be started or terminated by using the analyzer software on the computer attached to the analyzer or with push buttons on the front panel of the Smart VacPrep. The push button control on each port provides the ability to attach the sample tube with heating mantle, and immediately start degassing a sample.

There are two methods of starting the degas process from the analyzer software:

- **Start Degas** menu option. Accessed from the ***Smart VacPrep > Unit [n] > Start Degas*** menu selection. Provides the option to use degas conditions from selected sample files or a degas conditions files for each Smart VacPrep port. When using the *Start Degas* menu option, the degas process is started using the **Start** button on the *Start Degas* window. This option is more convenient for samples that have specific degas requirements that should be associated with the sample file when it is created.

- **QuickStart** menu option and Smart VacPrep panel buttons. Accessed from the ***Smart VacPrep > Unit [n] > QuickStart Degas Conditions*** menu selection. Provides the option to enter commonly used degas conditions for each Smart VacPrep port without the need for a sample information file.

  This option provides three ways to load information into *QuickStart Degas Conditions*:

  1. Load degas conditions from an existing degas conditions file
  2. Copy conditions from another Smart VacPrep unit and port
  3. Enter degas conditions manually

  When using the *QuickStart* menu option, the degas process is started using the **Select** button and **Start** buttons for each port on the Smart VacPrep unit.

  Using the *Start Degas* option does not change the degas conditions set up in *QuickStart*. *QuickStart* degas conditions are remembered for the next push button operation.

Sample tubes are attached to the sample ports. Heating mantles are then installed and secured to the sample tube with a metal clip. If using straight wall sample tubes, heating mantles with elastic cords may be purchased separately.The mantle is plugged into the power and thermocouple connectors on the front panel. The degassing process can then be started either by pressing the **Select** and **Start** buttons on the Smart VacPrep unit or by going to ***Smart VacPrep > Unit [n] > Degas > Start Degas***.

A Check Seal or TransSeal can be used to transfer air-sensitive samples from the Smart VacPrep to the analyzer's port without atmospheric contamination. To order these parts, reference the Parts and Accessories section for the analyzer. Instructions for using the Check Seal or TranSeal are included with the ordered part.

Smart VacPrep Components Table

Component	Description
Cancel button	Stops the degas process on all ports where the *Select* indicator is lit.
Ethernet port	Located on the left side panel. Use to connect an Ethernet switch.
Power Switch (not shown)	Located on the back of the unit, turns the unit on or off.
Select buttons	Used to select the ports to be degassed.
Start button	Starts the degas process on all ports where the *Select* indicator is lit.
Status Indicator	Three lights indicate the current status of the station. The lights flash when the valves are open. **Green**. Indicates the station is idle and not hot. The setpoint temperature is below 30 ºC and the actual temperature is below 50 ºC. **Red**. Indicates when the setpoint temperature is greater than 30 ºC and heat is being applied to the station. **Yellow**. Indicates the station is in use, but not currently being heated. The heating mantle may still be above 50 ºC.

SPECIFICATIONS FOR THE SMART VACPREP

The Smart VacPrep degasser has been designed and tested to meet the following specifications:

Specifications for the Smart VacPrep

Specification	Description
Backfill Options	Automatically backfill or wait for user command
Capacity	6 ports
Degas Method	Heat and vacuum
Physical Dimensions	Height: 69 cm (27 in.) Width: 53 cm (20.75 in.) Depth: 44 cm (17 in.) Weight: 32 kg (70 lbs)
Evacuation Period	Determined by selected elapsed time or achievement of selected low pressure target
Evacuation Rate	Selectable evacuation rate from 1.0 to 50.0 mmHg/s
Heating of Samples	Heating mantles
Heating Ramp Rate	Selectable from 0.1 ºC per minute to 10 ºC per minute
Soak Time	Zero to 999 minutes in one minute increments (Zero = Manual Cancel).
Temperature Accuracy	Deviation less than ±10 ºC at the sensing thermocouple embedded in the heating mantle
Temperature Control	One ramp during evacuation phase, five additional ramps during subsequent phases
Temperature Range	40 to 450 ºC in one degree increments
Vacuum Control	Selectable target pressure controls switchover from restricted to unrestricted evacuation.

SET THE VOLTAGE FOR THE SMART VACPREP

The Smart VacPrep voltage setting is housed in a compartment adjacent to the power connector on the back of the unit.

1. Use a flat head screwdriver to gently pry open the cover of the compartment.
2. Firmly press down on the voltage indicator and roll the indicator to display the appropriate voltage for your environment. Selections are: 100 Vac, 120 Vac, 230 Vac, and 240 Vac.
3. Firmly press the compartment cover shut. Ensure it closes securely. The selected voltage will appear in the window on the compartment cover.

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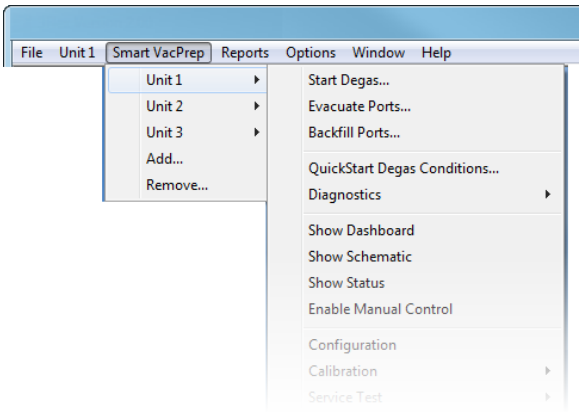
2 About the Smart VacPrep Software



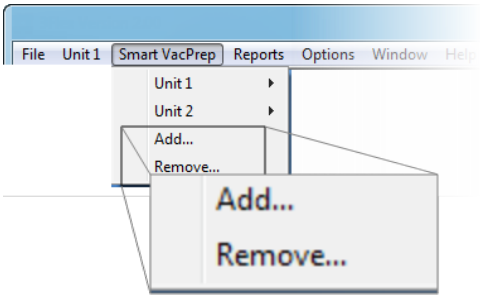
If a software update is required for the analyzer to support the Smart VacPrep, the software can be downloaded from the internet at this location:

http://www.micromeritics.com/Smart-VacPrep-Software.aspx

The degasser software is installed as a part of the analyzer installation. During installation, an IP address will be assigned to the unit allowing the unit to connect automatically during startup. Up to three additional degasser units can be installed.

Menu options for the Smart VacPrep are provided on the Smart VacPrep menu.

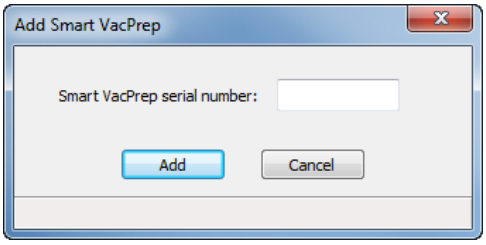
Add or Remove a Smart VacPrep



A Smart VacPrep unit must be removed from one application before it can be added to another application if they are running on the same subnet.

ADD A SMART VACPREP UNIT

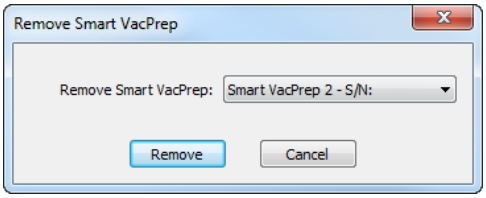
Smart VacPrep > Add



1. When prompted, enter the serial number of the unit being added. Duplicate serial numbers are not allowed.
2. Click **Add**. Initialization begins.

REMOVE A SMART VACPREP UNIT

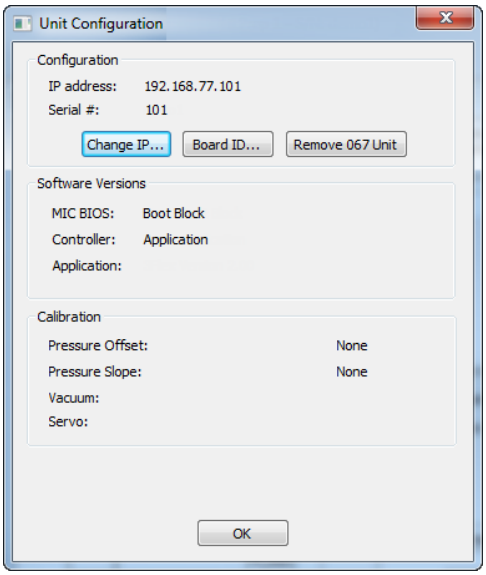
Smart VacPrep > Remove



1. Select the serial number to remove.
2. Click **Remove**.

SMART VACPREP CONFIGURATION

Smart VacPrep > Unit [n] > Configuration



Displays the Smart VacPrep configuration and software versions.

Smart VacPrep Unit Configuration Fields and Buttons Table

Field or Button	Description
Board ID	Click to display the board ID.
Change IP	Click to display the *Unit IP Setup* window. The IP address and Subnet mask assigned during installation display. Do not edit these fields unless instructed by a Micromeritics service representative.
OK	Saves and closes the active window.
Remove 067 Unit	Click to remove the selected Smart VacPrep unit.

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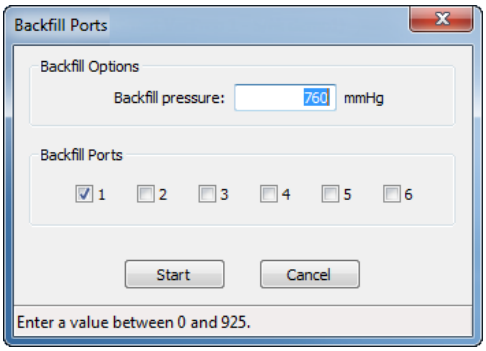
3 Operate the Smart VacPrep

Backfill the Smart VacPrep Ports

Smart VacPrep > Unit [n] > Backfill Ports

Use to backfill ports with gas.

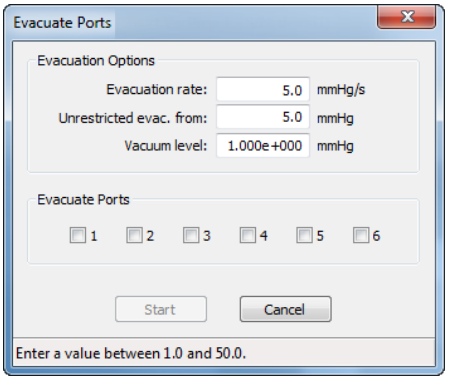


1. Enter the backfill pressure in the Backfill pressure field.
2. Select the ports to be backfilled.
3. Click **Start** to start the process or **Cancel** to stop the process.

Evacuate the Smart VacPrep Ports

Unit [n] > Smart VacPrep > Evacuate Smart VacPrep Ports

Allows manual evacuation of up to six degas ports.



Smart VacPrep Evacuate Ports Fields and Buttons

Field or Button	Description
Cancel	Discards any changes or cancels the current process.
Evacuate Options	• **Unrestricted evac. pressure.** Pressure value at which unrestricted sample evacuation should begin. • **Vacuum level.** Specify the vacuum level to be achieved before evacuation begins. • **Evacuation rate.** Rate of evacuation.
Evacuate Port[s]	Select the ports() to evacuate.
Start	Starts the operation.

QUICKSTART DEGAS CONDITIONS

Smart VacPrep > Unit [n] > QuickStart Degas Conditions

The *QuickStart Degas Conditions* option provides the ability to enter commonly used degas conditions for each Smart VacPrep port without the need for a sample information file. Degassing can then be easily started using the front panel buttons on the Smart VacPrep unit.

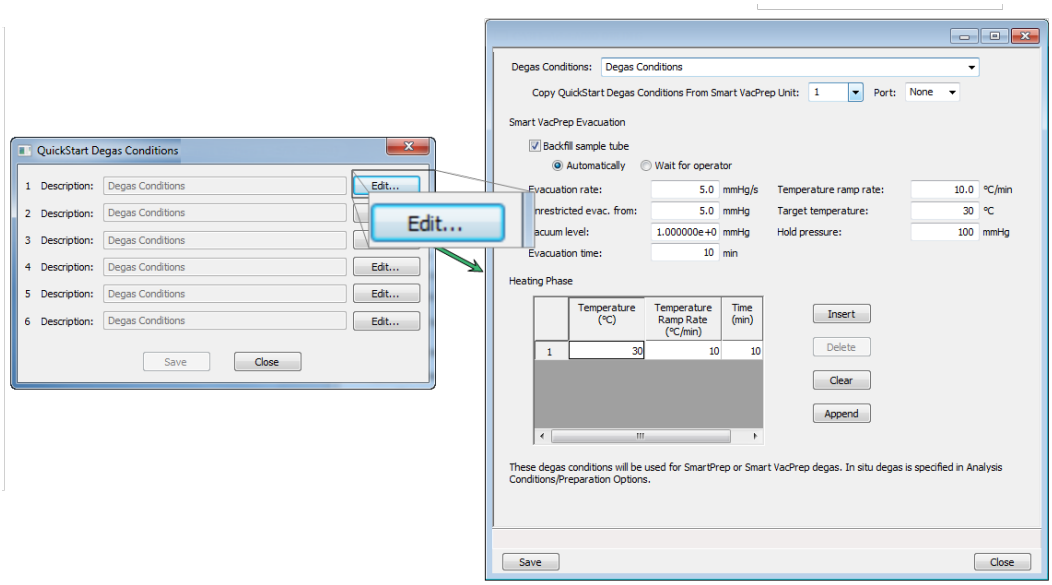
This option provides three ways to load information into *QuickStart Degas Conditions*:

1. Load degas conditions from an existing degas conditions file.
2. Copy conditions from another Smart VacPrep unit and port.
3. Enter degas conditions manually.

Degas conditions can be copied into sample files after degassing for record keeping.

When using the *QuickStart* menu option, the degas process is started using the **Select** button and **Start** buttons for each port on the Smart VacPrep unit.

QuickStart degas conditions are remembered for the next push button operation.



1. Go to ***Smart VacPrep > Unit [n] > QuickStart Degas Conditions***.
2. The six ports are represented by row numbers. Ports that are busy are grayed out and disabled. Click **Edit** for the port to be configured.
3. Enter degas conditions using one of the following methods:
   - To select degas conditions from a Degas Conditions file, click the dropdown arrow to the right of the *Degas Conditions* field. If the file is not shown, select *Browse* , locate the file, then click **Load**.
   - To copy QuickStart Degas Conditions from another Smart VacPrep unit, select the unit and port number to the right of the *Copy QuickStart Degas Conditions from Smart VacPrep Unit* field..
   - To enter degas conditions manually, complete the fields on the window. Reference the *Degas Conditions* section of the analyzer operator manual
4. Click **Save**. Repeat these steps for each port to be configured. If multiple ports will have the same degas conditions parameters, click **Edit** on the port and use the *Copy QuickStart Degas Conditions from Smart VacPrep Unit [n] Port [n* field to load the settings from the first port.
5. Repeat these steps for each applicable unit and port.
6. If using a Check Seal, verify that the Check Seal opener is installed in the Smart VacPrep port. Follow the instructions included with the Check Seal for instructions on inserting the opener.
7. Load the sample into the sample tube. If using a Check Seal or TranSeal, insert it into the sample tube.
8. Load the sample on the Smart VacPrep sample port.
9. Attach the heating mantle using the metal clip. If a straight wall tube is used, heating mantles with elastic cords are available. Reference the Parts and Accessories section of the analyzer operator manual.

10. Install the safety shield.
11. Press the **Select** button for the port to be degassed. A blue LED is lit.
12. Press the **Start** button on the Smart VacPrep panel to start the degas process for all ports that have a blue LED lit.
13. When degassing is complete and the sample has cooled, the sample can be transferred to an analysis port.

LOAD QUICKSTART DEGAS CONDITIONS INTO A SAMPLE FILE

QuickStart Degas Conditions can be copied into a sample file. Reference the Degas Conditions section of the analyzer operator manual.

Degas Conditions Fields and Buttons Table

Field or Button	Description
Close	Closes the active window.
Degas Conditions	Use to browse for a .DEG file that contains degas condition parameters to be used in the analysis.
Heating Phase	This option is applicable when degassing with either a Smart VacPrep or a SmartPrep. Enter up to five stages of degas conditions. • **Temperature.** Degas temperature. • **Temperature Ramp Rate.** Rate at which the temperature is to change. • **Time.** Amount of time to heat the sample. Use to modify the table contents. • **Insert.** Inserts one row above the selected row. • **Delete.** Deletes the selected row. • **Clear.** Clears all table entries and displays only one default value. • **Append.** Inserts one row at the end of the table.

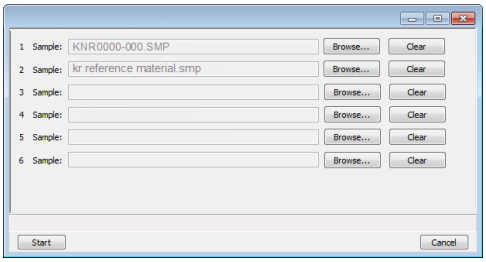
Degas Conditions Fields and Buttons Table (continued)

Field or Button	Description
Save	Saves changes to the active window.
Smart VacPrep Evacuation	This option is applicable only when degassing with a Smart VacPrep. • **Backfill sample tube.** Indicate if the sample tube should be backfilled automatically or wait for operator response. • **Evacuation Rate.** Rate used for evacuation. • **Unrestricted evac. from.** Pressure at which the unrestricted evacuation is to begin. • **Vacuum level.** Pressure for unrestricted evacuation. • **Evacuation time.** Length of time for preliminary evacuation before proceeding with the *Heating Phase* temperature schedule. The timer starts when the vacuum level is reached. • **Temperature ramp rate.** Rate at which the temperature is to change when advancing to the target pressure. • **Target temperature.** Targeted pressure for evacuation. • **Hold pressure.** Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold* pressure. This prevents damage to the sample structure due to 'steaming' and /or elutriation due to excessive escaping gas velocity.

START SMART VACPREP DEGAS

Smart VacPrep > Unit [n] > Start Degas

This option allows the degassing of up to six samples simultaneously. A sample can be added to any idle port and a degas operation started. When adding a sample, the degas operation in progress will be suspended until the sample reaches the target pressure, then degassing on all ports will resume. If other samples are still in the initial evacuation phase, the new samples will wait until the fast evacuation is started. Samples can be removed from any of the six ports without disturbing the degas operations already in process. Starting several samples as a batch allows them to perform the initial evacuation at the same time.



Degas conditions remain the same from degas to degas unless the *Degas Conditions* file is changed or replaced by the operator. A manual evacuation can be performed on any idle degas port.

The six ports are represented by row numbers. Ports that are busy are grayed out and disabled.

1. Click **Browse** to search for a sample file or a degas conditions file. Select the file, then click **Open**. Do this for each Smart VacPrep port to be used.
2. If using a Check Seal, verify that the Check Seal opener is installed in the Smart VacPrep port. Follow the instructions included with the Check Seal for instructions on inserting the opener.
3. Load the sample into the sample tube. If using a Check Seal or TranSeal, insert it into the sample tube.
4. Load the sample on the Smart VacPrep sample port.
5. Attach the heating mantle using the metal clip. If a straight wall tube is used, heating mantles with elastic cords are available. . *See "Parts and Accessories for the Smart VacPrep" on page 7 - 1.*
6. Install the safety shield.
7. Click **Start** on the *Start Degas* window to begin the degas process for the selected Smart VacPrep ports.
8. Click **Start** to begin the degas process for the selected Smart VacPrep ports.
9. Allow the samples to cool before transferring them to the analysis ports to start the analysis.

Smart VacPrep Start Degas Fields and Buttons Table

Field or Button	Description
Browse	Searches for a file. Select a file from the *Name* column or from the library, then click **Open**. Alternatively, double click the file name to open (or import) the file.
Cancel	Discards any changes or cancels the current process.
Clear	Clears the field.
Sample	Displays the path and file name of the selected .SMP file.
Start	Starts the degas operation for the selected ports.

Degas ports can also be canceled using buttons on the Smart VacPrep unit. Press **Select** on the unit to select the port, then press the **Cancel** button.

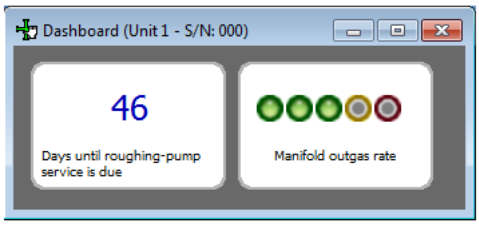
SHOW SMART VACPREP DASHBOARD

Smart VacPrep > Unit [n[> Show Dashboard

The dashboard displays the following:

- Number of days until roughing pump maintenance is due
- Manifold outgas rate

Data for the dashboard comes from the logged diagnostic data. The dashboard is automatically kept current as the relevant diagnostic data items are updated. The gauges will be updated even if the dashboard window is not open.



Red numbers on the dashboard require attention. To reset the dashboard numbers, right click on the dashboard setting, then click **Reset**.

Smart VacPrep Dashboard Gauges and Descriptions Table

Field or Button	Description
Days until roughing-pump service is due	Annual maintenance is recommended. The number of days until the anniversary of the last pump maintenance is shown. The displayed value is updated at least once per day and when the maintenance time is reset. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.
Manifold outgas rate	Provides the qualitative indication of the outgas rate in the dosing manifold. LED images constitute a bidirectional bar graph of the outgas rate. The gauge is updated after each outgas rate measurement. • Three green LEDs are lit if outgas rate is below 30% of outgas rate limit. • At 30%, the left LED turns off. • At 60%, the center LED turns off. • At 90%, three green LED lights turn off and the center yellow LED is

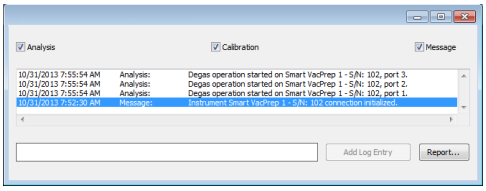
Smart VacPrep Dashboard Gauges and Descriptions Table (continued)

Field or Button	Description
	turned on. • At 110% and above, only the red LED is lit and attention is required.

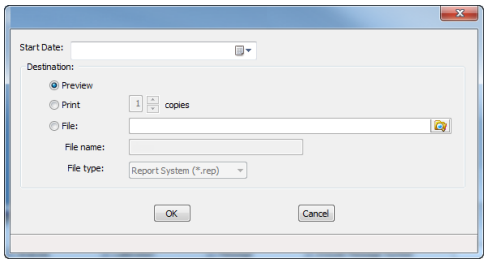
SHOW SMART VACPREP LOG

Smart VacPrep > Unit [n] > Show Log

Use to display a log of recent analyses, calibrations, or messages.



Analyzer Log Fields and Buttons Table

Field	Description
Add Log Entry	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Analysis / Calibration / Message	Select the logs to display.
Report	Click to display the *Instrument Log Report Settings* window to specify report output options.  • **Start Date.** Displays a calendar to select the start date for the report. • **Preview.** Previews the predefined report on the screen. • **Print.** Sends the report to the default printer. • **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected.

Analyzer Log Fields and Buttons Table (continued)

Field	Description
	• **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.

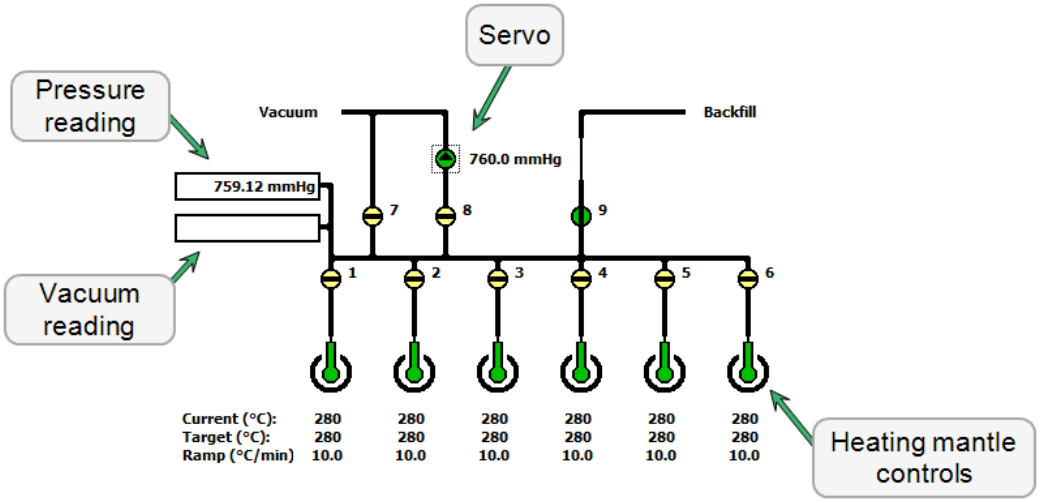
SHOW SMART VACPREP SCHEMATIC

Unit [n] > Show Instrument Schematic

Use to display an analyzer schematic. To operate the valves and elevator from this window, manual control must be enabled (***Smart VacPrep > Unit [n] > Enable Manual Control***).

Smart VacPrep Schematic Components Icon Table

Field or Button	Description
	Open Valve. Green indicates an open valve.
	Closed Valve. Yellow indicates a closed valve.
	Servo Valve. Closed.
	Servo Valve. Open.
	Sample Tube. Cannot be manually controlled.



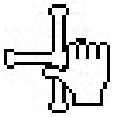
Smart VacPrep Schematic Components Table

Schematic Components	Description
1-6	Sample port valves
7, 8, 9	Vacuum and backfill valves
Heating mantle controls	Sets the ramp rate and target temperature

SMART VACPREP SCHEMATIC SHORTCUT MENUS

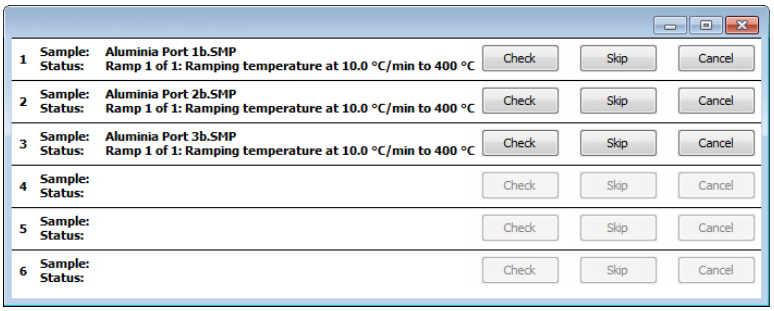
Each manually controlled schematic component has a shortcut menu displaying the operations available for that particular component. To access the shortcut menu, hover the mouse pointer over the component and right click.

Smart VacPrep Schematic Shortcuts Table

Schematic Shortcut Icon	Available Options:
Valve options 	• **Close** - Closes the selected valve. • **Open** Opens the selected valve. • **Set** - Use to set the servo valve target pressure and to dose or evacuate.
Temperature control options 	• **Disable**. Select to disable the temperature control options for the selected port. • **Set**. Select to set the following: ○ Enable or disable temperature control ○ Control sample temperature ○ Control furnace temperature ○ Cool the sample to less than 50 °C ○ Set heater power percent

SHOW SMART VACPREP STATUS

Smart VacPrep > Unit [n] > Show Status



The *Smart VacPrep Status* window allows the monitoring of degas operations for a manual degas operation.

Smart VacPrep Status Fields and Buttons Table

Field or Button	Description
Cancel	Cancels the current degas operation.
Check	Click to check the outgassing rate of the samples on the selected port. The following process occurs: • The current degassing step is suspended. • The vacuum valves are closed and the vacuum level on the selected ports is monitored. • The *Status* window indicates that the degassing operation is being checked and displays the outgassing rate as it becomes available. During the degas check, the **Check** button changes to **Continue**. When **Continue** is clicked, the valves open and the degassing operation resumes. If the outgassing rate indicated that the sample has been freed of contaminants (minimal pressure increases), click **Skip** to advance to the next state of the degassing operation. For example, if degassing is checked after the setpoint is attained, **Skip** advances you to the ramping stage.
Sample	The name of the sample file.
Skip	Skips the current degas operation.
Status	Indicates the current status of the degas.

Blank Page

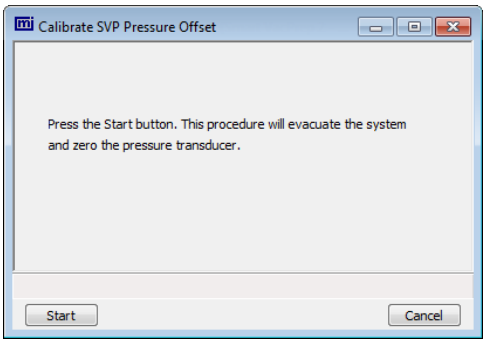
4 Smart VacPrep Calibration

Smart VacPrep > Unit [n] > Calibration

Various calibration procedures and service tests are included in the operating program. These procedures and tests are designed to provide a service representative with instrument readouts, as well as to assist in troubleshooting potential problems.

Smart VacPrep Pressure Offset

Smart VacPrep > Unit [n] > Calibration > Pressure Offset



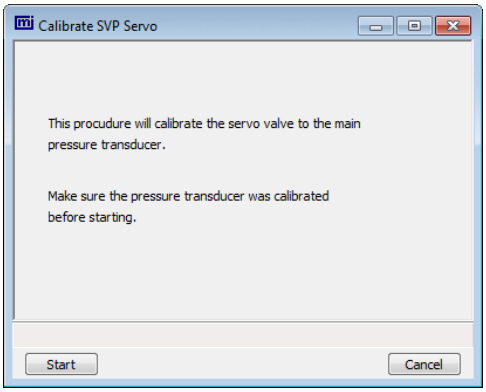
Use to perform system calibration. This option evacuates the system and zeros the pressure transducer.

Disabled calibration options can be accessed only with the assistance of an authorized Micromeritics service representative.

Click **Start** to begin the process.

Smart VacPrep Servo Valve Calibration

Smart VacPrep > Unit [n] > Calibration > Servo Valve



Use to calibrate the servo valve to the manifold pressure transducer. The servo valve should always be recalibrated after a pressure calibration has been performed. The pressure transducer should be calibrated before starting this calibration procedure.

1. Go to ***Smart VacPrep > Unit [n] > Calibration > Servo Valve***.
2. Click **Start**. The window closes when the calibration is complete. Click **Cancel** to stop the calibration process.

Servo Valve Fields and Buttons Table

Field or Button	Description
Cancel	Discards any changes or cancels the current process.
Start	Starts the operation.

5 Smart VacPrep Diagnostics

Diagnostic Test Report for Smart VacPrep

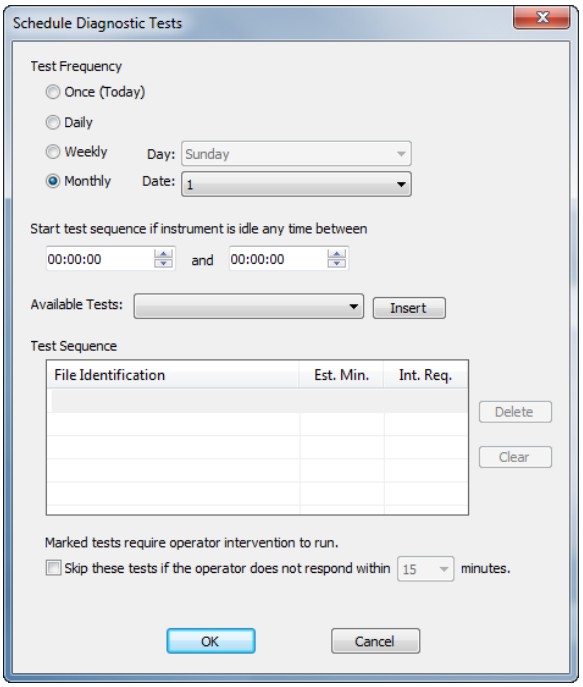
Smart VacPrep > Unit [n] > Diagnostics > Diagnostic Test Report

Displays previously run diagnostic tests. Separate directories store tests run once, daily, weekly, and monthly.

Schedule Smart VacPrep Diagnostic Test

Smart VacPrep > Unit [n] > Diagnostics > Schedule Diagnostic Tests

Allows the specification of one-time or periodic running of a sequence of diagnostic tests. A separate list of tests is saved for each of the possible test frequencies. Tests are categorized and flagged as requiring intervention or not. If tests requiring intervention are scheduled, the operator has the option of skipping these tests if the operator does not respond within a specified time after an initial prompt is displayed, before the test is started. Events are logged in the instrument log for all starting, ending, and skipped tests.



Schedule Diagnostics Fields and Buttons Table

Field or Button	Description
Available Tests drop-down list	Select one or more tests to run unattended. Select the test and click the Insert button for the test to display in the Test Sequence box.
Cancel	Discards any changes or cancels the current process.
Intervention Required	Check this option if any test requiring operator intervention should be skipped if the operator does not respond within the specified time.
OK	Saves and closes the active window.
Start Test Sequence if Instrument is Idle Any Time Between	Enter a From and To time for an unattended test to begin if the instrument is idle at any time during the entered time frame.
Test Frequency	Select how often the test is to run unattended.
Test Sequence	Provides the test file identification and estimated run time. A check mark in the Intervention Required column indicates that operator intervention is required. Click Delete to remove the selected test or Clear to clear the entire table of all entries. To add a test to the test sequence, highlight a row in the Test Sequence box, select a test from the Available Tests list and click Insert. The new test will be inserted above the highlighted row. Select a row and click Delete to remove the test from the sequence. Select Clear to remove all entries from the Test Sequence box.

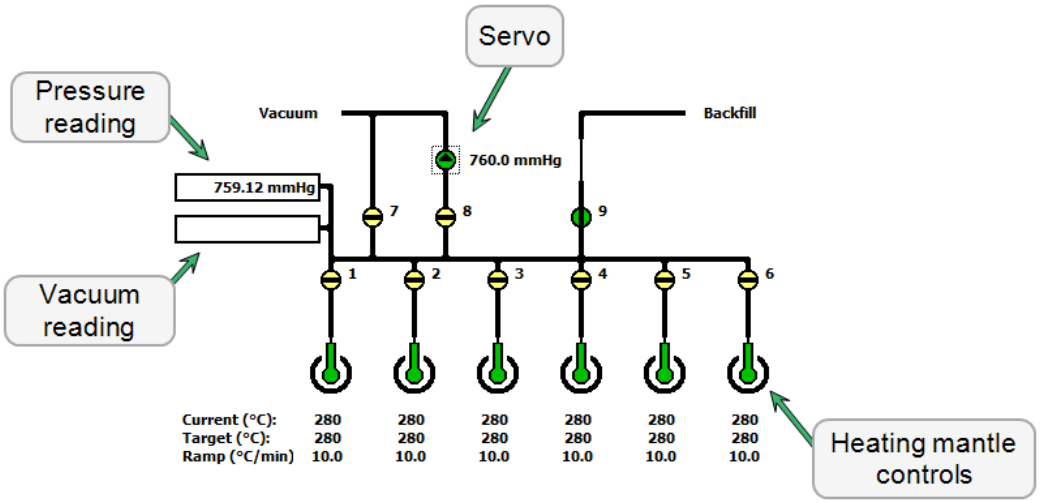
6 Maintenance and Troubleshooting

Enable Manual Control for Smart VacPrep

Smart VacPrep > Unit [n] > Enable Manual Control

Use to enable the manual control of certain system valves and elevator components. When this option is enabled, a checkmark appears to the left of ***Unit [n] > Enable Manual Control***.

If the schematic is not immediately visible, go to ***Unit [n] > Show Instrument Schematic***.



Smart VacPrep Schematic Components Table

Schematic Components	Description
1-6	Sample port valves
7, 8, 9	Vacuum and backfill valves
Heating mantle controls	Sets the ramp rate and target temperature

Smart VacPrep Schematic Components Icon Table

Field or Button	Description
	Open Valve. Green indicates an open valve.
	Closed Valve. Yellow indicates a closed valve.
	Servo Valve. Closed.

Smart VacPrep Schematic Components Icon Table (continued)

Field or Button	Description
	Servo Valve. Open.
	Sample Tube. Cannot be manually controlled.

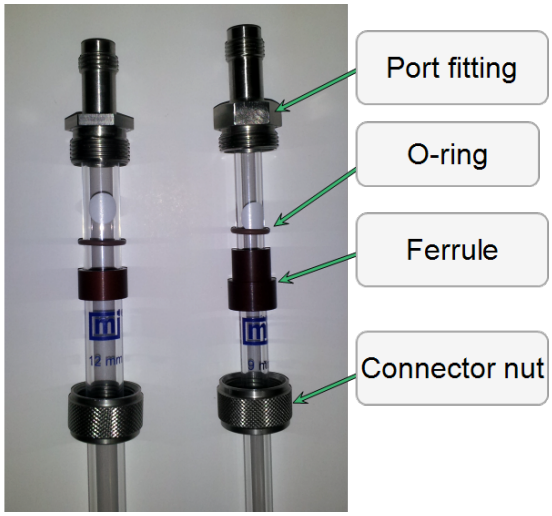
Replace the Smart VacPrep Fuse

The Smart VacPrep uses two 5 amp fuses located in a compartment adjacent to the power connector on the back of the unit.

1. Use a flat head screwdriver to gently pry open the cover of the compartment.
2. Use the flat head screwdriver to gently pry the fuses out of the holder.
3. Insert a new fuse into each holder, then push the holders back into the slot until snug.
4. Press the compartment cover shut. Ensure it closes securely.

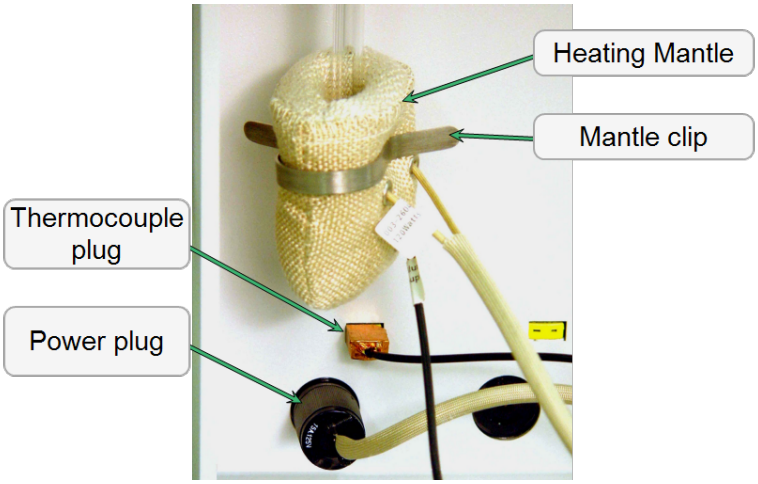
Sample Tube Installation on the Smart VacPrep

1. While holding the degas port plug, turn the connector nut counterclockwise to loosen. Remove both the plug and the nut.



2. If using a rubber stopper, remove it from the sample tube.If using a Check Seal or TransSeal, do not remove them from the sample tube. Place the port connector nut, ferrule, and O-ring onto the sample tube.

3. If using a Check Seal, verify that the Check Seal opener is installed in the sample port.
4. To attach the sample tube set to the degas port, push the sample tube in to a full stop. Secure the sample tube in place by sliding the connector nut, ferrule, and O-ring up onto the degas port and turning the connector nut clockwise. Tighten the nut securely by hand.
5. Place a heating mantle over the sample tube bulb and secure the mantle in place with a mantle clip.



6. Insert the heating mantle thermocouple plug into the thermocouple connector.
7. Insert the heating mantle power plug into the power connector and twist the power plug to lock securely.
8. After installing the sample tubes, install the safety shield around the sample tubes and heating mantles to minimize the risk of receiving a burn by touching hot components.

OIL BASED VACUUM PUMP

INSPECT AND CHANGE VACUUM PUMP OIL

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

The oil in the vacuum pump should be changed every three months when the efficiency of the vacuum pump declines (requiring increased time to reach vacuum levels) or if it becomes discolored.

Inspect the Oil

View the vacuum pump oil through the oil-level window. The oil level should be midway between the indicators on the oil-level window when the pump is running. Oil in good condition is clean, clear or light in color and transparent. Change the oil if it has darkened.

Change or Add Oil

Drain the oil while the pump is warm and disconnected from the power source.

Use oil supplied by Micromeritics or refer to the vacuum pump manual for other acceptable oils.

1. Unplug the vacuum pump from the power source.
2. Loosen the wing nut on the clamp at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.

3. Grasp the handle on top of the vacuum pump and place the pump on a work surface.
4. Place a waste container under the drain spout.

5. Remove the drain plug and drain the oil into the waste container.
6. Replace the drain plug.
7. Remove the drain plug from the oil-fill port on top of the pump.

8. Slowly add oil to the port until the level is midway between the indicator lines in the oil-level window when the pump is running.

Do not allow oil to rise above the midway position when the pump is running. Doing so may cause oil to splash into the oil filter causing contamination.

9. Check the washer or O-ring used at the oil-filling port and replace if necessary.
10. Insert the oil-fill plug and turn clockwise to tighten.
11. Check the alumina in the oil vapor trap. If most of the pellets are no longer white, replace the alumina before reattaching the vacuum pump. *See "Replace the Alumina in the Oil Vapor Trap" below.*
12. Reconnect the vacuum pump hose.
13. Reconnect the power cord to the power source.
14. Turn the vacuum pump on and recheck the oil level.
15. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the fresh fluid and to produce efficient vacuum operations.

Replace the Alumina in the Oil Vapor Trap

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

The activated alumina in the oil vapor trap becomes saturated during use. The alumina should be inspected periodically and replaced when most of the alumina pellets are no longer white.

Do not perform the following procedure on used alumina. The resultant oil vapors may cause a fire.

1. Disconnect the vacuum pump from the analyzer and place it on a work table.
2. Loosen the wing nut on the clamp at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.

3. Loosen the wing nut on the clamp at the bottom of the oil vapor trap. Open the clamp and remove the trap.

4. Remove one end fitting from the trap body and dispose of the used alumina in an appropriate manner.
5. Wash the trap body with a detergent-based soap. Rinse with water, then rinse with isopropyl or ethyl alcohol. Set the trap aside and allow to dry thoroughly.

Exposure of the trap body to oil vapor may cause small cracks on the inside surface of the trap body. Under normal circumstances, these cracks will not cause problems or leaks.

Prepare fresh alumina

1. Preheat the oven to 300 °C.
2. Pour approximately 180 grams of fresh alumina into a glass or metal container (approximately 250 mL if a graduated beaker is used). Place the container in the oven.
3. Bake the alumina for two hours at 300 °C.
4. Remove the baked alumina from the oven and allow to cool until luke warm. A desiccator may be used to speed the cooling process.
5. Use a small spatula to gently pry the O-ring from the end fittings of each end of the trap body.

6. Inspect the O-rings. If dusty, clean with a lint-free tissue. If damaged, replace with a new O-ring.
7. Screw one of the end fittings onto the trap body.
8. Ensure the trap body is dry and the alumina is lukewarm. Pour the alumina pellets into the trap until level with the top of the trap body.
9. Screw the other end fitting back onto the trap and tighten securely by hand.
10. Lightly tap both ends of the trap body on the work surface to remove remaining dust from the pellets.

11. Inspect the centering ring before replacing it onto the intake port. If it appears to be flattened, replace it. A flattened centering ring can cause vacuum leaks. There are two types of centering rings. Use the one with the smaller opening at the intake port.
12. Place the centering ring on the intake port.
13. Place the trap on the centering ring.

14. Open the clamp and place it around the flange of the intake port and the flange of the trap. Swing the clamp fastening screw toward the intake port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.

15. Reconnect the hose from the analyzer to the oil vapor trap.
    a. Place the clamp around the vacuum pump hose flange and vapor trap.

16. Plug the pump power cord into the power source. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the fresh oil and to produce efficient vacuum operations.

REPLACE THE VACUUM PUMP EXHAUST FILTER

The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

The gases used by the analyzer are exhausted by the oil-filled vacuum pump. The filter minimizes the release of oil vapor and should be replaced when it becomes so saturated with oil that it is ineffective.

Exhaust filters are used to minimize the release of oil vapors. The gases are diluted substantially upon release from the vacuum pump; however, it may be necessary in some locations to provide a fume hood for protection from hazardous gases and vapors released into the work area.

1. Turn off the power to the pump and disconnect the pump from the power source.
2. Loosen the clamp wing nut at the vacuum pump exhaust port. Swing the clamp away from the exhaust port and remove it.

3. Remove and discard the exhaust filter. Ensure the centering ring remains in place on the exhaust port.
4. Place the new filter on the centering ring.
5. Open the clamp and place it around the exhaust port flange and the exhaust filter flange. Swing the clamp fastening screw toward the exhaust port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.
6. Turn on the power to the pump.

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7 Parts and Accessories for the Smart VacPrep

Part Number	Item and Description
Cables	
003-63801-01	Cable, Ethernet straight-thru; for connecting instrument to control module (computer)
Gas Cylinder Accessories	
004-25549-00	Reducer, 1/8 in tube × 1/4 in tube
004-33601-00	Expansion kit; adds an additional outlet to the gas regulator
004-33602-00	Pressure relief kit; prevents excessive gas pressure in the event of regulator failure (not to be used with toxic gases)
004-62230-58	Gas regulator, CGA 580 fitting, 30 psig (He, N2, Kr, Ar)
230-02001-00	Gas inlet line, 1/8 in. × 6 ft., copper
Heating Mantle and Accessories	
003-26044-00	Smart VacPrep heating mantle 24V, 120W
067-14701-00	Heating Mantle Shield, (1) replacement shield
067-33002-00	Heating Mantle Shield Option, includes (2) shields, each shield covers (3) heating mantles
230-25808-00	Smart VacPrep heating mantle clip
Operating Supplies	
004-25466-00	O-RING, -010 70 DURO BUNA-N; for use with sample port dust frit
004-27041-00	Sample port dust frit, 20 microns
067-33601-00	Operating supplies, sample tube o-rings (1/2 in. and 12 mm), sample port dust frit , o-rings for sample port dust frit (6-month supply)
067-33602-00	Extended operating supplies, sample tube o-rings (1/2 in. and 12 mm), sample port dust frit , o-rings for sample port dust frit (12 month supply)
Sample Tubes and Accessories	
004-25013-02	O-ring, -013 70 DURO VITON; for use with 12 mm sample tubes (brown)
004-25022-00	O-ring, -012 70 DURO BUNA-N; for use with 3/8 in sample tubes
004-25044-00	O-ring, -013 70 DURO BUNA-N; for use with 1/2 in sample tubes (black)
004-25466-00	O-ring, -010 70 DURO BUNA-N; for use with 1/4 in sample tubes
004-25647-01	O-ring, -904, VITON; for use with 9 mm sample tubes (brown)
067-25840-00	Sample tube ferrule, 12 mm (red)
067-25840-01	Sample tube ferrule, 9 mm (red)
067-33603-00	Option kit, 1/4 in. ferrules and O-rings (6 ferrules and 12 O-rings)

Part Number	Item and Description
067-33604-00	Option kit, 3/8 in. ferrules and O-rings (6 ferrules and 12 O-rings)
067-33605-00	Option kit, 9 mm ferrules and O-rings (6 ferrules and 12 O-rings)
260-25843-00	Sample tube ferrule, 1/2 in
300-25824-00	Sample tube nut
300-25825-00	Sample tube ferrule, 1/4 in
300-25826-00	Sample tube ferrule, 3/8 in
Smart VacPrep Systems	
067-00000-00	Smart VacPrep (no vacuum pump)
067-00000-11	Smart VacPrep with 110/120V oil-sealed vacuum pump system
067-00000-22	Smart VacPrep with 220/240V oil-sealed vacuum pump system
067-00001-00	Smart VacPrep with hybrid turbo vacuum pump system
Vacuum Pump and Accessories	
004-16003-01	Oil, vacuum pump, 1 Liter
004-16830-00	Activated alumina, 500 grams; for oil vapor trap
004-25491-02	Centering ring, NW 40, BUNA-N, AL; for use with hybrid turbo vacuum pump
004-25509-00	Clamp, NW 10/16
004-25626-08	Flex tube, 3/4-in. OD x 48-in. long, NW-16, NW-40
004-25630-00	Centering ring, NW 16
004-25652-00	O-ring, size 217, for oil vapor trap
004-25860-00	Fitting, elbow, NW-16; for connecting to the vacuum inlet
004-27040-00	Filter, for vacuum pump exhaust
004-62023-01	Service kit, for vacuum pump P/N 062-00004-00 (for Edwards and VacuuBrand)
062-00000-11	Vacuum pump with built-in anti-suckback valve, 100/120 VAC, includes hose kit
062-00000-23	Vacuum pump with built-in anti-suckback valve, 220/240 VAC, includes hose kit
062-00004-00	Hybrid turbo vacuum pump assembly
062-33002-00	Activated alumina oil vapor trap, for one oil-based vacuum pump
260-25896-00	Adapter, KF-16 to 7/16 hose

8 Error Messages

Program error messages are listed numerically. If the *Action* response indicates to contact a Micromeritics service representative, record the error message, then make backup copies of any files involved in the operation.

4407 Error searching for installed Smart VacPreps. The registry could not be read..

Cause: The application was not installed properly.

Action: Reinstall the application. If the problem persists, contact your Micromeritics service representative.

4408 The %s in %s already has Smart VacPrep S/N %d. The Smart VacPrep must be removed from the %s before it can be added.

Cause: The Smart VacPrep was already installed for another application / unit.

Action: Remove the Smart VacPrep from the installed unit before adding it to the preferred unit.

4409 A free IP address on the same subnet as %s could not be found."

Cause: All IP addresses on the network for the Ethernet card specified during installation are in use by other Micromeritics applications on this computer.

Action: Uninstall unused Micromeritics applications.

Action: Configure a different Ethernet card for use by the application using the application installer.

4410 The .INI file could not be updated with configuration for Smart VacPrep S/N %s."

Cause: The application .INI file is opened by another application and could not be updated.

Action: Close all open applications and add the Smart VacPrep again using the Smart VacPrep menu.

4411 Error dosing.

Cause: The backfill timed out.

Action: Ensure there is gas available and the pressure regulator is set to the appropriate pressure. Also ensure that the gas supply regulator shut-off valve is open.

4412 Error calibrating the servo.

Cause: Calibration results are out of range.

Action: Follow standard calibration procedures and try again. If the problem persists, contact your Micromeritics service representative.

4413 Error overheating on port [0]. Current = [TEMPC0] [TEMPC-U], Target = [TEMPC0] [TEMPC-U], Limit = [TEMPC0] [TEMPC-U].

Cause: The temperature of the indicated mantle exceeded the maximum allowed value.

Action: Ensure the power and thermocouple connectors for the mantle are properly installed. If they are, the mantle may be defective; contact your Micromeritics service representative.

4414 Error thermocouple unplugged on port [0]. Target = [TEMPC0] [TEMPC-U]."

Cause: The thermocouple is unplugged or has malfunctioned.

Action: Ensure the thermocouple is plugged in. If the problem persists, contact your Micromeritics service representative.

4420 Communications error.

Cause: The application failed to connect to the Smart VacPrep.

Action: Ensure the unit is powered and properly connected to the network specified during installation. Check the power cable, power switch, and Ethernet cable, then reconnect to the Smart VacPrep either through the Smart VacPrep menu for this instrument or by restarting the application. If the problem persists, contact your Micromeritics service representative.

4421 Smart VacPrep S/N %s is busy and could not be removed.

Cause: The Smart VacPrep cannot be removed because it is currently performing an operation.

Action: Wait until the Smart VacPrep completes the current operation and try again.

4422 The file %s does not exist.

Cause: The selected file does not exist on the disk drive.

Action: Select an existing file. Ensure that the file has been created before use.

4423 The sample %s is already selected on port %d.

Cause: The selected sample file is already selected for use on a different port.

Action: Select another sample file for this port.

4424 The file %s on port %d could not be opened. Check if the sample file is already in use for editing or analysis.

Cause: The selected sample file is already open by this or another application.

Cause: The selected sample file is damaged.

Action: Select another sample file.

4425 The sample %s on port %d has an invalid status and cannot be used for degassing.

Cause: The status of the file is not consistent with the current operation.

Action: Select a sample file that has not been used for an analysis. Only sample files with a status of No Analysis or Prepared may be selected.

4426 Port %d is currently in use. Operation cannot be started.

Cause: The current operation cannot be completed because the port is already in use.

Action: Wait for port to terminate operation or perform the desired operation on an unused port.

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