



3Flex™
Surface Characterization Analyzer
Operator's Manual
V1.01

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International Sales - (770) 662-3660

Domestic Repair Service - (770) 662-3666
Customer Service - (770) 662-3636

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1. USING THE ANALYZER

Introduction

This manual provides details on program menu options and operating instructions. This chapter contains information on the system hardware and software components.

To help you operate the analyzer system more efficiently, refer to:

- Chapter 1, USING THE ANALYZER - prior to operating the analyzer and software
- Chapter 2, OPERATIONAL PROCEDURES - for step-by-step instructions for common operations

Organization of the Operator's Manual

This operator's manual is organized as follows:

Chapter	Description
Chapter 1	USING THE ANALYZER Provides a general description and features of the analyzer, organization of the manual, and software and instrument interface.
Chapter 2	OPERATIONAL PROCEDURES Provides step-by-step procedures for the operations available using the application.
Chapter 3	FILE MENU Provides a description of the File menu options and field and button definitions.
Chapter 4	UNIT MENU Provides a description of the Unit menu options and field and button definitions.
Chapter 5	REPORTS MENU Provides a description of the Reports menu options and field and button definitions.
Chapter 6	OPTIONS MENU Provides a description of the Options menu options and field and button definitions.
Chapter 7	DIAGNOSTICS Provides information on using system diagnostics.

Chapter	Description (<i>continued</i>)
Chapter 8	<p>TROUBLESHOOTING AND MAINTENANCE</p> <p>Provides user maintenance and service information.</p>
Chapter 9	<p>ORDERING INFORMATION</p> <p>Provides ordering information for the application and analyzer system components.</p>
Appendix A	<p>FORMS</p> <p>Provides a sample information worksheet used to assist in obtaining a sample mass.</p>
Appendix B	<p>ERROR MESSAGES</p> <p>Program error messages are listed numerically. If the Action response indicates to contact a Micromeritics service representative, record the error message and make backup copies of any files involved in the operation.</p> <p>Provides a list of program error messages, causes, and actions.</p>
Appendix C	<p>CALCULATIONS</p> <p>Provides calculations used in reports.</p>
Appendix D	<p>FREE SPACE CORRECTION</p> <p>Provides a discussion of the free space measurements for the analyzer.</p>
Appendix E	<p>MAINTAINING HIGH PURITY GASES</p> <p>Provides information on the importance of maintaining high purity gases.</p>
Appendix F	<p>DFT MODELS</p> <p>Provides information on DFT models.</p>
Appendix G	<p>USER-DEFINED REPORTS, PYTHON MODULE</p> <p>Provides information on accessing and creating user-defined reports using Python.</p>
Index	<p>INDEX</p> <p>Provides quick access to a subject matter.</p>

Conventions

This manual uses the following icons to identify notes of importance, warnings and cautions.



Notes contain important information pertinent to the subject matter.



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



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

Equipment Description



The analyzer is an automated gas adsorption analyzer with three ports allowing up to three samples to be analyzed simultaneously. The core analytical engine is composed of an advanced vacuum system, a versatile analysis manifold, and sensitive pressure transducers on each analysis port. The basic configuration consists of 2 or 3 micropore ports. Standard ports can be individually upgraded for high quality micropore analyses with 10 torr and 0.1 torr transducers on each micropore port.

The analyzer also features a dedicated port for measuring the saturation pressure (P_0) on a continuous basis. Surface areas as low as $0.01 \text{ m}^2/\text{g}$ can be measured using nitrogen and as low as $0.001 \text{ m}^2/\text{g}$ with krypton as the adsorptive. Up to 1000 data points can be collected allowing the observation of minute details of the isotherm. The analyzer must be equipped with a 10 mmHg transducer and a high-vacuum pump to provide this capability.

Up to four analyzers can be operated with one computer. The system consists of the analyzer, an optional SmartPrep degasser for preparing samples, a dry diaphragm roughing vacuum pump, and a computer for entering analysis and report options.

Windows 7 Professional or higher operating system is recommended for the best user experience. If the computer is to be connected to a network, two Ethernet ports are required.

Vacuum Pump

The analyzer requires a dry diaphragm roughing vacuum pump for sample analysis. Appropriate vacuum pumps are available from Micromeritics. Refer to **ORDERING INFORMATION**, page 9-1.

Vapor Option

Vapor adsorption allows analyses with hydrocarbon vapors or water vapor. The instrument 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. The instrument is quickly and easily configured with the vapor option.

Micropore Option

Each port on the standard instrument 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.

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

Degasser Options

- **SmartPrep 065**

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.

The SmartPrep is the recommended degassing unit for the instrument.

- **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 6 samples. It lets you choose the temperature, gas, and flow rate best suited for your sample material and application.

The FlowPrep is an independent unit and not controlled by the instrument.

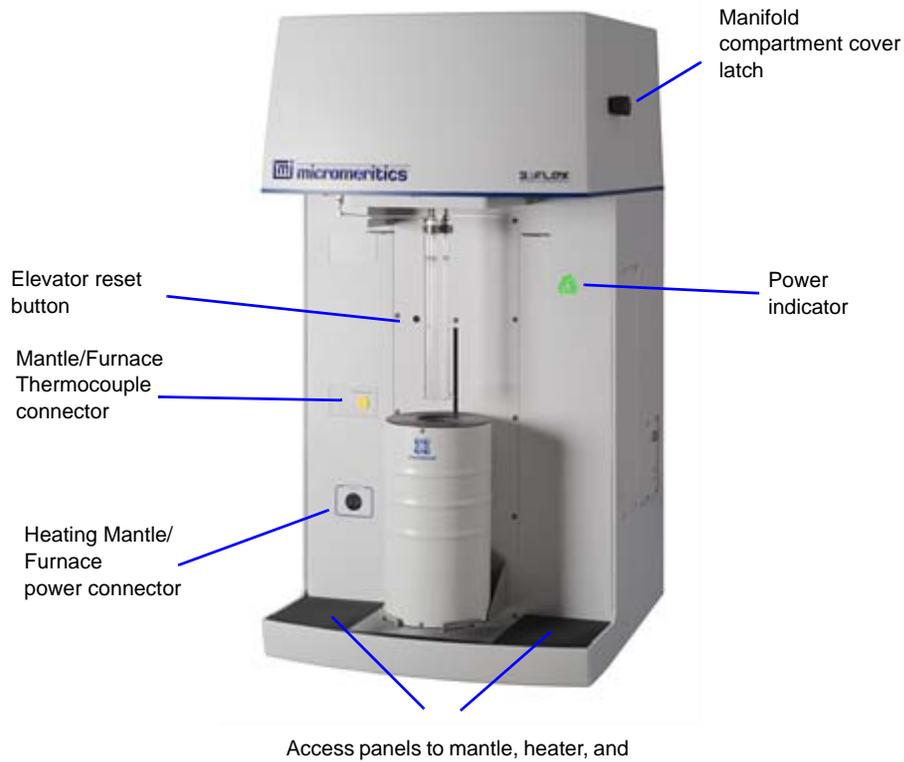
- **VacPrep 061**

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 6 samples. This combination allows you to choose the preparation method that is best suited to your material or application. Needle valves are also provided allowing you to introduce the vacuum slowly to prevent fluidization of samples.

The VacPrep is an independent unit and not controlled by the instrument.

Instrument Components and Connectors

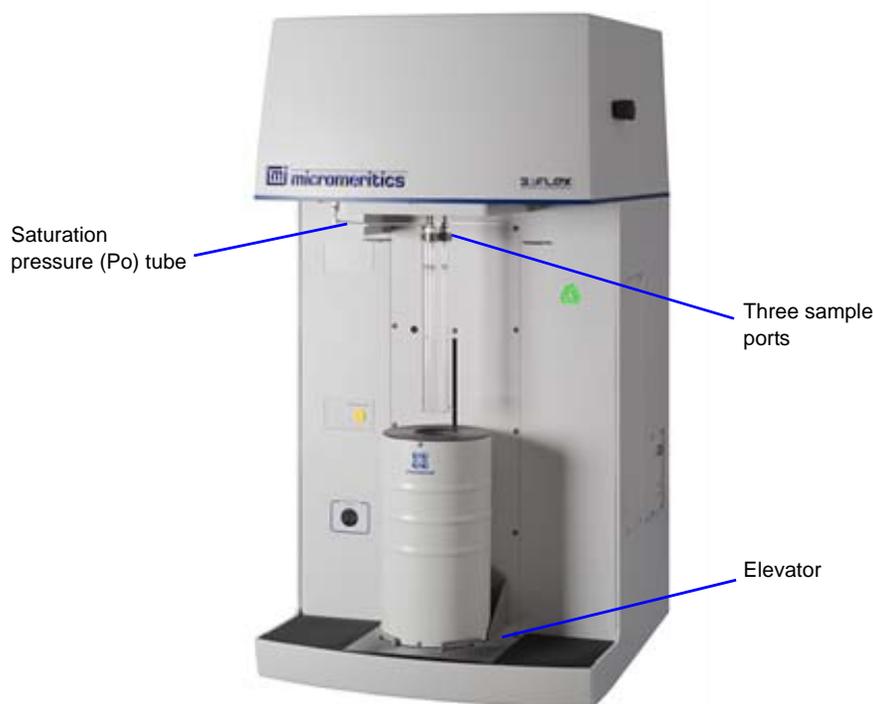
Front Panel



Component	Description
Power indicator	Blinks when power is applied to the analyzer; illuminates when analysis program is initiated and ready for operation.
Manifold compartment cover latch	Latches to hold the removable manifold compartment cover.
Elevator reset button	Resets the elevator in case of failure. The message Elevator Circuit Breaker Open on the instrument schematic indicates this reset button should be pushed.
Thermocouple connector	Connector for the thermocouple.
Mantle/Furnace power connector	Power connector for the mantle or furnace.

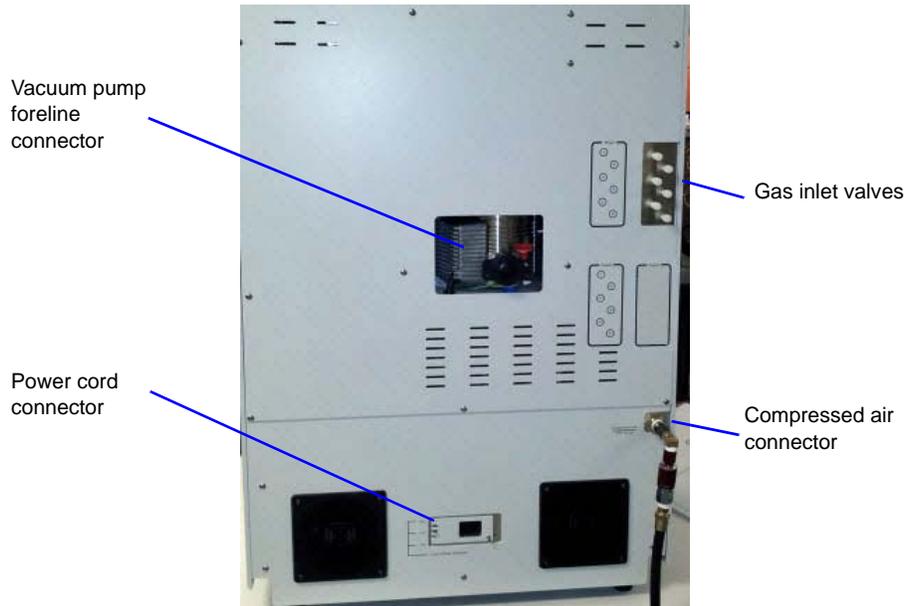
Component	Description (<i>continued</i>)
Access panels	Lift the pad then lift the access panel to access the reset buttons for the mantle, heater, and transformer.

Sample Compartment



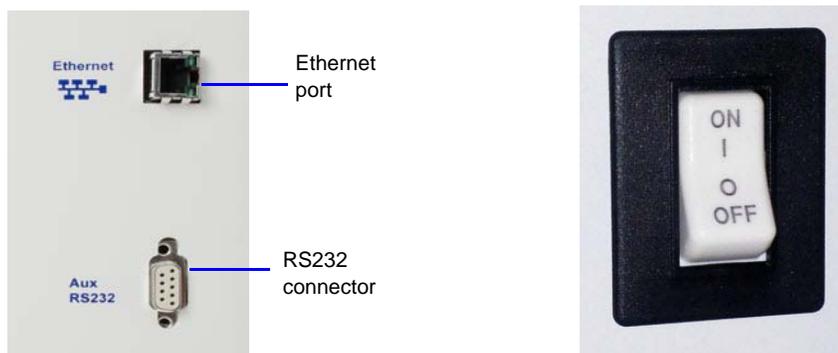
Component	Description
Sample ports	For installing up to three sample tubes.
Po tube	For measuring the saturation pressure.
Elevator	Allows placement of the Dewar around the sample and Po tubes. The elevator is raised automatically when the analysis is started and lowers automatically upon completion. During analysis, the elevator <i>optionally</i> lowers after the free-space measurement to allow evacuation, then is raised and continues the analysis.

Rear Panel



Component	Description
Gas inlet valves	Inlet valves 1-6 for analysis gases.
Vacuum pump foreline connector	For attaching the dry diaphragm roughing vacuum pump hose.
Power cord connector	For setting the power voltage and connecting the analyzer to the power supply.
Compressed air connector	For compressed air supply for the pneumatically actuated, hard seal valves.

Side Panels



Connector	Description
Ethernet port	Port for an Ethernet cable allowing communication between the analyzer and the computer.
RS-232 connector	Used to connect the SmartPrep.
On/Off switch	For turning the analyzer on and off.

Turning the Analyzer On and Off

Turning the Analyzer On

1. Place the computer, monitor and printer ON/OFF switches in the ON position.
2. Place the analyzer ON/OFF switch in the ON position.
3. Turn on the dry diaphragm roughing vacuum pump.



The pump must warm for approximately two hours before performing analyses.

Turning the Analyzer Off



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

It is recommended that the analyzer be turned on at all times. If it does become necessary to turn it off, perform the following steps:

1. Go to **File > Exit** from the analyzer program (or use the **Alt+F4** keyboard shortcut).

If an analysis is in progress, 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

Select **Yes** and the analysis program closes. The analysis continues and data continue to be collected. Queued reports under the printer 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.

Select **No** and the program remains open and the analysis continues to run.

2. Shut down the computer using standard Windows procedure.
3. Place the monitor and printer ON/OFF switches in the OFF position.
4. Place the analyzer ON/OFF switch in the OFF position.
5. Turn off the dry diaphragm roughing vacuum pump.

Software

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

The MicroActive program offers a Windows interface with an easy way to collect, organize, archive and reduce isotherm and store sample information files for later use. The reports can be generated to screen, paper, or exported for use in other programs. Cut and paste graphics, scalable and editable graphs, and customized reports are easily generated. There are two report functions:

- User-defined reports
- MicroActive reports

Report options can be specified when creating the sample information file. When an analysis is performed, data collected 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.

MicroActive Reports

MicroActive reports are automatically generated 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 *Complete*, *Analyzing*, 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. From this window numerous reports are accessible from a dropdown menu, including:

- BET Surface Area
- Langmuir Surface Area
- t-Plot
- Alpha-S Method
- BJH Adsorption
- BJH Desorption
- Dollimore-Heal Adsorption
- Dollimore-Heal Desorption
- Horvath-Kawazoe
- DFT Pore Size
- DFT Surface Energy
- Dubinin-Radushkevich
- Dubinin-Astakhov

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 displayed in the window.

Report Features

- 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.
- Reports can be customized with a choice of fonts and a logo can be added to the report header.

Using the Software

The analysis program operates in the Windows environment and requires familiarity with standard Windows operations, for example, using the mouse, menus and windows. While this manual provides brief instructions for such standard operations, if necessary, refer to Windows documentation or its online help system to clarify any Windows functions. The application uses a standard window and libraries for file selection. Refer to [Libraries](#), page [1-17](#) for details on the File Selector.

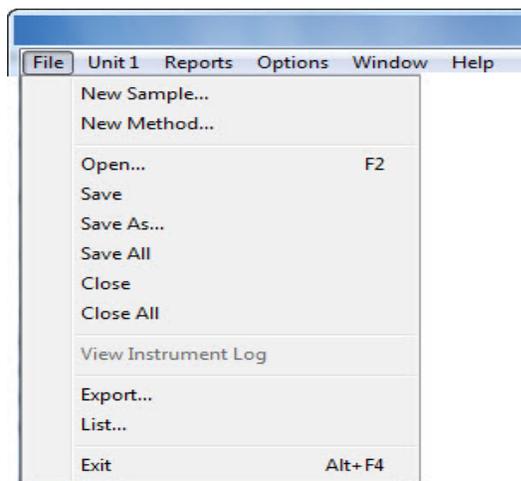
Shortcut Menus

Shortcut menus are available for:

- the instrument schematic when Manual Control is enabled. Refer to [Enable Manual Control](#), page [4-9](#).
- onscreen graphs and tabular reports. Refer to [Report Shortcut Menus](#), page [5-20](#).

Shortcut Keys

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.



Another shortcut method of accessing a menu or function is to use the **Alt** key plus the underlined letter in the menu command. For example, to access the **F**ile 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 underscored letters do not display menus and windows, press the Alt key on the keyboard.

Shortcut Key(s)	Function
F1	Accesses the online operator's manual.
F2	Displays the File Selector screen.
F3	When in the File Selector screen, displays the file search box.
F4	When in the File Selector screen, opens the address bar.
F6	Cascades open windows.
F7	Tiles open windows.
F8	Opens the File Selector allowing you to start a report from a selected .SMP file.
F9	Closes all open reports.
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Shift + F9	Accesses shortcut menu of (1) selected component on instrument schematic, when manual control is enabled or (2) onscreen reports.

Files

File Status and Description

In the **File Selector** window, the **Mic Description** column and the **Mic Status** column display file description and file status, respectively.

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 SmartPrep degasser.
Preparing	Sample information files currently being used in an automatic degas operation. This status is applicable only if using the SmartPrep degasser.

File Name Conventions

For sample information files, a default file name (the next available sequence number) and a default file extension display. For Sample tube, Degas conditions, Analysis conditions, Adsorptive properties and Report options, only a default file extension displays.

The following table lists the default file extensions assigned to program files:

File Type	Default File Name Extension
Alpha-s curve	.ALS
Adsorptive properties	.ADP
Analysis conditions	.ANC
Degas conditions	.DEG
Heat of Adsorption Report	.HOA
Methods	.MTH

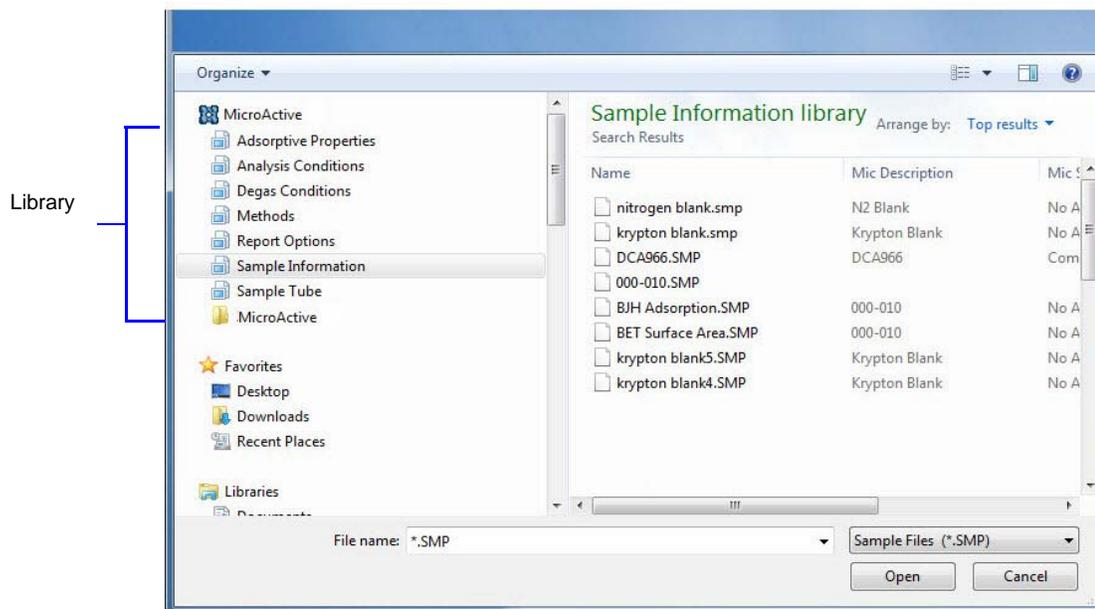
File Type (<i>continued</i>)	Default File Name Extension
Report options	.RPO
Sample information	.SMP
Sample tube properties	.STB
Thickness curve	.THK

The following file types are available when printing or exporting reports:

Report	.REP
Spreadsheet	.XLS
ASCII	.TXT

Libraries

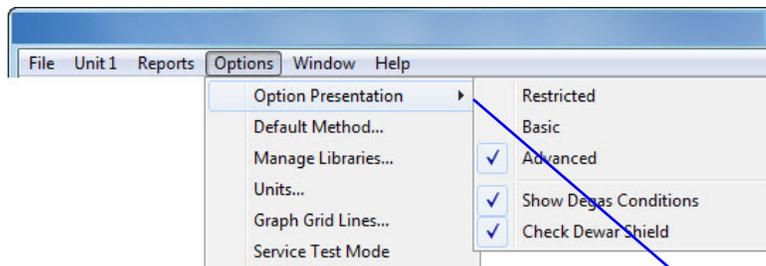
The application contains libraries of the Micromeritics application specific files. The library is located within the File Selector window and can only be viewed within the application. The library provides an easy way to locate and open application specific files. For example, to locate and open a sample information file, click the Sample Information library folder on the left. Then select the .SMP file on the right and click **Open**.



Library Name	Default Directory	File extension
Adsorptive Properties	...\Param	.ADP
Analysis Conditions	...\Param	.ANC
Degas Conditions	...\Param	.DEG
Methods	...\Data	.MTH
Report Options	...\Param	.RPO
Sample Information	...\Data	.SMP
Sample Tube	...\Param	.STB

Menu Structure

All program functions are located on menus accessed from the menu bar. Each menu contains commands and, in some cases, a submenu. A submenu is indicated when the command is followed by an arrow.



Arrow indicates a submenu is available

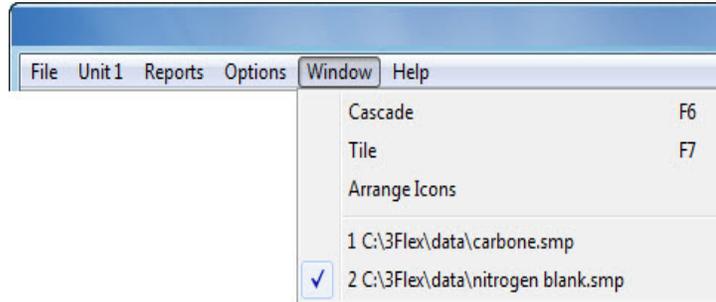
Main Menu Bar

All program functions are accessed from the main menu bar. The following table contains brief menu descriptions and links to additional information on each item:

Menu Item	Description
File	Use to manage files. Refer to FILE MENU , page 3-1 .
Unit [n]	Use to perform analyses, calibrations and other instrument operations. Refer to UNIT MENU , page 4-1 .
Reports	Use to run reports and view the results. Refer to REPORTS MENU , page 5-1 .
Options	Use to edit default method, specify system configuration and data presentation formats. Refer to OPTIONS MENU , page 6-1 .
Window	Use to arrange open windows and display a list of open windows. Refer to Window Menu , page 1-19 .
Help	Use to access this operator's manual, Micromeritics web page, and tutorials. Refer to Help Menu , page 1-20 .

Window Menu

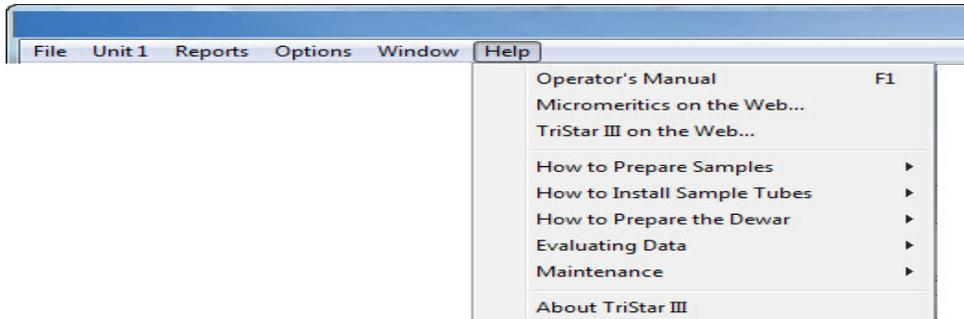
The **Window** menu lists open files and provides the ability to rearrange the way open windows display. A check mark appears to the left of the active window.



Window Menu Option	Description
Cascade	Stacks open windows in a fanned format so that each window title bar is visible. F6 can also be used as a keyboard shortcut.
Tile	Resizes open windows and arranges windows horizontally so that multiple windows can be viewed at once. F7 can also be used as a keyboard shortcut.
Arrange Icons	Arranges the symbols for all minimized windows.

Help Menu

The Help menu provides access to the online operator's manual, the Micromeritics web page, tutorials, and information about the analyzer.



Help Menu Option	Description
Operator's Manual	Provides access to the online operator's manual.
Tutorials	Provides access to instructional tutorials.
Micromeritics on the Web	Provides a link to the Micromeritics web page: www.Micromeritics.com
3Flex on the Web	Provides an Internet link to additional analyzer information: 3Flex on the Web
About 3Flex	Provides the analysis program version number.

Setup Program

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

- Reinstall the application
- 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



If the IP address needs to be changed on the computer connected to an analyzer, reference the Entering Ethernet Settings in Windows 7 tutorial accessed from the Help menu. The IP address for the computer and the IP address specified in the setup program must match.

2. OPERATIONAL PROCEDURES

Introduction

This chapter contains information on:

- working with Interactive Reports
- specifying method defaults
- creating sample files in advanced, basic, and restricted formats
- defining parameter files
- manually entering isotherm data
- preparing samples and performing an analysis
- generating reports
- exporting and listing file contents to the screen, printer, or file, and listing file statistics
- generating graph overlays

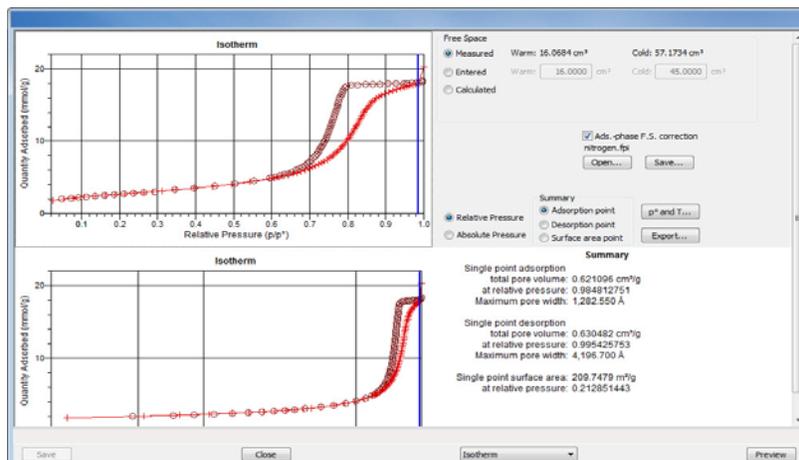
Working with Interactive Reports

When opening a sample file that contains data from a complete or in progress analysis, the interactive reporting feature is enabled. To view a tutorial on interactive reporting, choose one of the following options:

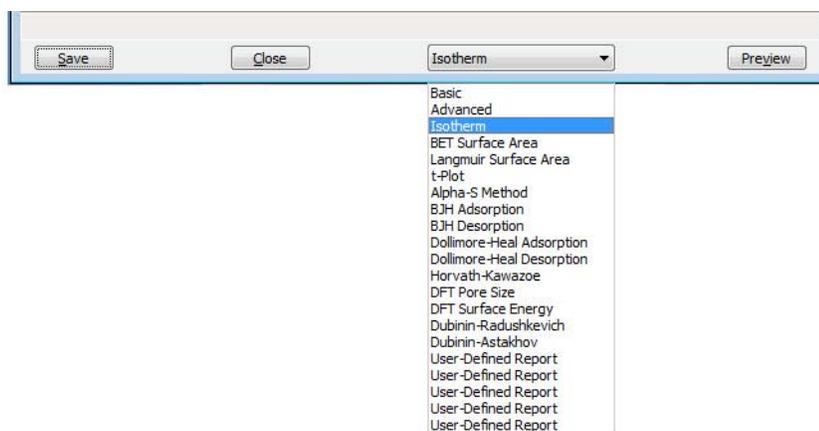
- click the following link: [Working with Interactive Reports](#)
- go to **Help > Tutorials > Working with Interactive Reports**

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



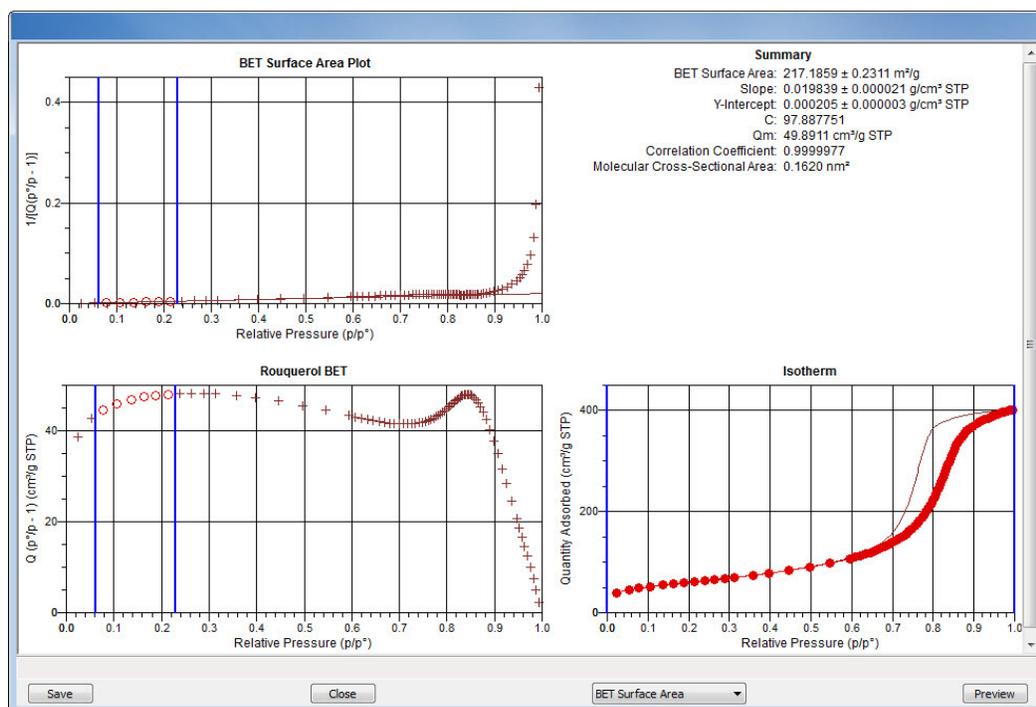
2. To view the plots in either **Relative** or **Absolute** pressure, click the **Relative Pressure** or **Absolute Pressure** option.
3. To view another report, click the **Isotherm** drop-down arrow and make another selection.



The choices in this list allow you to:

- display the sample information window in either select basic or advanced format and modify certain file parameters, or
- select another model from the list and edit the parameters or data range used, or edit the model calculations contained in the plot in various ways.

For example, if **BET Surface Area** is selected, a window similar to the following displays:



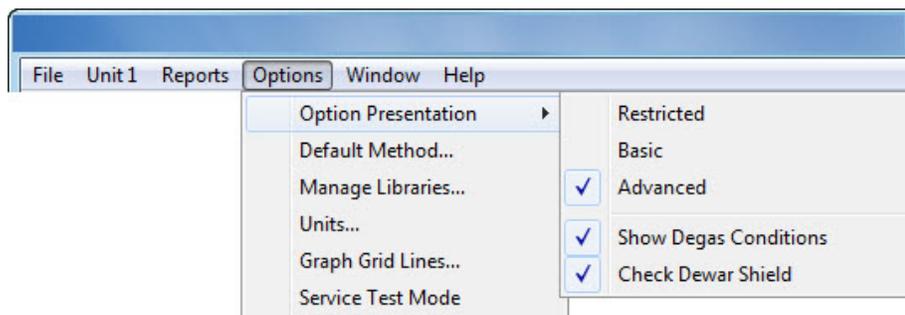
When ranges are edited, the changes are reflected immediately in the plots and the summary data displayed in the window. Some editing options are:

- Drag the blue bars to increase or decrease the range of data included in the plot.
 - Press **CTRL**, then left-click the mouse on a data point in the Isotherm Linear Plot to include or omit the data point from the BET plot.
 - Right-click the mouse 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.
4. After editing the report, save the changes in the sample information file by clicking **Save**.

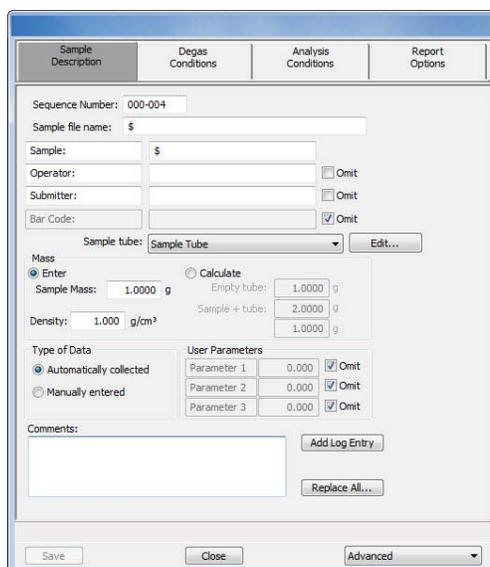
Editing the Default Method

Sample files include the information required by the analyzer to perform analyses and collect data. A method is a template for sample files that contains the parameters to be used for an analysis. The analysis software contains a default method. When a new sample information file is created, all the parameters are filled with the values in the default method. They can then be edited if necessary.

1. Go to **Options > Option Presentation** and select **Advanced** on the popup menu. Ensure a checkmark appears to the left of **Advanced**.



2. Go to **Options > Default Method**.

A screenshot of the 'Default Method' dialog box in a software application. The dialog has several tabs: 'Sample Description', 'Degas Conditions', 'Analysis Conditions', and 'Report Options'. The 'Sample Description' tab is active. It contains fields for 'Sequence Number' (000-004), 'Sample file name' (\$), 'Sample' (\$), 'Operator', 'Submitter', and 'Bar Code'. There are 'Omit' checkboxes for 'Operator', 'Submitter', and 'Bar Code'. Below these is a 'Sample tube' dropdown menu set to 'Sample Tube' with an 'Edit...' button. The 'Mass' section has radio buttons for 'Enter' (selected) and 'Calculate'. Under 'Enter', there are fields for 'Sample Mass' (1.0000 g) and 'Density' (1.000 g/cm³). Under 'Calculate', there are fields for 'Empty tube' (1.0000 g), 'Sample + tube' (2.0000 g), and another 'Sample + tube' (1.0000 g). The 'Type of Data' section has radio buttons for 'Automatically collected' (selected) and 'Manually entered'. The 'User Parameters' section has three rows: 'Parameter 1' (0.000), 'Parameter 2' (0.000), and 'Parameter 3' (0.000), each with an 'Omit' checkbox. At the bottom, there are 'Save', 'Close', and 'Advanced' buttons, along with an 'Add Log Entry' button and a 'Replace All...' button.

3. In the **Sequence Number** text box, specify an optional default alphanumeric file sequence string. This field must contain a minimum of 3 numbers. As files are created, this number is incrementally sequenced as a part of the file name and will also display in the **Sample** text box when a sample file is created.
4. 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 and will also display in the **Sample** text box when a sample file is created. The \$ character must remain in this field.
5. In the text box to the right of the **Sample** field, enter a format for the default sample identification. Include the \$ character to automatically include the contents of the **Sequence Number** field and **Sample file name** field as part of the sample identification.

6. Enter default **Operator**, **Submitter**, and **Bar Code** identification information in the respective text boxes. This information will display in the **Sample Description** tab of new sample information files.



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

7. To specify default sample tube parameters click **Edit** to the right of the **Sample tube** dropdown list. Refer to [Sample Tube](#), page **2-14** for instructions on editing and saving the sample tube parameters to make them available in the **Sample tube** dropdown list.
8. In the **Mass** group box, indicate if mass is to be manually entered by the operator (**Enter**) or calculated by the system (**Calculate**). Refer to [Mass group box](#), page **3-12**.
9. 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.
10. The optional user-defined fields in the **User Parameters** group box may be used to enter and track information from another instrument or source, along with other statistical process control (SPC) data. To omit these fields in sample files, select the **Omit** checkbox to the right of the field. Refer to [Sample Information Files](#), page **3-10**.
11. Use the **Comments** text box to enter notes about the **Method**.
12. After completing the **Sample Description** window, select the parameter tabs to edit other sample information file parameters. The saved parameter defaults become the defaults for new sample files. Refer to [Defining Parameter Files](#), page **2-14**.
13. Click **Save**, then click **Close**.

Defining Sample Information Files

A sample information file must be created for each analysis. This file can be created prior to or at the time of analysis. The sample information file identifies the sample, guides the analysis, and specifies report options.

A sample information file may be created in Advanced, Basic, or Restricted format.

Format	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 the entire sample information file in a single window with no tabs across the top. This option is used once the parameter files have been created. The previously entered or default parameter files are then accessible using dropdown lists.
Restricted	Displays the sample information file in a single window similar to the Basic format with certain functions disabled. A password must be entered when the restricted format is selected. That same password must be entered to exit the format. This format is typically used in laboratories where analysis conditions must remain constant, for example, in the pharmaceutical industry. The Advanced format option is not available from the Restricted format.

Creating Sample Files in Advanced Format

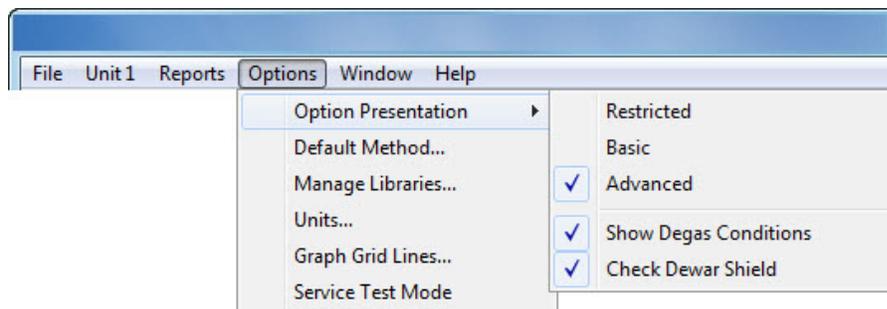
The values specified in the parameter portions of the default sample file (Degas Conditions, Analysis Conditions, and Report Options) are saved as the defaults for new sample files. To navigate from one set of parameters to another, select the parameter tab at the top of the window.

- Sample Tube files are created on the Sample Description tab.
- Adsorptive Properties files are created on the Analysis Conditions tab.



The Degas Conditions tab displays only if enabled in *Options > Option Presentation > Show Degas Conditions*.

1. Ensure that the **Advanced** option is checked in the *Options > Option Presentation* menu.

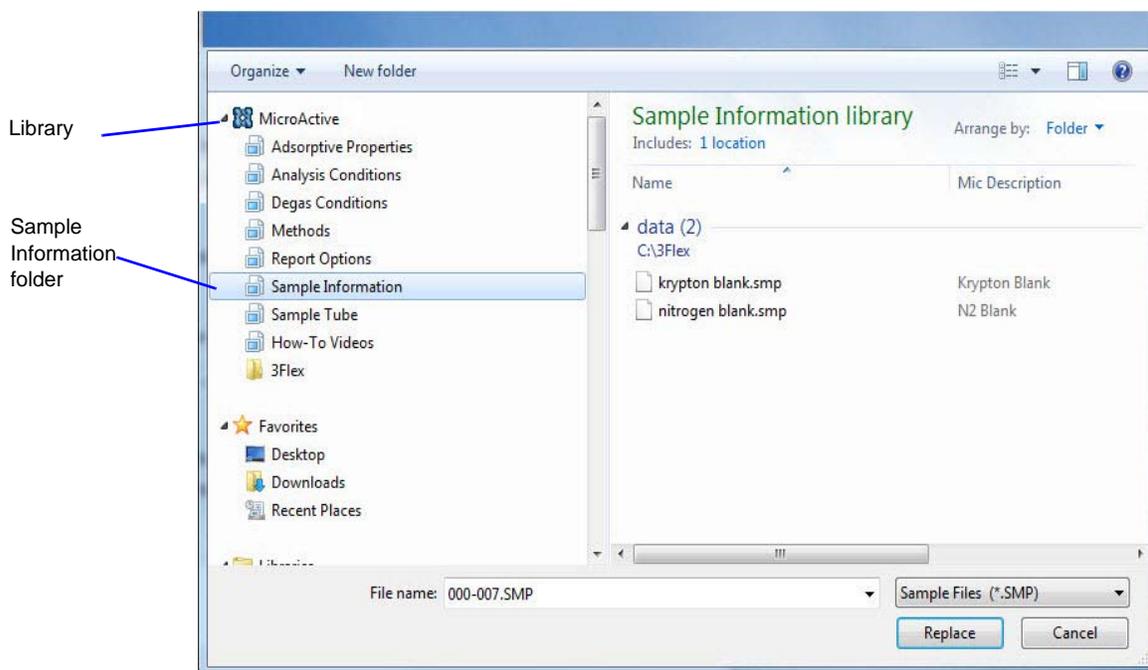


2. Go to **File > New Sample**.

Some text boxes may have different field labels if renamed or may not display if omitted in Default Method.

3. Select a **Method** from the **Method** dropdown list.
4. Enter a sample description in the **Sample** text box.
5. Enter **Operator**, **Submitter**, and **Bar Code** identification information in the respective text boxes. Some text box fields may have been renamed or may not display if modified in **Options > Default Method**.
6. In the **Sample Tube** dropdown list, select a sample tube from the list or click **Edit** to specify a sample tube description, empty tube properties for calculated free space, isothermal jacket, filler rod, and vacuum seal type. The text entered into the **Description** field will display in the **Sample Tube** dropdown list of the previous window. Click **OK** to return to the previous window. Refer to [Sample Tube](#), page 2-14.
7. In the **Mass** group box, indicate if mass is to be manually entered by the operator (**Enter**) or calculated by the system (**Calculate**). Refer to [Mass group box](#), page 3-12.
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. Select **Automatically collected** for all sample runs where the data are collected. Select **Manually entered** when another sample has been run on a different instrument or different model instrument so that data can be analyzed or used for comparison. If **Manually entered** is selected, the data are entered in the Isotherm interactive report. Refer to [Manually Entering Isotherm Data in a Sample File](#), page 2-23.

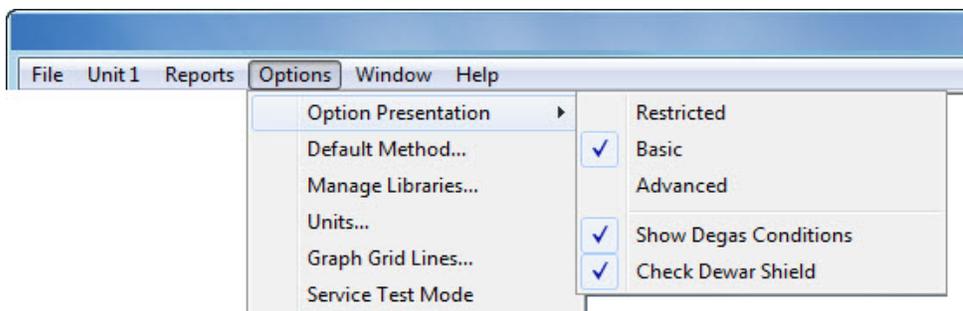
9. If SPC (Statistical Process Control) information is to be reported, enter appropriate information in the **User Parameters** group box. These are user-definable parameters that can be entered and tracked along with other statistical process control data.
10. Enter any pertinent information about the sample information file in the **Comments** text box. Comments are displayed in the report header.
11. Click the **Add Log Entry** button to enter notes for the instrument log report. Create entries that cannot be recorded automatically through the application software. For example, record that the port filter was changed.
12. To auto-populate fields from another .SMP file, click the **Replace All** button and select a .SMP file that contains the desired parameters. Select the file and click **Replace**.
13. After completing the **Sample Description** window, click the parameter tabs to edit other sample information file parameters. See [Defining Parameter Files](#), page 2-14.
14. Click **Save**, then click **Close** to save the file with the default file name. To save as a different file name, go to **File > Save As** and enter a new file name. The file can later be retrieved from the Sample Information folder in the library.



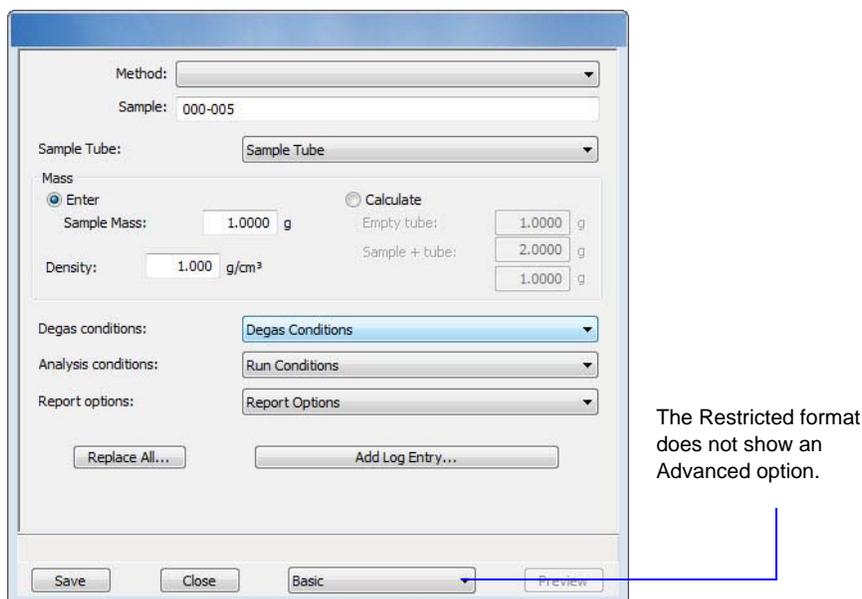
Creating Sample Files in Basic or Restricted Format

The Basic and Restricted formats use predefined parameter files to create a sample information file. Refer to [Sample Information Files](#), page 3-10.

- When using the Basic format, switch to the Advanced format to edit parameter file values.
 - When using the Restricted format, parameter files cannot be edited.
1. Ensure that the **Basic** or **Restricted** option is checked in the *Options > Option Presentation* menu.

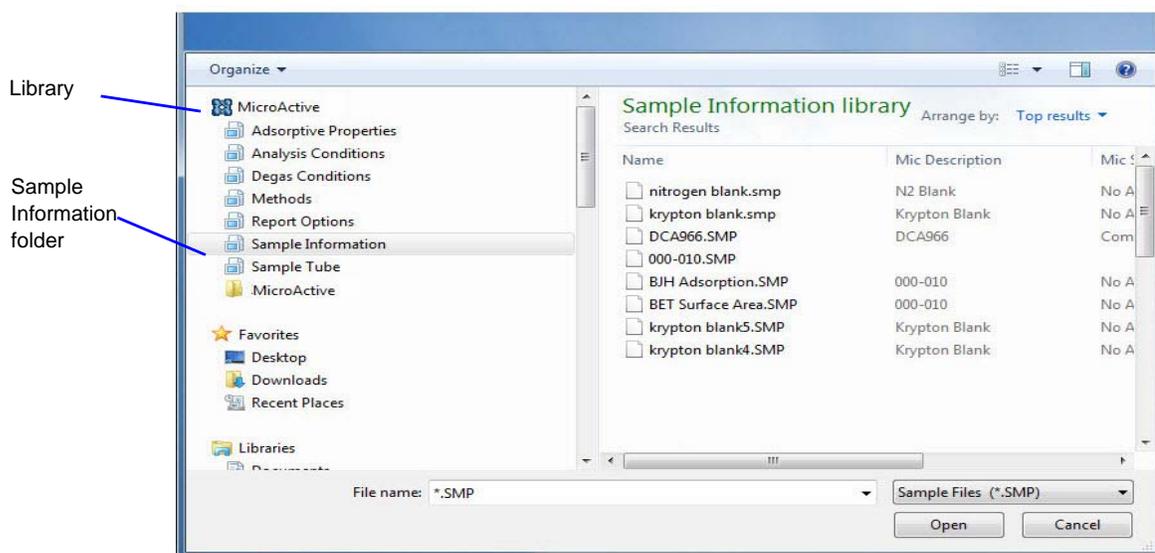


2. Go to *File > New Sample*.
3. Select a Method from the **Method** dropdown list. Refer to [Editing the Default Method](#), page 2-3.

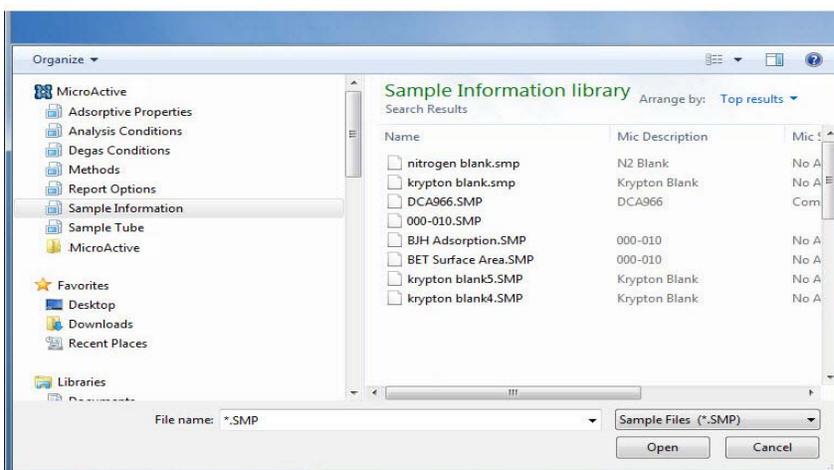


4. In the **Sample** field, enter a sample description.
5. Select a sample tube from the **Sample Tube** dropdown list.

- In the **Mass** group box, indicate if mass is to be manually entered by the operator (**Enter**) or calculated by the system (**Calculate**). Refer to **Mass group box**, page 4-10.
- Click the dropdown arrows to select default parameter files for **Degas conditions**, **Analysis conditions**, and **Report options**.
- To auto-populate fields from another .SMP file, click the **Replace All** button and select a .SMP file that contains the desired parameters. Select the file and click **Replace**.
- Click the **Add Log Entry** button to enter notes for the instrument log report. Create entries that cannot be recorded automatically through the application software. For example, record that the port filter was changed.
- Click **Save**, then click **Close**. The file can be retrieved later from the Sample Information folder in the library.



Opening Sample Information Files

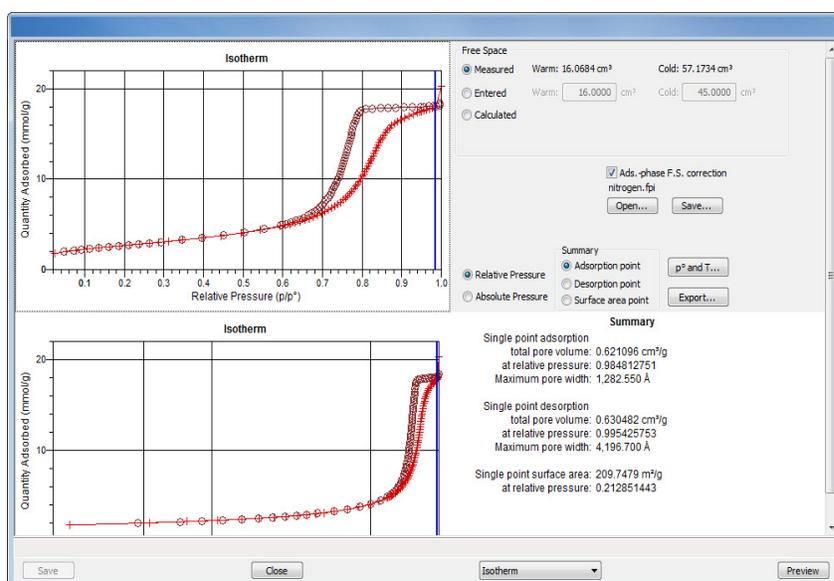


When working with an existing file, it is recommended that a copy of the file be used rather than the original.

Columns in the File Selector window can be sorted by clicking the column header.

Opening Files with a Status of Complete, Analyzing, or Entered

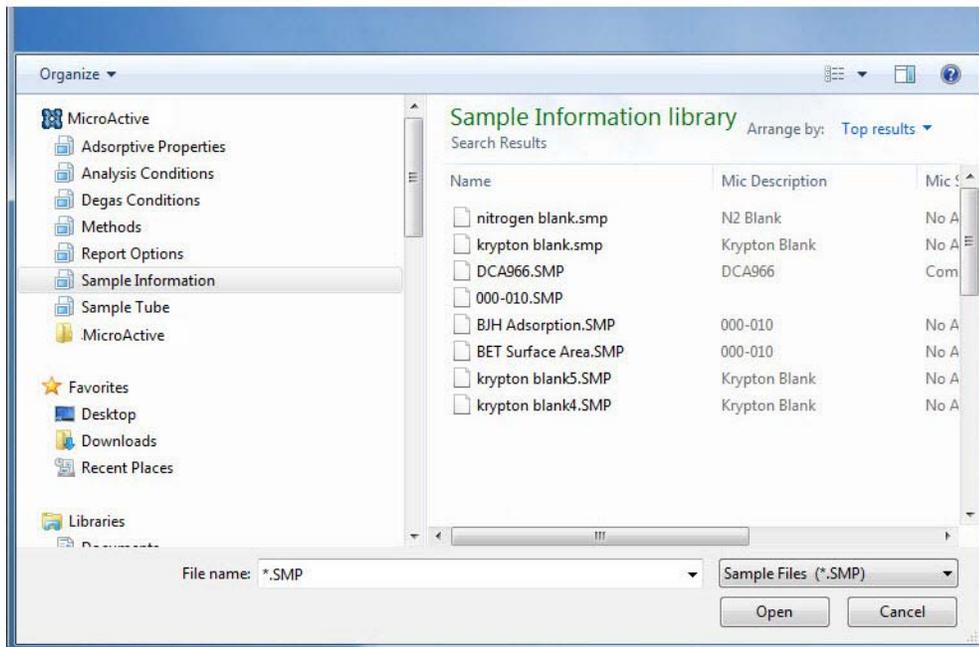
1. Go to **File > Open** or use the **F2** keyboard shortcut.
2. From the Sample Information library folder, select a .SMP file with a status of *Complete*, *Analyzing*, or *Entered* and click **Open** (or double click the file name). The interactive reporting window displays. Refer to [Working with Interactive Reports](#), page 2-1.



To view a tutorial on interactive reporting, go to **Help > Tutorials > Working with Interactive Reports**.

Opening Files with a Status of *Preparing*, *Prepared*, or *No Analysis*

1. Go to **File > Open** or use the **F2** keyboard shortcut.
2. From the Sample Information library folder, select a .SMP file with a status of *Preparing*, *Prepared*, or *No Analysis* and click **Open** (or double click the file name).



3. The **Sample Description** window displays in the specified format (Advanced, Basic, or Restricted). The following window is shown in Advanced format.

The screenshot shows the "Sample Description" window in Advanced format. The window has tabs for "Sample Description", "Degas Conditions", "Analysis Conditions", and "Report Options". The "Sample Description" tab is active. The fields are:

- Method: Default
- Sample: 000-005
- Operator: (empty)
- Submitter: (empty)
- Bar Code: (empty)
- Sample tube: Sample Tube (dropdown menu)
- Mass: Enter (selected) / Calculate (unselected)
- Sample Mass: 1.0000 g
- Empty tube: 1.0000 g
- Density: 1.000 g/cm³
- Sample + tube: 2.0000 g
- Type of Data: Automatically collected (selected) / Manually entered (unselected)
- User Parameters: Parameter 1: 0.000, Parameter 2: 0.000, Parameter 3: 0.000
- Comments: (empty text area)
- Buttons: Add Log Entry, Replace All...
- Bottom buttons: Save, Close, Advanced (dropdown menu), Preview

Defining Parameter Files

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

- Sample Tube Files .STB file extension
- Degas Conditions .DEG file extension
- Analysis Conditions .ANC file extension
- Adsorptive Properties .ADP file extension
- Report Options .RPO file extension
- Method .MTH file extension

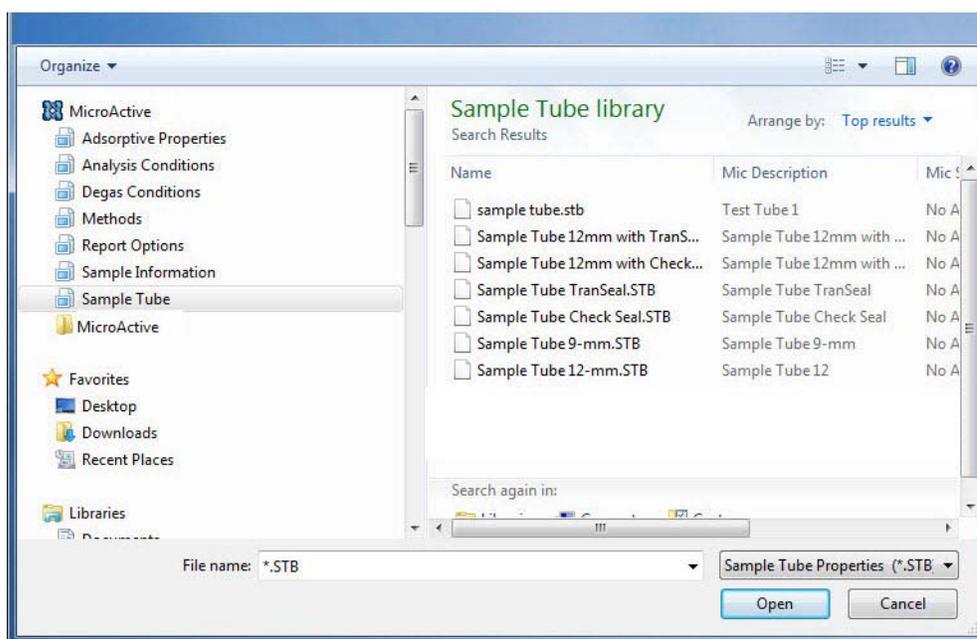
Default parameter files can be used for multiple analyses without having to re-enter the values each time an analysis is performed.

Predefined parameter files are included with the program and can be edited as needed or new parameter files can be created. The **Replace** button can be used to overwrite values from an existing file. Changes can be made as needed to the new file while the original file remains unchanged.

Sample Tube

Sample Tube files specify information about the sample tube.

1. Go to **File > Open**. Select the **Sample Tube** library folder and enter a file name in the **File name** field.



2. Click **Open**.

3. Click **OK** when prompted to create a new file.
4. Enter a description of the sample tube in the **Description** field. This description displays in the **Sample tube** dropdown list on the **Sample Description** tab.

5. If using calculated free space, enter the warm free space, cold free space and non-ideality factor; or click **Load from Sample File** to import the information from an existing sample information file.
6. Indicate if an isothermal jacket and/or filler rod will be used by selecting the **Use isothermal jacket** checkbox and/or **User filler rod** checkbox. 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.
7. Select the vacuum seal type to be used.
8. Click **Save**, then click **Close**.

Degas Conditions

Degas Conditions files contain degassing information for sample preparation.

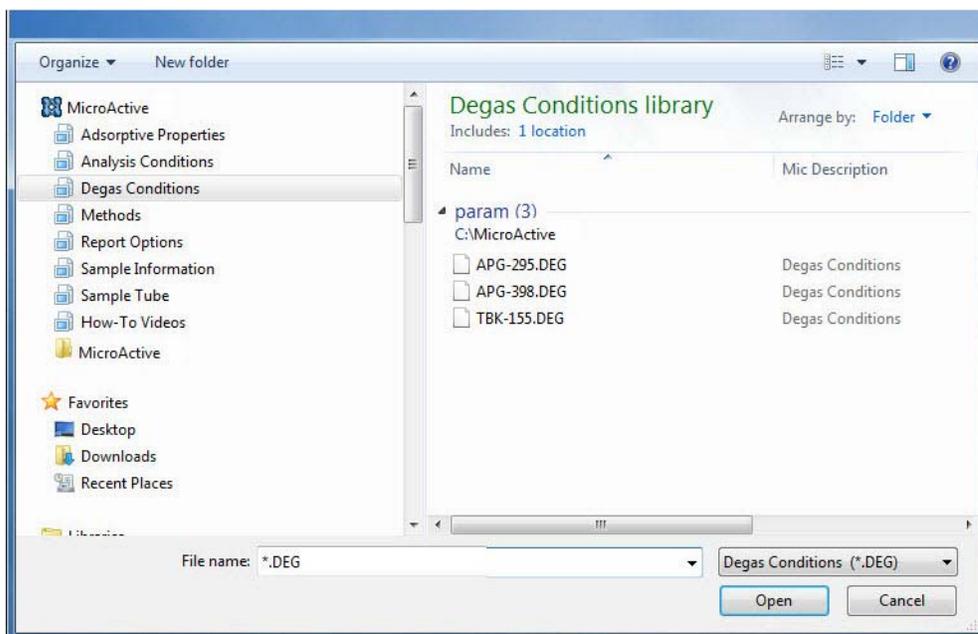


Degassing is a required step in preparation for the analysis; however, the Degas Conditions tab is only applicable if using the SmartPrep Degasser. This section contains degassing instructions that will be sent to the SmartPrep Degasser equipment. Refer to [Preparation button](#), page 3-21 for in situ degassing.

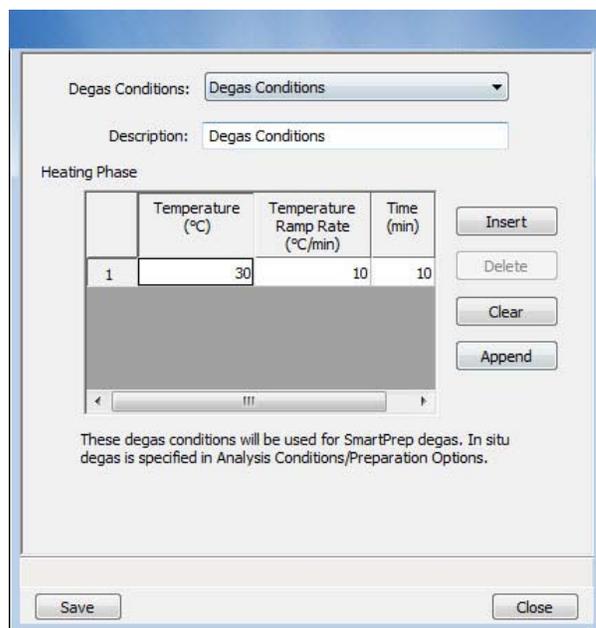


The Degas Conditions tab only displays if enabled in *Options > Option Presentation > Show Degas Conditions*.

1. Go to **File > Open**. Select the **Degas Conditions** library folder and enter a file name in the **File name** field.



2. Click **Open**.
3. Click **OK** when prompted to create a new file.



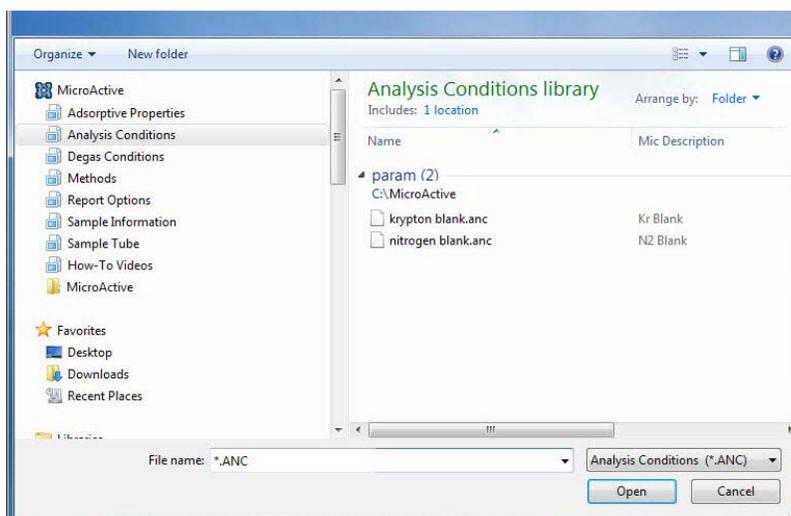
4. To overwrite degas conditions with parameters from another Degas Conditions file, click the **Degas Conditions** dropdown arrow and select a file from the list or click **Browse**, then locate and select the .DEG file containing the new parameters and click **Open**.

- Use the **Insert** button to enter up to three stages of degassing (temperature, ramp rate, and time). When using the SmartPrep degasser, the maximum temperature that can be entered is 450 °C.
- Click **Save**, then click **Close**.

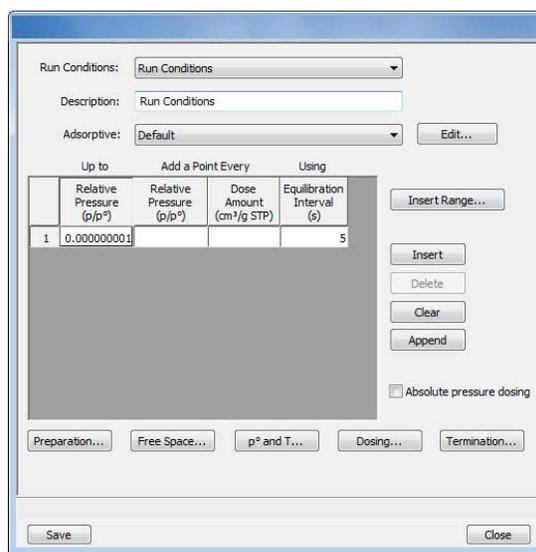
Analysis Conditions

Analysis conditions specify the data used to guide an analysis.

- Go to **File > Open**. Select the **Analysis Conditions** library folder and enter a file name in the **File name** field.



- Click **Open**.
- Click **OK** when prompted to create a new file.



4. To overwrite analysis conditions with parameters from another Analysis Conditions file, either click the **Run Conditions** dropdown arrow and select a file from the list or click **Browse** then locate and select the .ANC file containing the new parameters and click **Open**.
5. To overwrite adsorptive properties with parameters from another Adsorptive Properties file, click the **Adsorptive** dropdown arrow and select an adsorptive from the list or click **Browse**, then locate and select a .ADP file containing the new parameters and click **Open**. Refer to [Adsorptive Properties](#), page 2-19 and [Analysis Conditions Files](#), page 3-16.
6. To enter starting and ending relative pressure points, click the **Insert Range** button
7. To specify pressure targets in mmHg, mbar, or kPa instead of relative pressure, select the **Absolute pressure dosing** checkbox. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, H₂ adsorption on carbon at liquid nitrogen temperature.
8. Click the following buttons to specify:

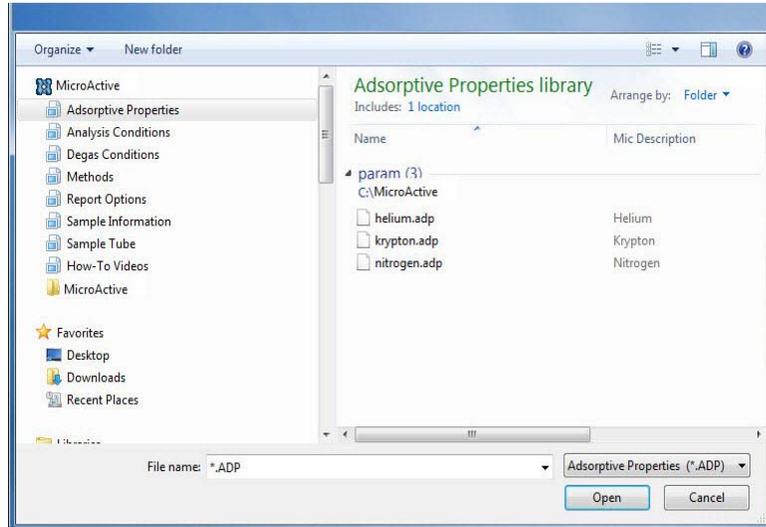
Button	Use to Specify...
<i>Preparation</i>	evacuation rate/time/level, leak test and time values, elevator prompts, and in situ degassing
<i>Free space</i>	how the free space is to be measured
<i>p° and T</i>	how the saturation pressure (Po) is to be measured or calculated and the analysis bath temperature
<i>Dosing</i>	options for absolute and/or relative pressure tolerance
<i>Termination</i>	backfill options after analysis

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

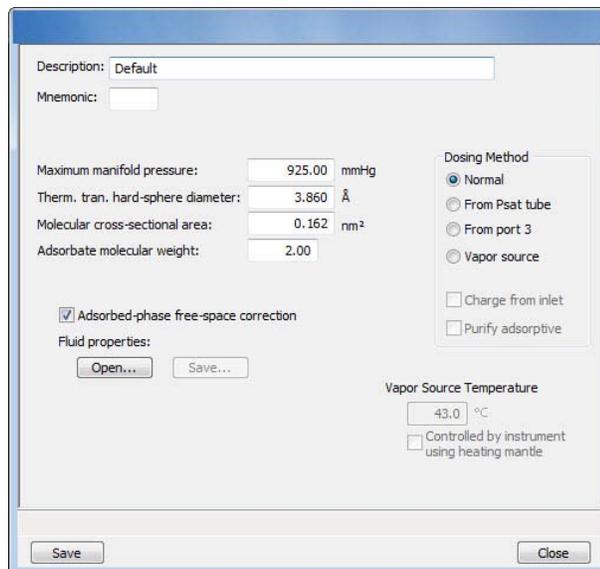
Adsorptive Properties

Adsorptive properties provide the properties of the fluid used for the analysis.

1. Go to **File > Open**. Select the **Adsorptive Properties** library folder and enter a file name in the **File name** field.



2. Click **Open**.
3. Click **OK** when prompted to create a new file.
4. Enter a description of the adsorptive in the **Description** text box (for example, the gas and the temperature). When saved, this description will display in the **Adsorptive** dropdown list of the Analysis Conditions tab.



5. Enter the mnemonic for the Adsorptive gas (for example, **N2**) in the **Mnemonic** text box.

6. Enter information in the following text boxes:
 - **Maximum manifold pressure** - the highest pressure that the manifold will be dosed. To avoid damage to the instrument, this number is limited to 925 mmHg. Low pressure sources, such as vapors, will require lower numbers.
 - **Therm. tran. hard-sphere diameter** - an estimate of molecular size used in calculating the thermal transpiration correction.
 - **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.
 - **Adsorbate molecular weight** - the molecular weight is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot.
7. 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. It should be deselected for blank tube analyses.
8. To import parameters from a Fluid Properties file, click **Open**, then locate and select the .FPI file containing the new parameters and 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.
9. In the **Dosing Method** group box, select the source to dose the adsorptive:
 - **Normal** - dose from a pressurized tank of gas attached to a gas inlet port.
 - **From Psat tube** - the Psat tube is filled with condensed adsorptive and dosed from the Psat tube. This is typically used for Krypton. The instrument will determine the maximum pressure that can be dosed based on the analysis temperature and the saturation pressure information in the Fluid Properties.
 - **From Port 3** - the tube attached to sample port 3 is filled with condensed adsorptive and dosed from Port 3. The instrument will determine the maximum pressure that can be dosed based on the analysis temperature and the saturation pressure information in the Fluid Properties.
 - **Vapor Source** - a container of condensed vapor is attached to the Psat port in place of the Psat tube, and is dosed from the Psat port.
 - **Charge from inlet** - select to have the tube automatically charged with condensate from a gas inlet port after the Dewar is raised.
 - **Purify adsorptive** - select to have the condensate in the tube purified after charging by evacuating the gas over the condensate. If **Charge from inlet** is selected, you may choose **Purify adsorptive** to have noncondensing contaminants automatically removed from the dosing 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.

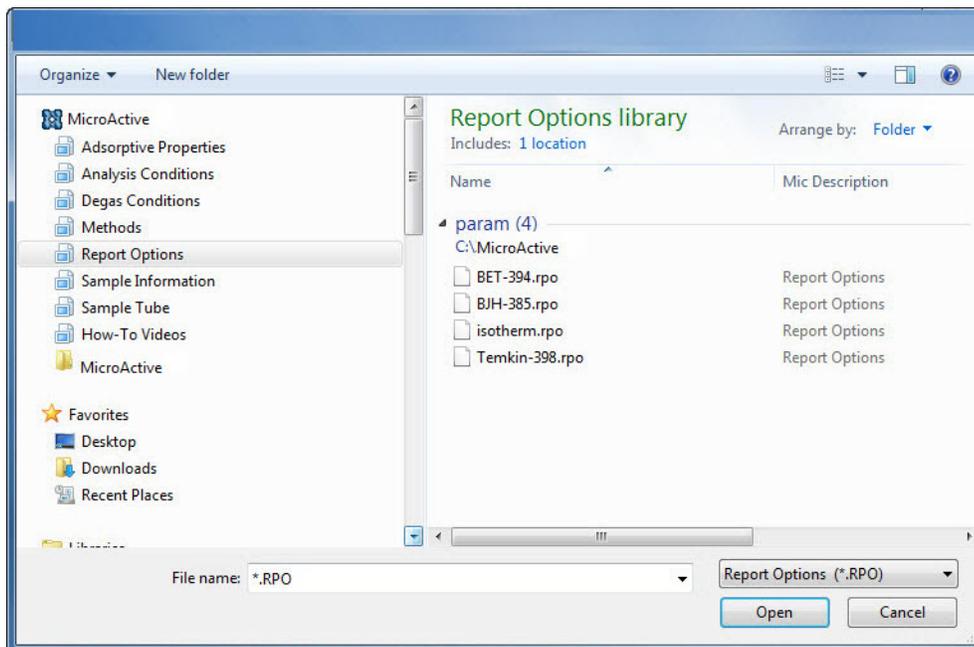
10. If **Vapor Source** is selected in the **Dosing Method** group box, the **Vapor Source Temperature** must be entered whether it is to be controlled by the instrument or not. The instrument will determine the maximum pressure that can be dosed based on this temperature and the saturation pressure information in the Fluid Properties.
11. 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.
12. Click **Save**, then click **Close**.

Report Options

Report Options files specify the type of reports that will be generated from an analysis or from manually entered data. They also contain report details, for example, axis scale, axis range, and column headings. Report options files may contain tabular reports, plots, or both, as well as user-defined report tables.

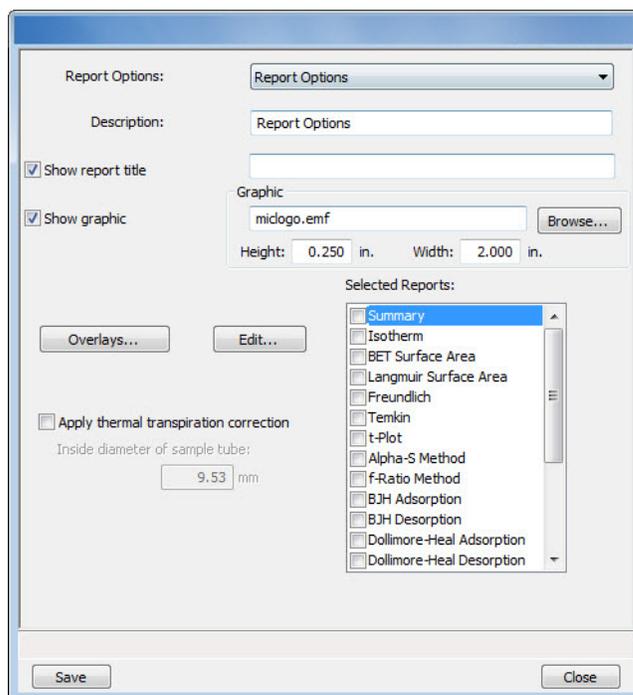
Report Options files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis. Refer to [Generating Graph Overlays](#), page 2-60.

1. Go to **File > Open**. Select the **Report Options** library folder and enter a file name in the **File name** field.



2. Click **Open**.

3. Click **OK** when prompted to create a new file.



4. To overwrite report options with parameters from another Report Options file, click the **Report Options** dropdown arrow and select a file from the list or click **Browse**, then locate and select the .RPO file containing the new parameters and click **Open**.
5. To display a title on the report header, select the **Show report title** checkbox and enter the report title in the text box.
6. To display a graphic on the report header, select the **Show report title** checkbox and enter the report title in the text box. Click the **Browse** button to locate a .BMP or .EMF file. Specify the graphic size in the **Height** and **Width** text fields.
7. The **Selected Reports** list box displays the reports that may be generated.
 - Select the checkbox to the left of the report to include in this file.
 - To specify report options, highlight the report in the **Selected Reports** list box and click **Edit**. Make changes as necessary. Click **OK**.
8. Click **Save**, then click **Close**.

For information on the **Overlays** and **Import** buttons, see [Generating Graph Overlays](#), page 2-60.

Manually Entering Isotherm Data in a Sample File

This process allows the manual entry of pressure data by importing or pasting data from a sample file with a *Complete* status.

Importing Manually Entered Isotherm Data from the Interactive Isotherm Window

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

ASCII text file format rules

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

Relative Pressure
 Absolute Pressure (mmHg)
 Absolute Pressure (kPa)
 Absolute Pressure (mbar)
 Quantity Adsorbed (mmol/g)
 Quantity Adsorbed (cm³/g STP)
 Quantity Adsorbed (cm³/g STP)

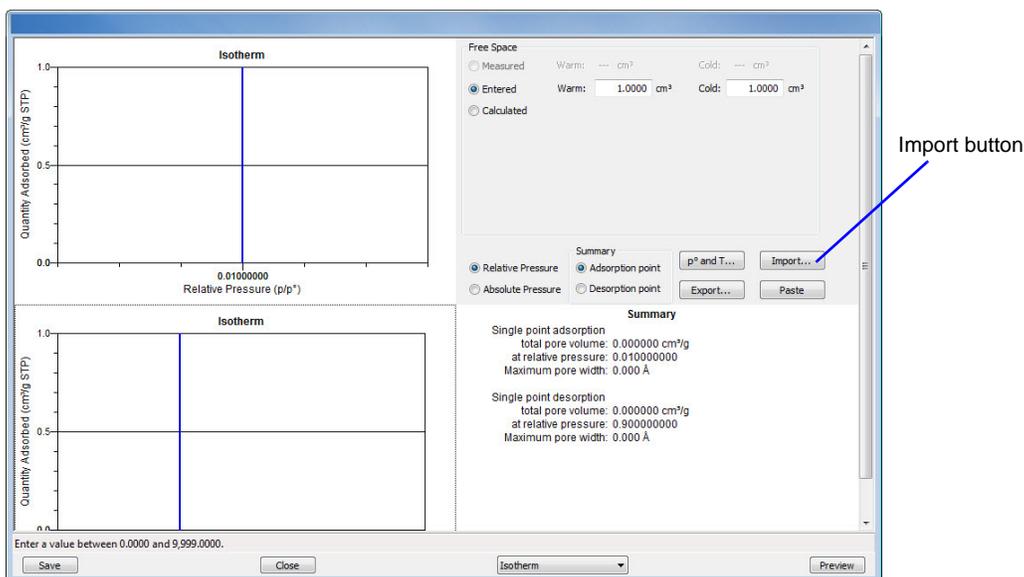
Sample ASCII text file

Relative Pressure	Quantity Adsorbed (cm ³ /g STP)
0.00156203	21.5917
0.0453336	42.9898
0.0667632	46.1971
0.0944588	49.4713
0.105895	50.6657
0.128984	52.9288
000-000 : Desorption	
Relative Pressure	Quantity Adsorbed (cm ³ /g STP)
0.969366	403.793
0.956297	402.889
0.944633	402.042
0.932647	401.191

To import the ASCII text file:

1. Go to **File > New Sample** and open a new sample information file.
2. On the **Sample Description** window, select **Manually entered** in the **Type of Data** group box.
3. In the dropdown list at the bottom of the **Sample Description** window, select **Isotherm**.

4. Resize the isotherm window until the **Import** button displays.

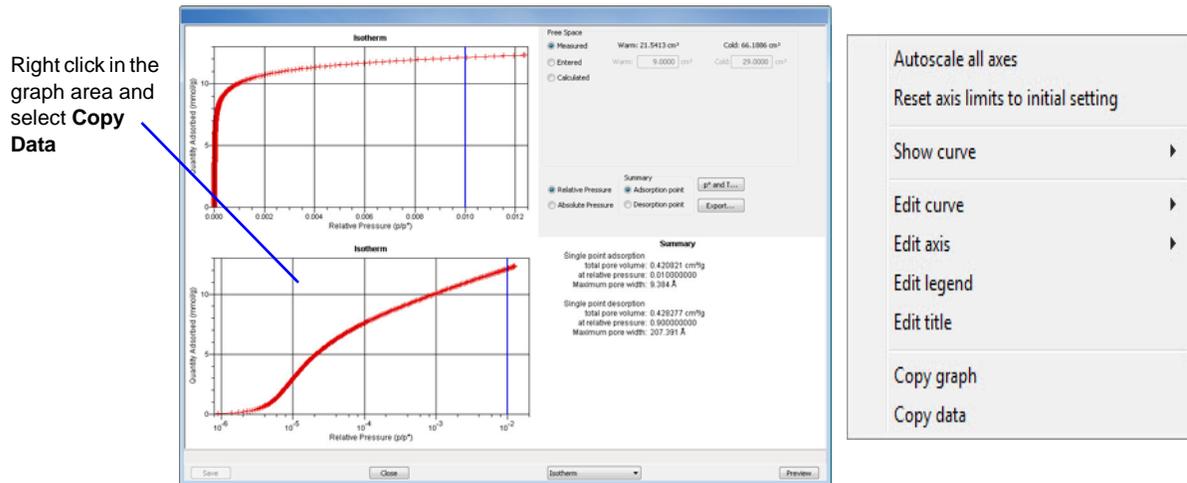


5. Ensure that all parameter fields are set appropriately then click **Import**.
6. On the File Selector window, locate and select the .TXT file and click **Open**. The isotherm data from the original sample file is imported and displays in the new sample file. If an error message appears instead, verify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

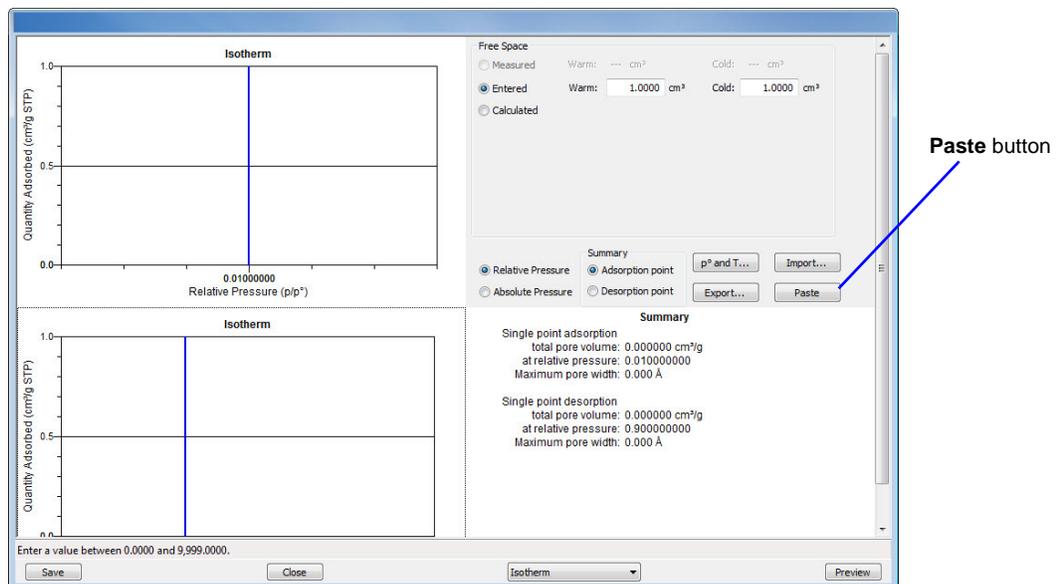
Copying / Pasting Manually Entered Isotherm Data

1. Go to **File > Open [.SMP file]** and select the sample information file that contains the isotherm data to be copied and pasted. This file must have a *Complete* status.
2. Click **Open**. The file will open to the interactive isotherm report window.

- Right click in the graph area of the interactive reports window and select **Copy Data**. This will copy the data from the active file to the clipboard.



- Go to **File > New Sample** and open a new sample information file.
- On the **Sample Description** window, select **Manually entered** in the **Type of Data** group box.
- In the dropdown list at the bottom of the window, select **Isotherm**.
- Resize the interactive isotherm window until the **Paste** button displays.



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

Preparing for Analysis

The following table outlines the tasks to properly prepare for an analysis and the location of the task procedure. It is recommended to perform the tasks in the following order:

Task	Name and Location
Clean the sample tube	Cleaning and Labeling Sample Tubes , page 2-26
Create the sample file	Defining Sample Information Files , page 2-6
Weigh the sample	Determining the Sample Mass , page 2-28
Degas the sample	Degassing the Sample , page 2-30
Load sample on sample port	Installing the Sample Tube , page 2-30
Fill Dewar and check LN2 level	Filling and Installing the Analysis Dewar , page 2-41

Cleaning and Labeling 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 table are recommended.

Materials Supplied by Micromeritics	Materials Supplied by User
<ul style="list-style-type: none"> • Sample tube • Filler Rod • Sample tube brush • Stopper for sample tube • Sample tube rack • Sample weighing support • Sample data worksheet (copied from Appendix A of this manual) 	<ul style="list-style-type: none"> • Drying oven • Ultrasonic cleaning unit • Detergent • Rubber gloves or lint-free cloth • Acetone or isopropyl alcohol • Safety glasses • Waste container • Analytical balance • Pipe cleaners

1. Preheat drying oven at 110 °C.
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). Ensure the detergent is dissolved before placing the sample tubes and filler rods into the water. If too much detergent is used, it may be difficult to rinse from the sample tubes.

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 and remove the sample tubes and filler rods from the unit.
6. Clean the interior of the sample tubes with the brush supplied with the system.
7. Rinse the sample tubes and filler rods thoroughly with hot water. Then 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.

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. Wipe a rubber stopper with a lint-free cloth.
11. Label the sample tube and stopper for identification.

Determining the Sample Mass

Analysis results are expressed in units of surface area per gram of sample; therefore, it is important the sample mass be known. The mass is best calculated as:

- weigh the empty Sample Tube Set (sample tube and stopper, check seal or TranSeal) *before degas*
- weigh the Sample Tube Set with sample *before degas* and subtract from the weight of the empty Sample Tube Set
- weigh the Sample Tube Set with sample *after degas* and subtract from the weight of the empty Sample Tube Set
- weigh the Sample Tube Set with sample *after analysis* and subtract from the weight of the empty Sample Tube Set

A [Sample Data Worksheet](#) for recording the weights and calculating the mass is included in Appendix A. Make copies as needed.

Use a copy of the Sample Data Worksheet to record the following:

1. Record the *Sample Tube Identification*.
2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero.

3. Place the sample tube set on the balance.



4. Record the stabilized weight on the Sample Data Worksheet as *[A] Mass for empty sample tube set*. Remove the sample weighing support and 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 and slowly pour the sample into the container.
6. Remove the rubber stopper (check seal or TranSeal) from the sample tube.
7. Use the sample tube funnel (provided in the accessories kit) and pour the sample from the weighing container into the sample tube.

Funnel



8. Replace the rubber stopper (check seal or TranSeal).
9. 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)*.
10. 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)*.

Degassing 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. Refer to **ORDERING INFORMATION**, page **9-1** for ordering information.

If using the SmartPrep degasser, go to **Unit [n] > Degas** and degas the sample using menu commands and information entered in the **Degas Conditions** window. Refer to the SmartPrep operator's manual for operating instructions.

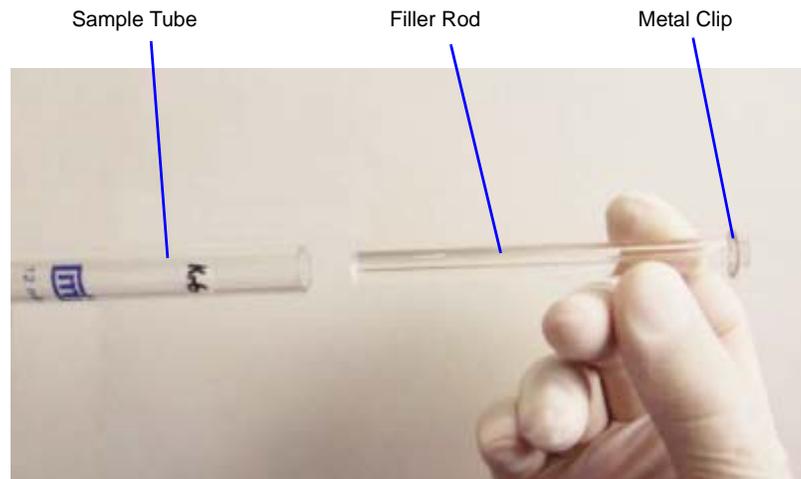
After degassing is complete, perform the following steps:

1. Weigh the sample tube set containing the sample and 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)*.

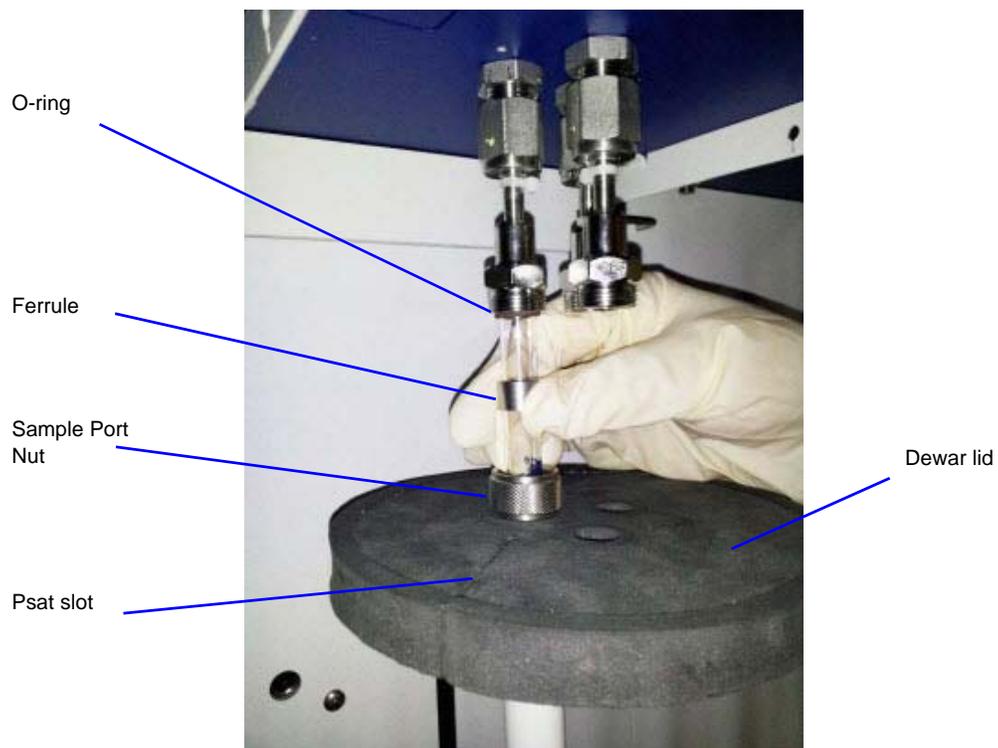
Installing the Sample Tube

Repeat the following steps for each sample to be installed. Up to three sample tubes can be installed. To install a sample tube to a port:

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.

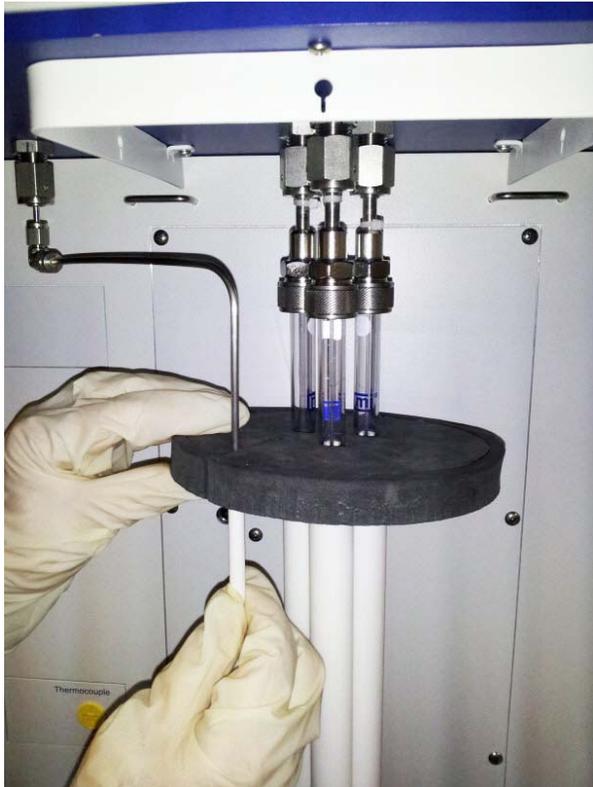


1. Loosen the connector nut on the Psat tube and rotate it out of the way.
2. Position the Dewar lid so that the slot for the Psat tube is on the left between ports 1 and 2.



3. Insert the sample tube through one of the holes in the Dewar lid.
4. Place the sample port nut, ferrule and O-ring onto the sample tube stem.

5. Insert the sample tube to the analysis port and ensure it is completely in the port. Securely screw the sample port nut onto the analysis port and hand tighten the nut.
6. Repeat for each sample tube.
7. Position the Dewar lid approximately 3/4 in (19 mm) below the sample port nut.
8. Slide the Psat tube into the Psat slot in the Dewar lid and retighten the Psat tube connector nut.
9. Insert the jacket onto the Psat tube and insert the Psat tube into the slot on the Dewar lid. Ensure that the Psat tube jacket is below the Dewar lid.



Degassing on the Analysis Port

In addition to preparing a sample using a device such as a SmartPrep, the sample may be further evacuated on an analysis port prior to starting an analysis.

This section includes instructions for installing two types of heating mantles.



It is recommended to degas micropore samples in situ after external degassing. To degas in situ, refer to [Preparation button](#), page [3-23](#) to setup the sample information file with degassing parameters.

1. Install the sample tubes and Dewar lid on the analysis port. Refer to [Installing the Sample Tube](#), page [2-30](#).
2. Go to *Unit [n] > Sample Analysis* and select the sample files. Refer to [Performing an Analysis](#), page [2-44](#).
3. Click **Start**. The sample analysis window will prompt you 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 up to touch the bottom of the Dewar lid.
5. Install the heating mantle using instructions on the following pages.

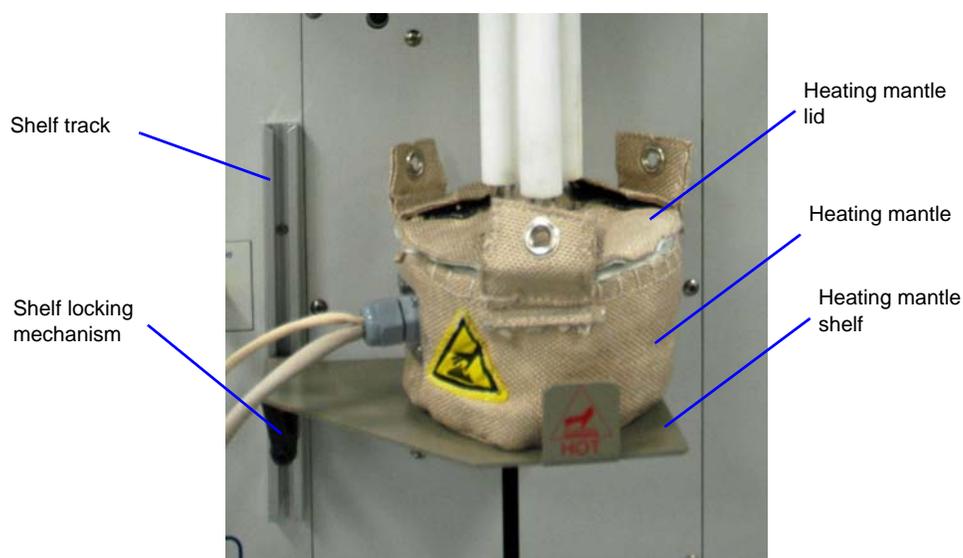
Installing the Heating Mantle using a Shelf Support



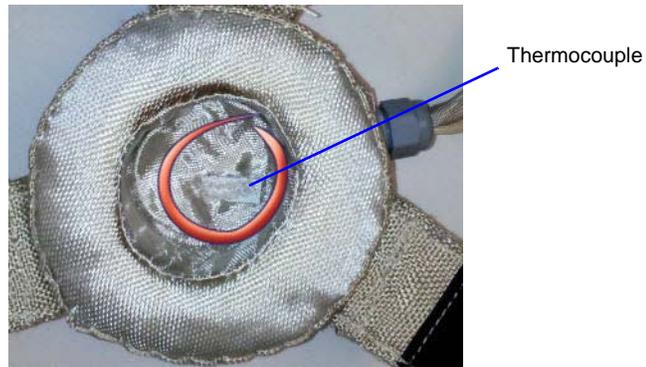
This device has been designed to be used for sample preparation via the instrument control panel. Any other use may damage this device or the analyzer.



Micromeritics offers two styles of heating mantles, either of which can be supported by chains or a shelf. Your heating mantle may differ slightly from the following photos. If installing on an analyzer without shelf support, reference [Installing the Heating Mantle using Chain Supports](#), page 2-38.



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.

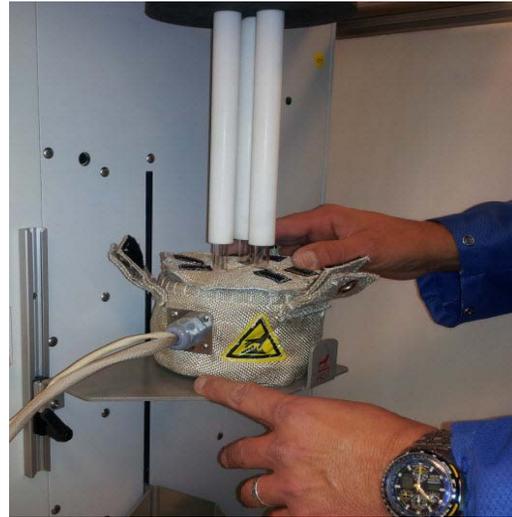
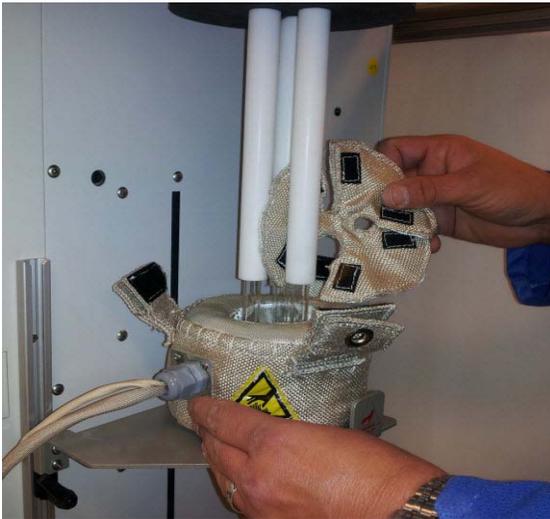


2. While supporting the heating mantle with one hand, slide the shelf locking mechanism into the shelf track. Raise the shelf on the track until the heating mantle rests securely on the shelf and the

sample tubes touch the bottom of the inside of the heating mantle. Turn the locking mechanism clockwise to secure the shelf.



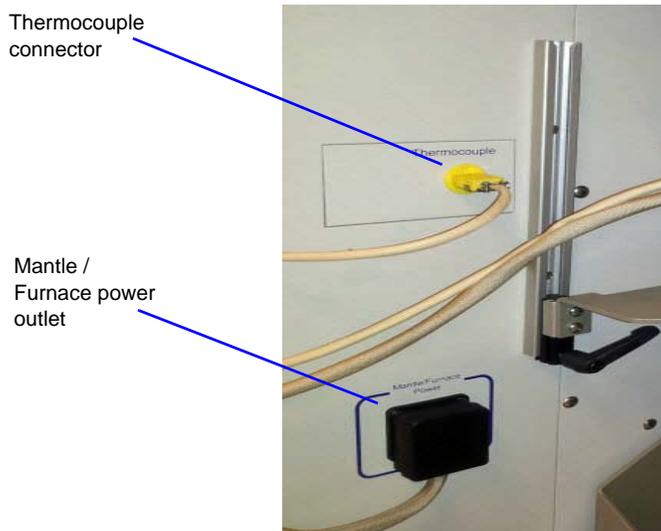
3. Slide the heating mantle cover between the sample tube bulbs and the bottom of the isothermal jackets so that the sample tubes fit within the slots of the mantle cover.



Take care 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 into the instrument's front panel thermocouple connector.



6. Insert the mantle power plug into the instrument's front panel mantle power connector.
7. 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 prompt you to remove the degas heating mantle and shelf, properly position the isothermal jackets and Dewar lid, and install the Dewar.



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

8. To remove the heating mantle, remove 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.

Installing the Heating Mantle using Chain Supports



This device has been designed to be used for sample preparation via the instrument control panel. Any other use may damage this device or the analyzer.



Micromeritics offers two styles of heating mantles, either of which can be supported by chains or a shelf. Your heating mantle may differ slightly from the following photos. If installing with shelf support, reference [Installing the Heating Mantle using a Shelf Support](#), page 2-34.

1. 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.



2. Place the mantle around the sample tube bulbs and attach the hooks on the three support chains through the holes in the Dewar shield skirt around the sample ports. Tighten the loops in the chains so that the mantle is pressing firmly against the bottom of the sample tubes. Ensure that the isothermal jackets are pushed up against the Dewar lid to avoid damage to the jackets.

3. Push the mantle cover down onto the mantle so that the hook and loop fasteners are firmly attached. 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.

Ensure there is a 1/2 in (12 mm) gap between the mantle cover and the bottom of the isothermal jacket.



4. Insert the mantle thermocouple into the instrument's front panel thermocouple connector.

Thermocouple connector

Mantle /
Furnace power
outlet



5. Insert the mantle power plug into the instrument's front panel mantle power connector.
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

prompt you to remove the degas heating mantle and shelf, properly position the isothermal jackets and Dewar lid, and install the Dewar.



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

7. Remove the heating mantle (it is not necessary to unplug the mantle), support the bottom of the tubes and remove the mantle cover.

Filling and Installing the Analysis Dewar

Prepare the analysis Dewar after installing the sample tubes.



Always handle glass Dewars with care. Any product incorporating a vacuum is a potential safety hazard and should be treated with caution. Always observe the precautions listed below.

When handling Dewars containing cryogenic liquids:

- Wear protection by using:
 - goggles (or a face shield)
 - an insulated or rubber apron
 - insulated gloves
- When transferring cryogenic liquids from one container to another:
 - cool the receiving container gradually to minimize thermal shock
 - pour the liquefied 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 cryogenic liquids (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's opening. If an object of sufficient weight is accidentally dropped into the Dewar, shattering may occur.

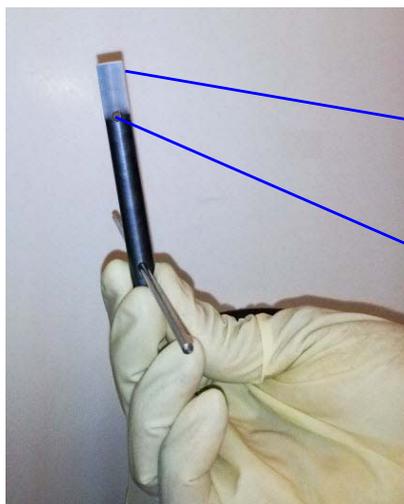
To fill and install the analysis Dewar:

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 into the Dewar and check the level of the analysis bath liquid. Condensation should not exceed the Level Indicator mark.



Wetness or frozen condensation indicates bath liquid level
 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 (or 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 instrument.



Performing an Analysis

Begin analysis after the sample has been degassed and transferred to the analysis port.

Sample Analysis

Allows one analysis using different analysis conditions to run on each port.

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 greater 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 Po gases if measured, and matching backfill gases.
- 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 and 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 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.

Additional analyses can be scheduled by clicking **Next** after the completion of the first series of analyses. The **Next** button appears 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*.

2. To manually close all instrument valves, click the **Close Valves** button.
3. For each port to be used, either click **Browse** and select a sample information file or click **New** to create a new sample information file.



If using port 3 as the vapor source, a sample tube cannot be attached to port 3.

4. Verify the information populated into the sample identification, **Density**, **Mass**, **Sample + Tube**, and **Empty Tube** fields. This information is pulled from the selected or newly created file. The **Density** value is applicable only if using the **Calculate** method for the free space determination.
5. Edit the **p°** and **Bath temperature** fields, if necessary.
6. Click **Report after analysis** to automatically generate reports 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.

- When the analysis is complete, remove the sample tube and store (or dispose of) the sample material as applicable.



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.

Vapor Analysis

A vapor analysis requires that a vapor source container be installed.



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.

Installing the Vapor Source Container using a Shelf Support



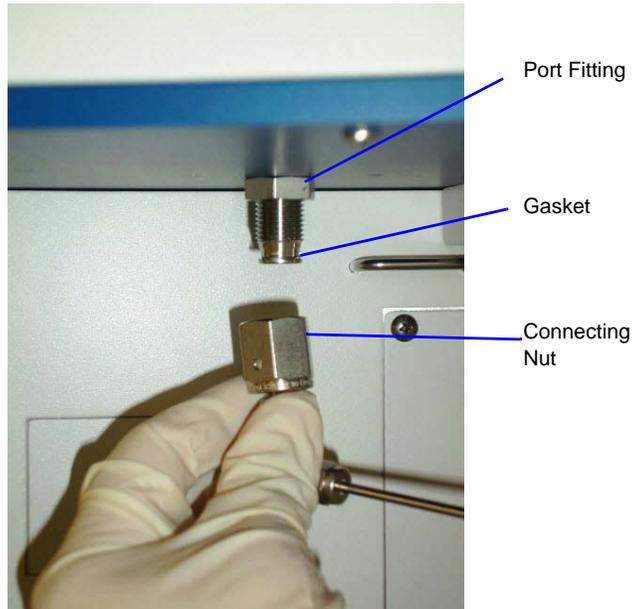
This device has been designed to be used for controlling the vapor source temperature via the instrument control software. Any other use may damage this device or the analyzer.



Each time the Psat tube or Vapor Source container is replaced, a new seal is required. Do not touch the sealing surfaces of the port fitting or seal with bare hands.

- Use an appropriate wrench to loosen the connecting nut from the port fitting by turning the connecting nut counterclockwise while using a second wrench to hold the port fitting stationary. Remove the connecting nut and the attached assembly. If the vapor source container will not be immediately installed, a 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 and insert a new seal.

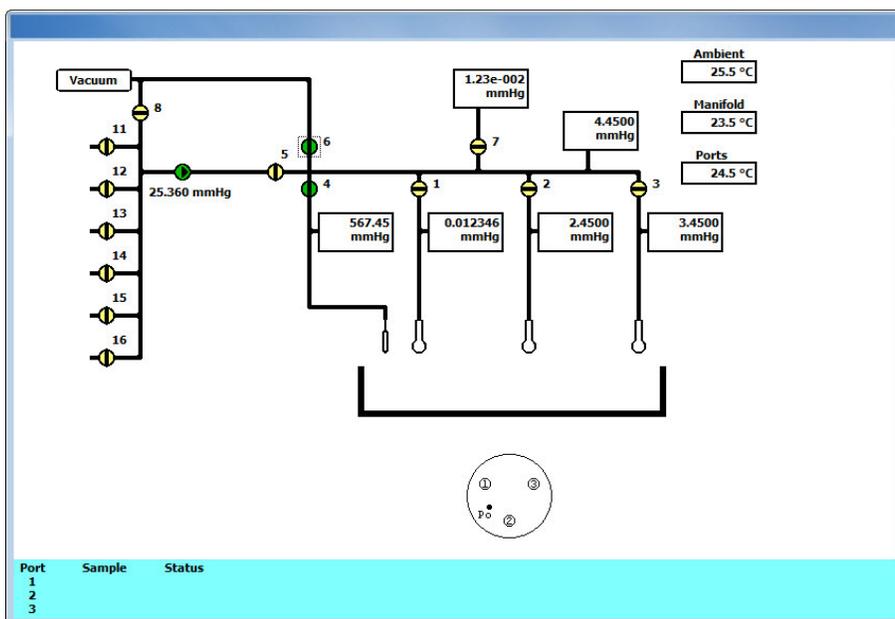


- Install the vapor source container by attaching the connecting nut to the port fitting. Hand tighten the connecting nut by turning clockwise. Use an appropriate size wrench to tighten the assembly an additional 1/8 to 1/4 turn beyond finger tight, while using a second wrench to hold the port fitting stationary on the instrument.

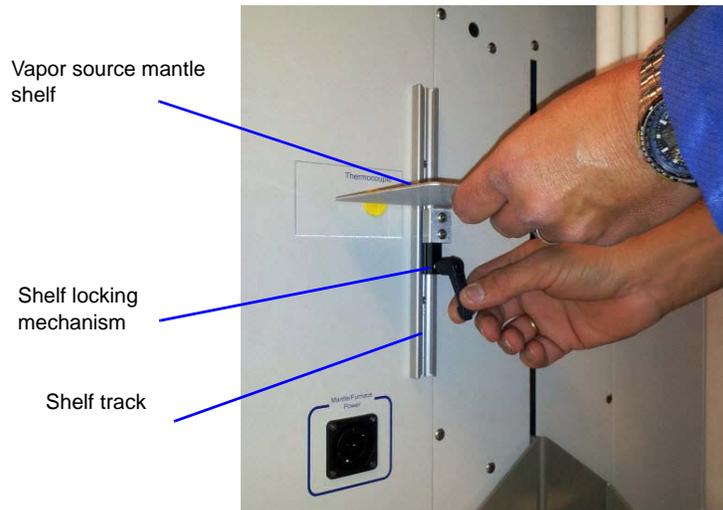


Turn the blue knob to adjust the vapor flow.

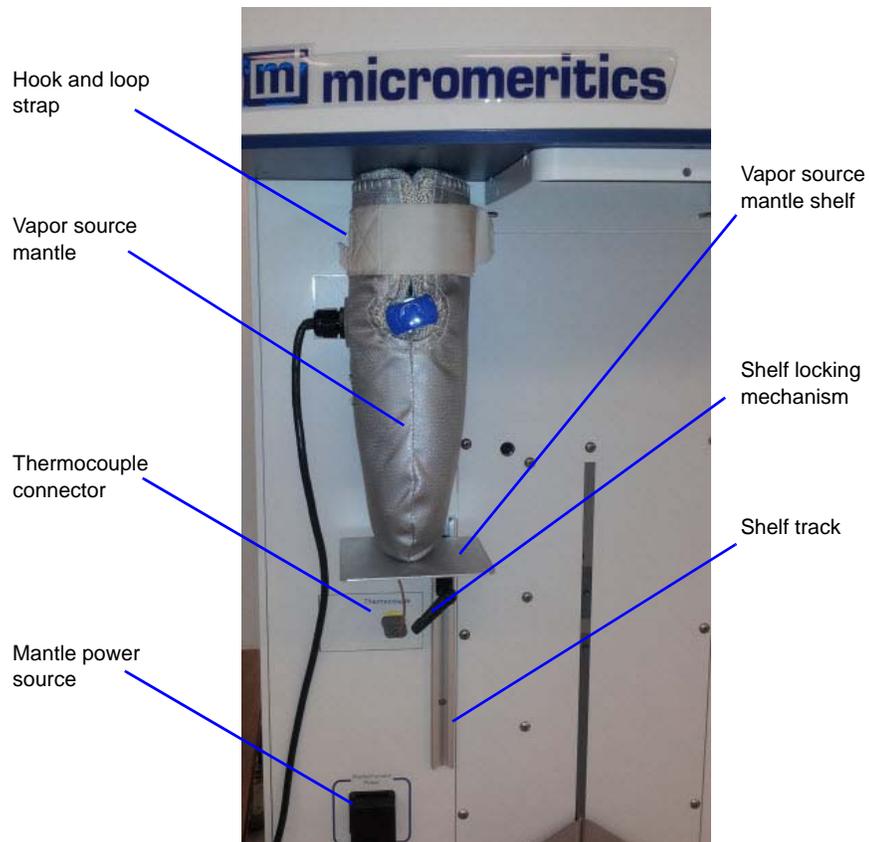
- Using the manual controls on the instrument schematic, evacuate the space above the vapor source by opening valves 4 and 6 with all other valves closed. Then close valve 4 before turning the blue knob on the vapor source to open the connecting valve.



- Slide the shelf locking mechanism of the vapor source mantle shelf into the shelf track on the front of the analyzer. Leave room between the shelf and the underside of the upper cabinet to install the vapor source mantle. To tighten the shelf, turn the locking mechanism clockwise.



- Slide the vapor source mantle over the vapor source container and secure with the hook and loop strap. If necessary, reposition the vapor mantle shelf until the vapor mantle sits securely on the shelf and the top of the mantle is pressed securely against the underside of the upper cabinet. Retighten the locking mechanism. This will prevent a cold spot from forming at the top fitting and condensing vapor.



6. Insert the thermocouple plug into the connector labeled **Thermocouple**. Insert the power plug into the outlet labeled **Mantle/Furnace Power**.

Installing the Vapor Source Container without a Shelf Support

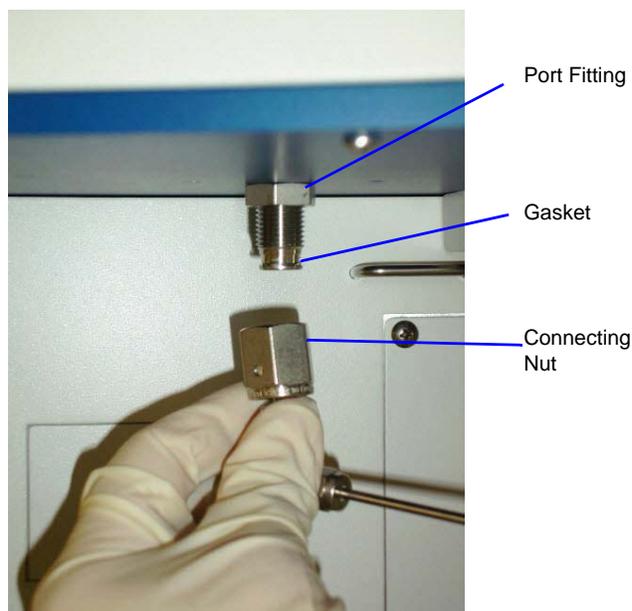


This device has been designed to be used for controlling the vapor source temperature via the instrument control software. Any other use may damage this device or the analyzer.



Each time the Psat tube or Vapor Source container is replaced, a new seal is required. Do not touch the sealing surfaces of the port fitting or seal with bare hands.

1. Use an appropriate wrench to loosen the connecting nut from the port fitting by turning the connecting nut counterclockwise while using a second wrench to hold the port fitting stationary. Remove the connecting nut and the attached assembly. If the vapor source container will not be immediately installed, a 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 and insert a new seal.

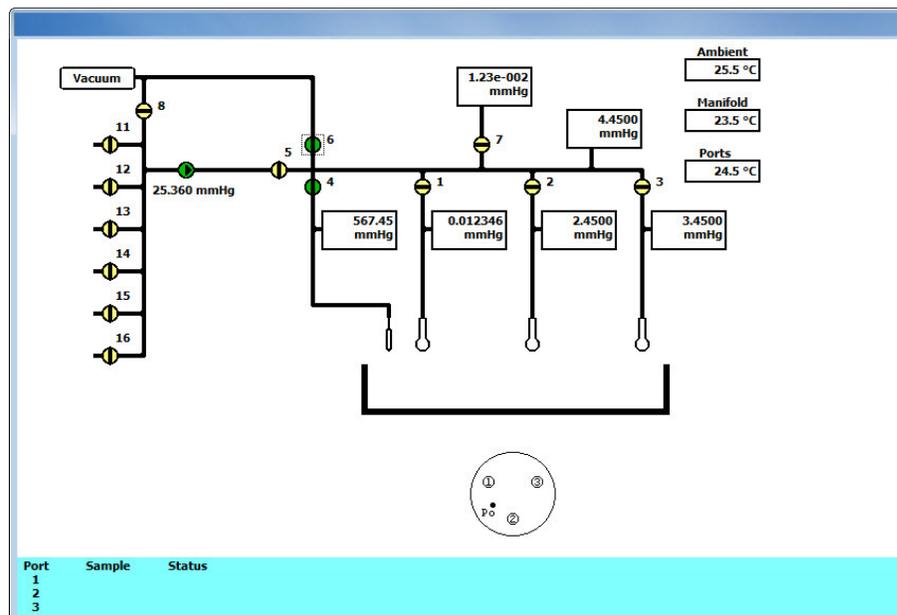


- Install the vapor source container by attaching the connecting nut to the port fitting. Hand tighten the connecting nut by turning clockwise. Use an appropriate size wrench to tighten the assembly an additional 1/8 to 1/4 turn beyond finger tight, while using a second wrench to hold the port fitting stationary on the instrument.



Turn the blue knob to adjust the vapor flow.

- Using the manual controls on the instrument 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 blue knob on the vapor source to open the connecting valve.



- Slide the vapor source mantle over the vapor source container and secure with the hook and loop strap. Insert the thermocouple plug into the connector labeled **Thermocouple**. Insert the power plug into the outlet labeled **Mantle/Furnace Power**.



Running a Vapor Analysis

- Open the sample file to be used for the analysis.
- Go to the **Analysis Conditions** tab. Verify that the selected **Adsorptive** is correct. If not, select the correct **Adsorptive** from the dropdown list.
 - Use the **Edit** button to the right of the **Adsorptive** dropdown to display the **Analysis Adsorptive Properties** window.
 - Select **Vapor source** in the **Dosing Method** group box. If running the vapor analysis on Port 3, select **From Port 3** and skip Step C.

- c.) Set the **Vapor Source Temperature** to the correct value, and select the **Controlled by instrument using heating mantle** checkbox if the vapor source is to be automatically heated to this temperature.

The screenshot shows a software dialog box with the following fields and controls:

- Description: Default
- Mnemonic: (empty)
- Maximum manifold pressure: 925.00 mmHg
- Therm. tran. hard-sphere diameter: 3.860 Å
- Molecular cross-sectional area: 0.162 nm²
- Adsorbate molecular weight: 2.00
- Adsorbed-phase free-space correction
- Fluid properties: Open... Save...
- Dosing Method:
 - Normal
 - From Psat tube
 - From port 3
 - Vapor source
- Charge from inlet
- Purify adsorptive
- Vapor Source Temperature: 43.0 °C
- Controlled by instrument using heating mantle

Buttons: OK, Cancel

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

Blank Analysis

A blank analysis is performed in the same manner as a sample analysis however, the sample tubes will not contain sample material.

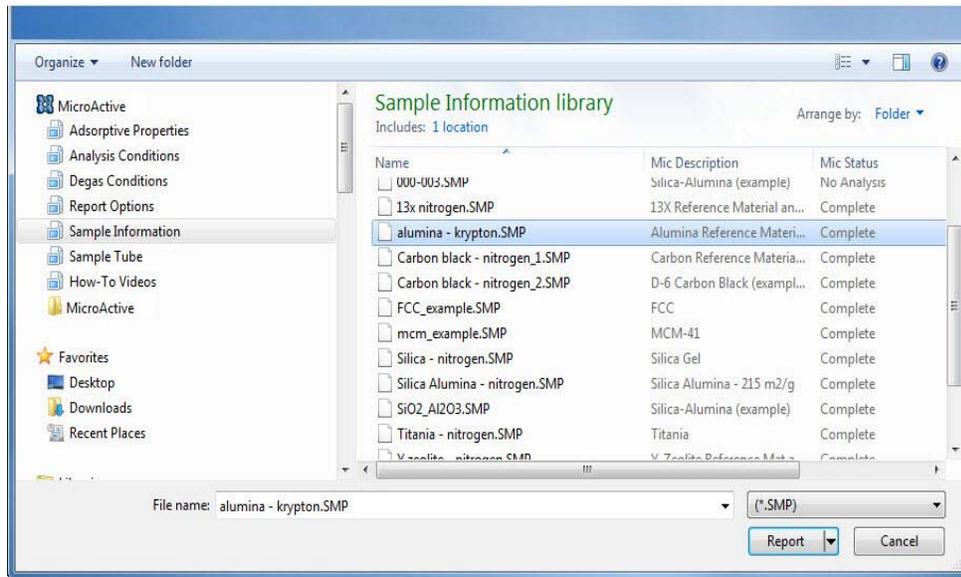
1. Install blank tubes with filler rods and isothermal jackets into each of the sample ports.
2. Be sure to use new O-rings that are new or in good condition on the sample tubes.
3. Make sure the Dewar lid is attached.
4. Verify that the isothermal jacket and filler rods are checked as being used in the sample tube file.
5. Reference [Defining Sample Information Files](#), page [2-6](#) for instructions on defining the sample information file.

Reference Material Analysis

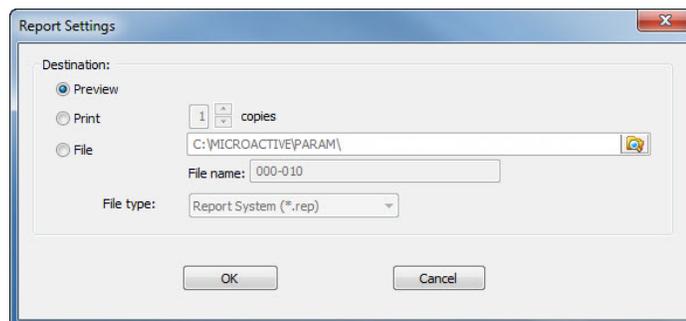
Refer to [Reference Analysis](#), page [4-7](#) for instructions on performing a reference material analysis.

Generating Reports

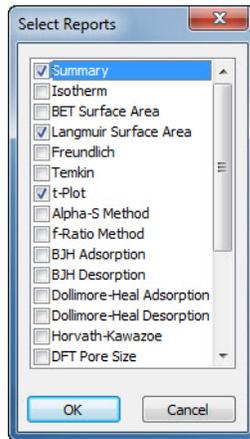
Reports > Start Report (or use the **F8** keyboard shortcut)



1. Select a .SMP file from the library. The selected report name appears in the **File name** text box. 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 and click **OK**.



- If only one file was selected in Step 1, the **Select Reports** window displays. Verify the reports to generate and select additional reports if necessary. Click **OK**. If multiple files were selected, this window is not displayed.



- Click a tab across the top of the window to review each report.

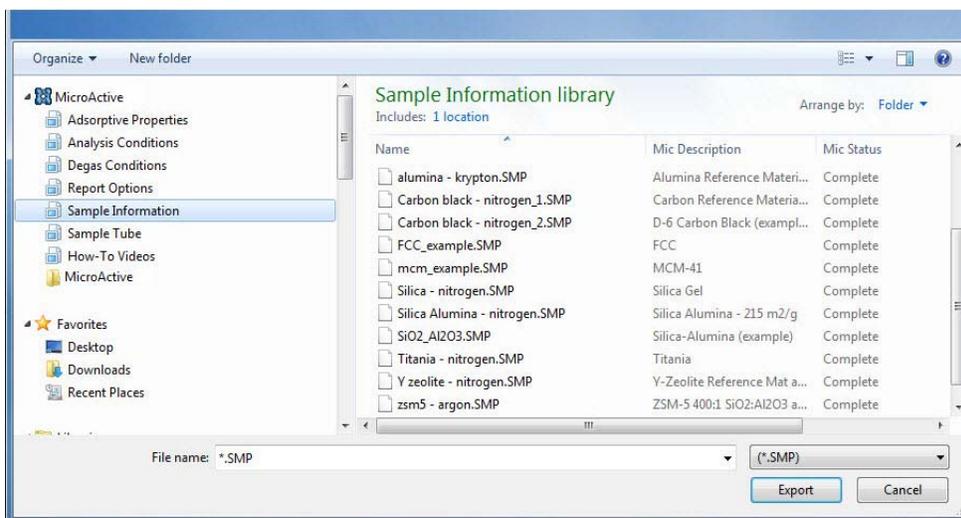
Relative Pressure (p/p*)	Absolute Pressure (mmHg)	Quantity Adsorbed (cm ³ /g STP)	Elapsed Time (h:min)	Saturation Pressure (mmHg)
0.101348134	76.997383	-0.0489	00:19	759.731262
0.203927450	154.930084	-0.2057	00:21	759.731628
0.303898115	230.880936	-0.4750	00:22	759.731384
0.404055123	306.973358	-0.8739	00:23	759.731384
0.504032677	382.929413	-1.4168	00:24	759.731384
0.603721721	458.666412	-2.1202	00:25	759.731323
0.703691546	534.616638	-3.0092	00:26	759.731506
0.803838509	610.701294	0.3167	00:26	759.731506
0.903533153	686.442383	0.0000	00:27	759.731323
0.953689587	724.547852	0.0000	00:29	759.731262
1.000000000	759.731323	0.0000	00:30	759.731323
0.949795532	721.589417	0.0000	00:36	759.731323
0.896114526	680.806274	0.0000	00:37	759.731323
0.796095004	604.818359	0.0000	00:38	759.731323
0.696043202	528.805908	0.0000	00:39	759.731384
			00:40	759.731445

Exporting Files

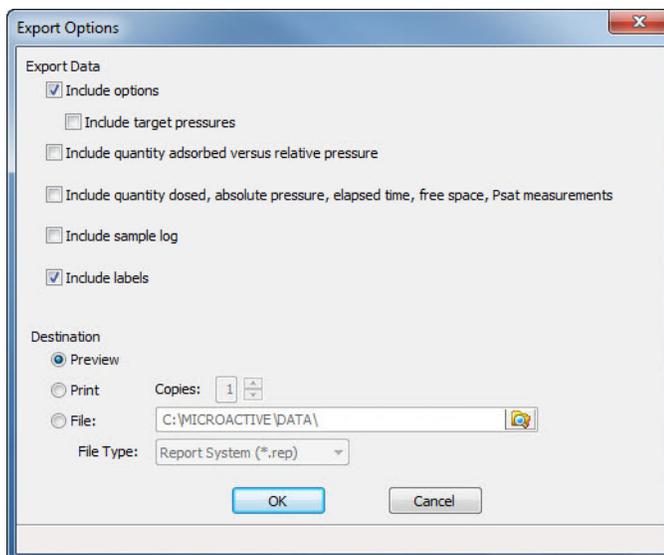
File > Export

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 .REP, .TXT, or .XLS file format. You can select the type of data to include or exclude during the export process. When exported to a file, the data can be imported into other applications that read .TXT or .XLS file formats.

1. Select a file 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.



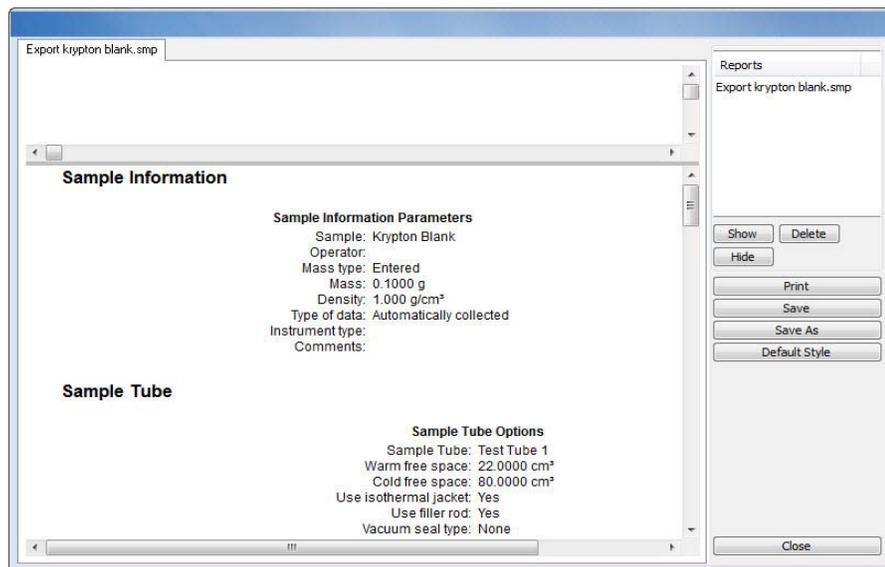
Types of data that can be included:

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

4. Specify the export destination in the **Destination** section of the window:

- **Preview** - to send the file to the screen.
- **Print** - to send the file 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), or an ASCII text (.TXT) file format.

5. Click **OK**. The following example shows a sample information file previewed on the screen.

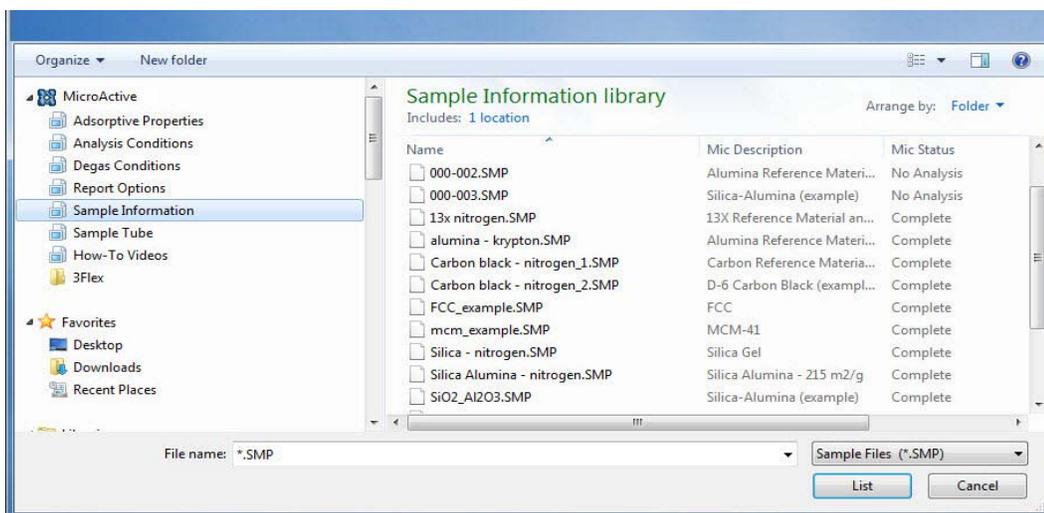


Listing Files

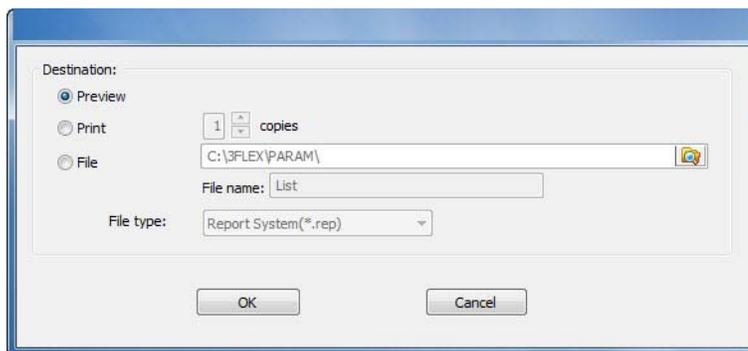
File > List

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.

1. Select a file 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** - to send the file to the screen.
 - **Print** - to send the file 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), or an ASCII text (.TXT) file format.



4. Click **OK**. The following example shows a sample information file printed to the screen.

No.	File Name	Date	Time	File Identification	Status
1	13x nitrogen.SMP	1/6/2012	5:35:16 PM	13X Reference Material analyzed with N2 at 77 K	Complete
2	000-002.SMP	7/17/2012	10:51:23 AM	Alumina Reference Material analyzed with Kr at 77K	No Analysis
3	alumina - krypton.SMP	1/6/2012	5:35:16 PM	Alumina Reference Material analyzed with Kr at 77K	Complete
4	Carbon black - nitrogen_1.SMP	1/6/2012	5:35:16 PM	Carbon Reference Material analyzed with N2 at 77 K	Complete
5	Carbon black - nitrogen_2.SMP	1/6/2012	5:35:18 PM	D-6 Carbon Black (example)	Complete
6	FCC_example.SMP	1/6/2012	5:35:32 PM	FCC	Complete
7	mcm_example.SMP	1/6/2012	5:35:28 PM	MCM-41	Complete
8	Silica Alumina - nitrogen.SMP	1/6/2012	5:35:18 PM	Silica Alumina - 215 m2/g	Complete
9	Silica - nitrogen.SMP	1/6/2012	5:35:18 PM	Silica Gel	Complete
10	000-003.SMP	7/17/2012	11:24:54 AM	Silica-Alumina (example)	No Analysis
11	SiO2_Al2O3.SMP	1/6/2012	5:35:16 PM	Silica-Alumina (example)	Complete
12	Titania - nitrogen.SMP	1/6/2012	5:35:18 PM	Titania	Complete

Generating Graph Overlays

Use the graph overlay function 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 Sample Overlays** - overlay up to 25 plots of the same type with that of the current plot.
- **Multiple Graph Overlays** - overlay two different types of plots from one sample. This type of overlay is available only for:
 - BJH Adsorption/Desorption
 - DFT Pore Size/Surface Energy
 - Dollimore-Heal Adsorption/Desorption
 - Horvath-Kawazoe
 - M-P Method

Only the Advanced format can be used to generate overlays. Go to **Options > Options Presentation > Advanced** to access the Advanced format or select **Advanced** from the dropdown list at the bottom of the window.

Multiple Sample Overlays



When working with an existing file, a copy of the file should be used rather than the original.

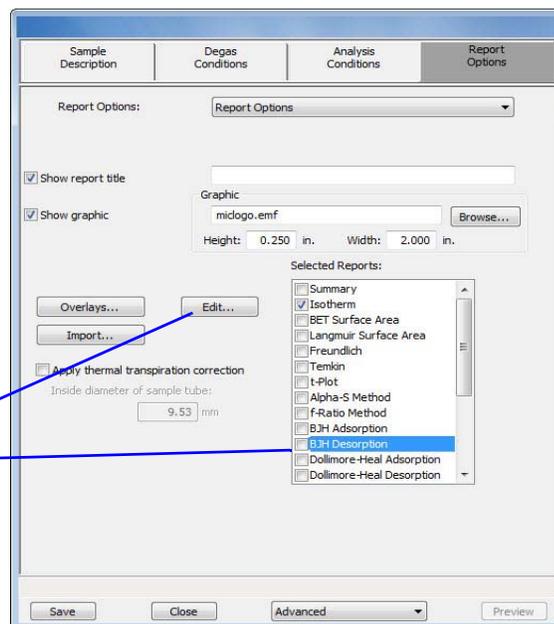
To overlay the same type of graph on multiple samples:

1. Go to **File > Open**.
2. Select the .SMP file and click **Open**.

If a file with a status other than *Preparing*, *Prepared*, or *No Analysis* is selected, the Isotherm plot displays. Select **Advanced** from the dropdown list at the bottom of the window.

3. Click the **Report Options** tab at the top of the window. Refer to the following table for instructions for the selected report.

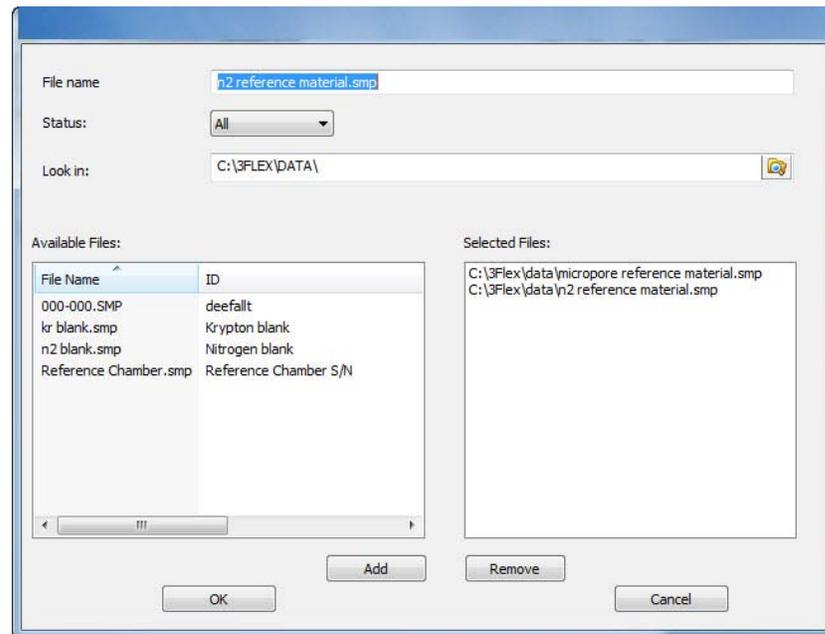
Highlight the report to overlay, then click **Edit**.



If overlaying this type of report..	Then...
<ul style="list-style-type: none"> • Isotherm 	<ol style="list-style-type: none"> a.) In the Selected Reports list box, highlight Isotherm and click Edit. b.) On the Isotherm Report Options window, select a plot in the Selected Reports group box then click the Options button to the right of the selected plot. c.) 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, select the Autoscale checkbox, otherwise, enter the From and To points for the axes. Click OK to save and close the window. d.) On the Report Options window, in the Plot Options group box, select Plot overlays. Click OK. e.) Continue with Step 4.
<ul style="list-style-type: none"> • BET Surface Area • Langmuir Surface Area • Freundlich • Temkin • t-Plot • Alpha-S • f-Ratio 	<ol style="list-style-type: none"> a.) In the Selected Reports list box, highlight one of the report options shown on the left and click Edit. b.) On the Report Options window, select the Overlay samples checkbox for the Transform plot and/or the Isotherm plot. Verify other fields. Click OK to return to the Report Options tab. c.) Continue with Step 4.
<ul style="list-style-type: none"> • BJH Adsorption • BJH Desorption • Dollimore-Heal Adsorption • Dollimore-Heal Desorption • MP-Method 	<ol style="list-style-type: none"> a.) In the Selected Reports list box, highlight a report option shown on the left and click Edit. b.) Select the report variable from the Selected Reports group box and click Edit. c.) Click the dropdown arrow on the Overlay field and select the Samples option. Verify other fields. Click OK to return to the Report Options window. d.) Click OK again to return to the Report Options tab. e.) Continue with Step 4.

4. On the **Report Options** tab, click the **Overlays** button.

5. 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.

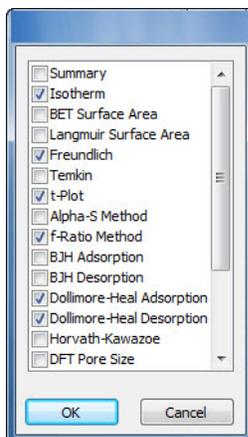


To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.

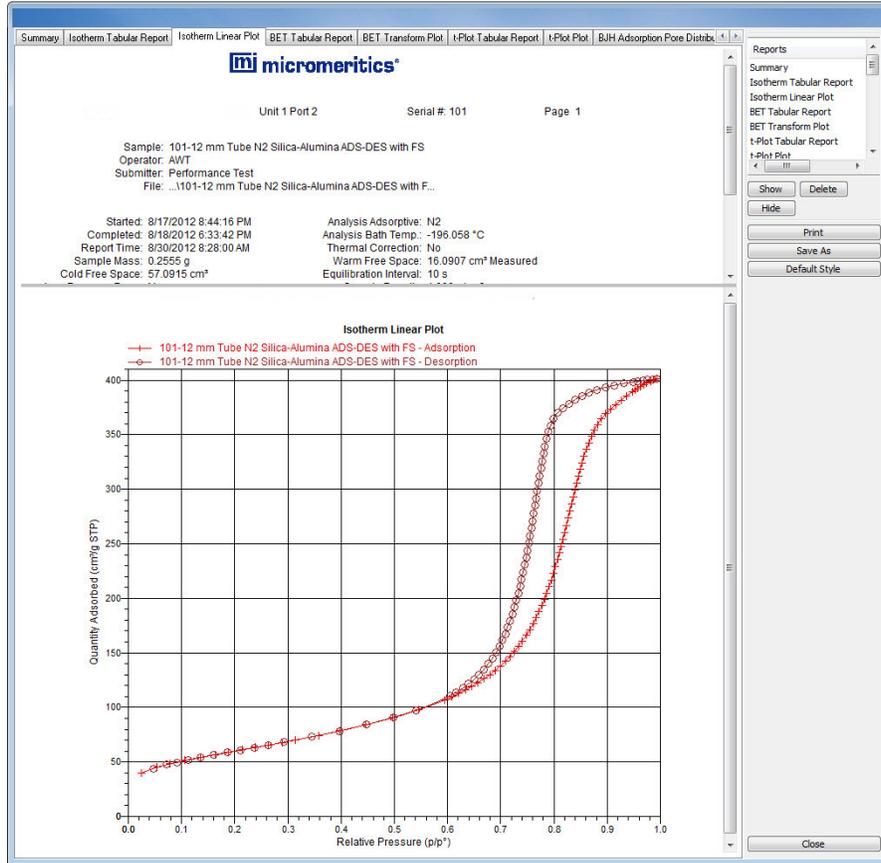
- To move a file from the **Available Files** box to the **Selected Files** box, either:
 - Double click a file name in the **Available Files** box, or
 - Select a file name in the **Available Files** box and click **Add**.
 - To move a file from the **Selected Files** box to the **Available Files** box, either:
 - Double click a file name in the **Selected Files** box, or
 - Select a file name in the **Selected Files** box and click **Remove**.
6. Click **OK**.

7. To view the report, click the **Preview** button on the sample file window.

If the sample file has been closed, go to **Reports > Start Report**. Select the file used in the previous steps and click **Report**. 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. Choose the report destination on the **Report Settings** window and click **OK**. If only one file was selected as an overlay, the **Select Reports** window displays. Verify the reports to generate and add or remove reports as necessary. Click **OK**. If multiple files were selected, the **Selected Reports** window will not display.



- 8. The report screen displays with tabs across the top of the screen. Click each tab to view the reports. Refer to [Report Tool Bar](#), page 5-17.



Multiple 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/Surface Energy
- M-P Method

ASCII text file format rules

- The 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 cm³/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 ads (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 the ASCII text file to generate graph overlays:

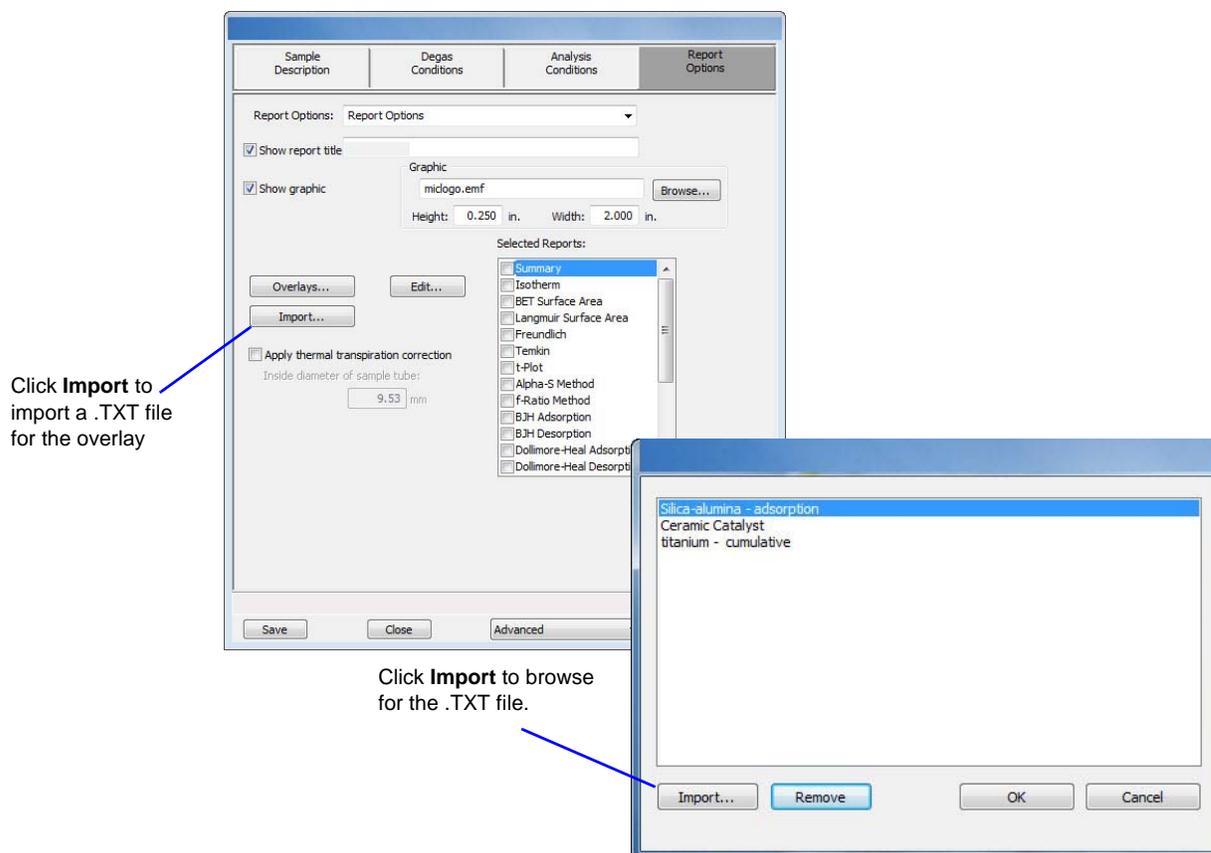
The following steps use BJH Adsorption as an example. Screen appearance will vary depending on the selected report.

1. Go to **File > Open**. Select a sample file to overlay graphs of 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**.

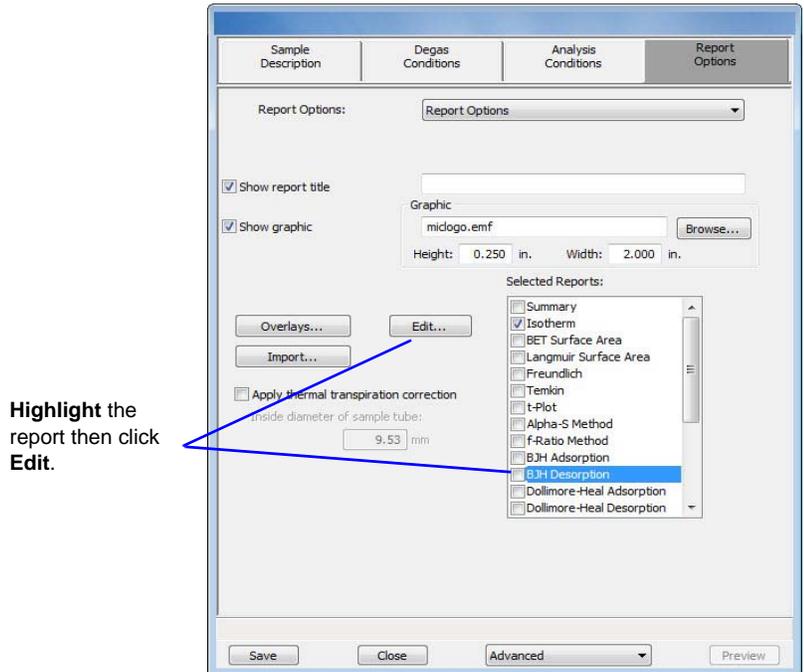
If a file with a status other than *Preparing*, *Prepared*, or *No Analysis* is selected, the isotherm plot displays. Select **Advanced** from the dropdown list at the bottom of the window to return to the **Sample Description** tab.

2. Click the **Report Options** tab, click the **Import** button.

If the ASCII text file does not display on the **Select Imported Overlays** 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. If an error message appears instead, verify that the .TXT file format (listed above) is correct. Select the entry and click **OK**.

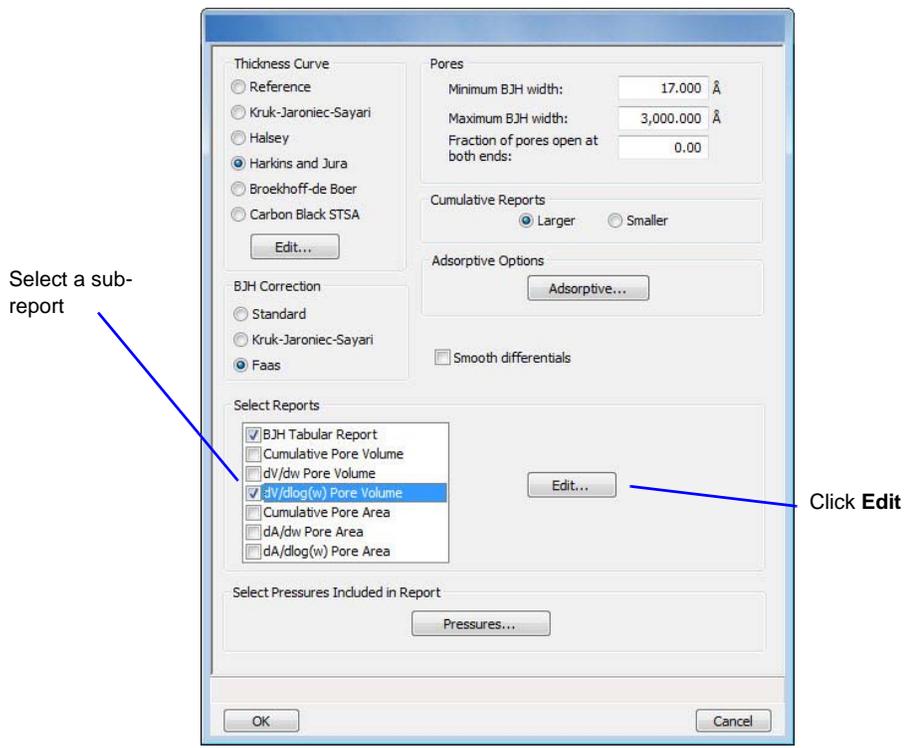


- In the **Selected Reports** list box, highlight the type of report to overlay with a graph and click **Edit**.



Highlight the report then click Edit.

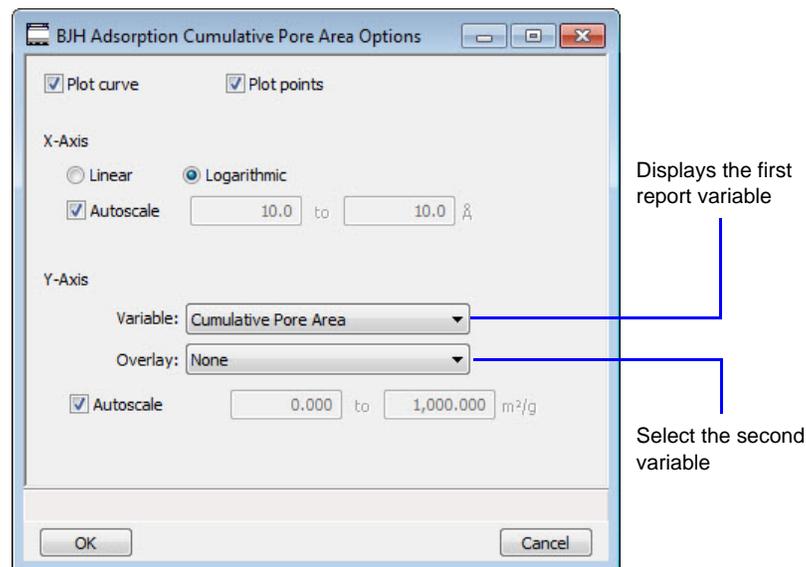
- From the **Report Options** window, in the **Selected Reports** list box, select a sub-report and click **Edit**.



Select a sub-report

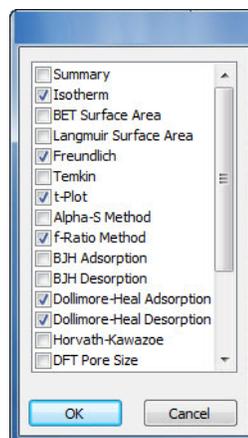
Click Edit

- Click the dropdown arrow at the **Variable** field and select a variable to overlay. Then click the dropdown arrow of the **Overlay** field and select the *Imported Data* entry. Click **OK** to return to the **Report Options** window.

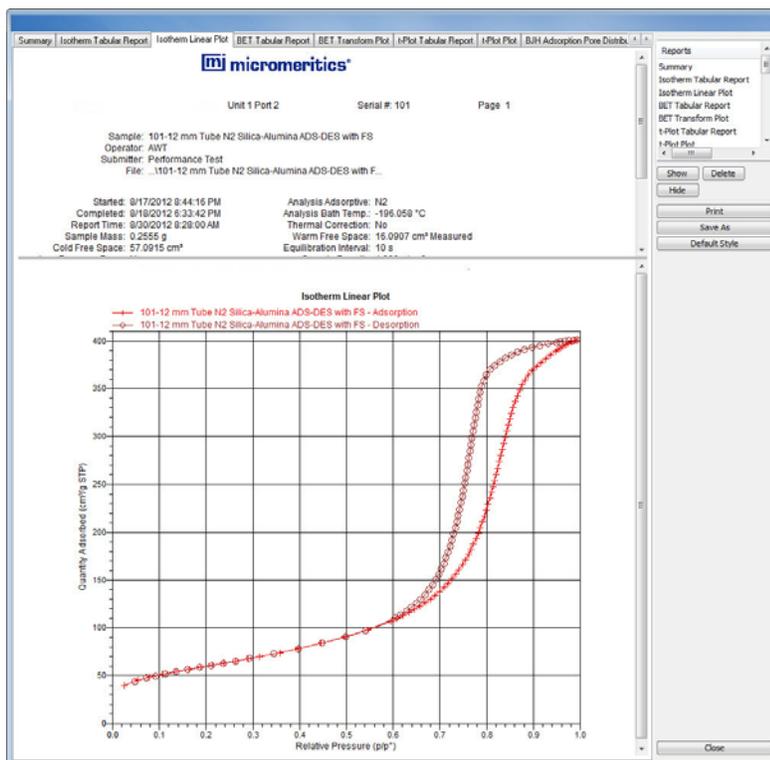


- Click **OK** again to return to the **Report Options** tab.
- Click **Save** to save the selections.
- To view the report, click the **Preview** button on the **Sample Description** window.

If the sample file has been closed, go to **Reports > Start Report**. Select the file used in the previous steps and click **Report**. 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. Choose the report destination on the **Report Settings** window and click **OK**. If only one file was selected as an overlay, the **Select Reports** window displays. Verify the reports to generate and add or remove reports as necessary. Click **OK**. If multiple files were selected, the **Selected Reports** window will not display.



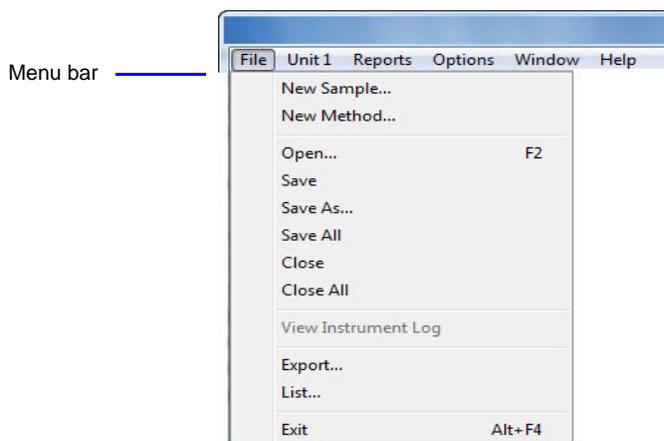
- The report window displays with tabs across the top. Click each tab to view the reports. Refer to [Report Tool Bar](#), page 5-17.



3. FILE MENU

Introduction

This chapter contains information specific to the File menu options used in sample and parameter files. This chapter provides details of File menu options, commonly used functions and buttons, and field descriptions.



Common field and button descriptions are listed in a Common table at the beginning of their respective chapters. Field and button descriptions not listed in the Common table are listed in their appropriate heading.

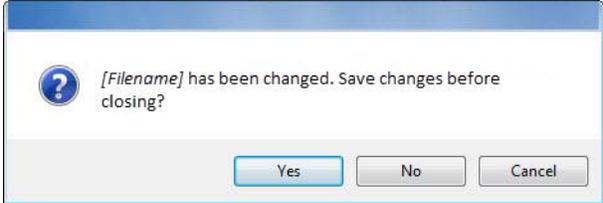


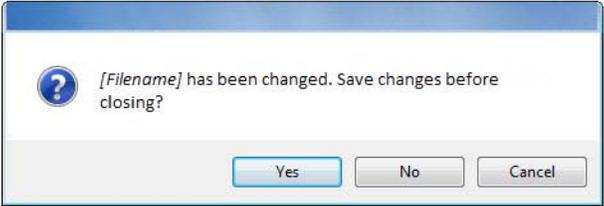
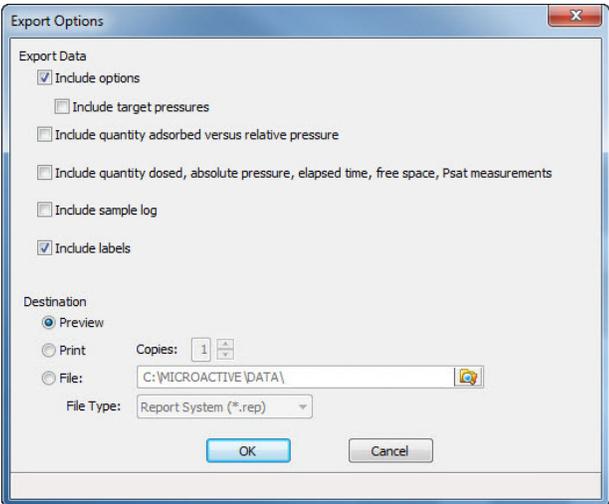
Refer to the Appendix section of this manual for further details on report calculations ([CALCULATIONS](#), page [C-1](#)), free space correction ([FREE SPACE CORRECTION](#), page [D-1](#)), and DFT models ([DFT MODELS](#), page [F-1](#)).

Common Fields and Buttons - File Menu Options

The following fields and buttons are common to many of the File Menu windows. Field and button descriptions not listed below are found in their respective sections.

Field or Button	Description
<i>Autoscale checkbox</i>	When enabled on report parameters screens, 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.
<i>Axis Range</i>	On report parameters screens, 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.

Field or Button	Description (<i>continued</i>)
Browse button or icon	Click to search for a file. Select a file from either the Name column or from the library and click Open or double click the file name to open (or import) the file.
Cancel button	Cancels any changes made to the screen.
Close	Closes the active window. If changes were made to the file and not yet saved, a prompt displays providing the option to save the file.
Close All	<p>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.</p> 
Delete button	When working with report parameters screens, click Delete to remove the selected report. Deleted reports will have to be regenerated if deleted in error.
Destination group box	<ul style="list-style-type: none"> • Preview - sends the file to the screen. Click Print on the report screen to send the file to the printer. • Print - sends the file to the default printer. • Copies - select the number of copies to print. This field is only enabled when Print is selected. • File - saves the report as a file. Click the Browse icon to the right of the text field to select the directory where the new file will be stored. Enter the new file name in the File name text box. • File Type - use to save the new file with a .TXT, .XLS or .REP file extension. This field is only enabled when File is selected. <p>.REP (Report system) - saves the report in a format that can be opened within any MicroActive program.</p> <p>.TXT (ASCII text) - saves the report as a text file.</p> <p>.XLS (Spreadsheet file) - saves the report in a format that can be opened within a spreadsheet program.</p>

Field or Button	Description (<i>continued</i>)
<i>Edit button</i>	When working with report parameters screens, highlight the item in the Selected Reports list box and click Edit to modify report details.
<i>Exit</i>	<p>Exits the program.</p> <p>If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the program without saving the changes.</p>  <ul style="list-style-type: none"> • Yes - saves the changes, then closes the window • No - closes the window without saving the changes • Cancel - cancels the Close command
<i>Export</i>	<p>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.</p> 
<i>File name text box</i>	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. Multiple files can be selected by holding down the Ctrl key on the keyboard while selecting multiple files.

Field or Button	Description (<i>continued</i>)
<i>From / To text boxes</i>	When working with report parameters screens, enter the From and To range for x- and/or y-axes.
<i>List</i>	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.
<i>Name column</i>	A list of files in the selected directory or library.
<i>OK button</i>	Click to save and close the active window.
<i>Open button</i>	Click to open the selected file (or double-click the file name in the Name column to open the file).
<i>Preview button</i>	Click to preview predefined reports. Click the tabs across the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled. Refer to Report Tool Bar , page 5-17 .
<i>Print button</i>	Sends the report to the selected destination (screen, printer or file).
<i>Remove</i>	Removes an item from a list.
<i>Replace button</i>	Click to select another file where the values will replace the current file's values.
<i>Save</i>	Saves the active window under the current file name.
<i>Save All</i>	Saves all active windows under the current file names.
<i>Save As</i>	<ul style="list-style-type: none"> • Saves a file in the active window under a different file name. • Saves a subset (parameter) of the sample file in the active window as a standalone parameter file. For example, to create a standalone parameter file of the analysis conditions portion of the active sample file, go to File > Save As, select the Analysis Conditions folder in the library and enter a file name in the File name field. Click Save.
<i>Table buttons</i>	<ul style="list-style-type: none"> • 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.

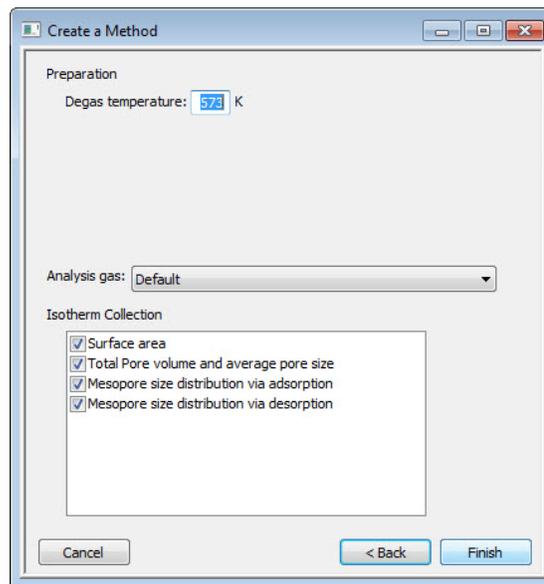
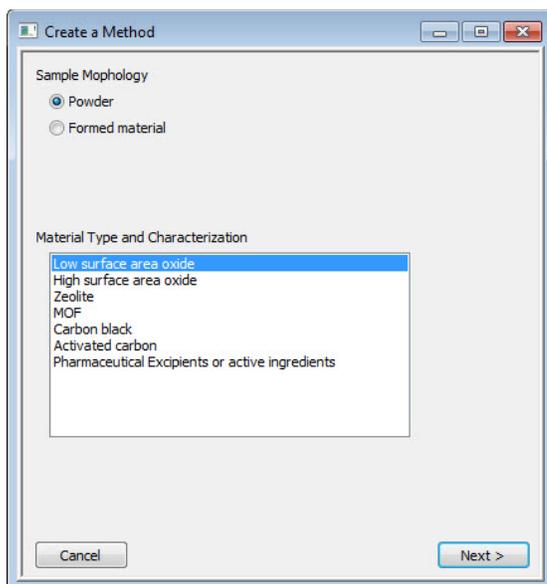
Field or Button	Description (<i>continued</i>)
View Instrument Log	For use by a Service Technician. Operators should use Unit [n] > Show Instrument Log . Refer to Show Instrument Log , page 4-20.

New Method

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.

Methods are created using a wizard that guides you through the process. The *Material Type and Characterization* list and the *Isotherm Collection* list are system defaults and cannot be modified.

Go to **File > New Method** to start the New Method wizard.



New Sample

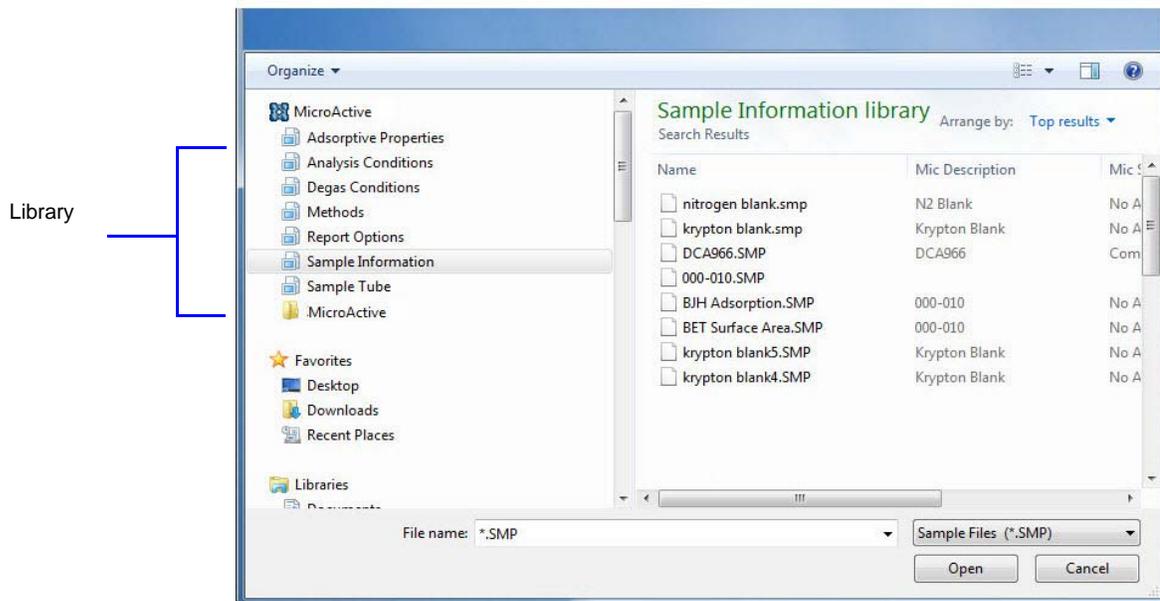
Provides the option to create a new sample file or parameter file. Refer to [Sample Information Files](#), page 3-10.

The screenshot shows the 'New Sample' dialog box with the following details:

- Method:** Default
- Sample:** 000-005
- Operator:** (empty)
- Submitter:** (empty)
- Bar Code:** (empty)
- Sample tube:** Sample Tube (with Edit... button)
- Mass:**
 - Enter
 - Calculate
 - Sample Mass: 1.0000 g
 - Empty tube: 1.0000 g
 - Density: 1.000 g/cm³
 - Sample + tube: 2.0000 g
 - (Sub-field): 1.0000 g
- Type of Data:**
 - Automatically collected
 - Manually entered
- User Parameters:**
 - Parameter 1: 0.000
 - Parameter 2: 0.000
 - Parameter 3: 0.000
- Comments:** (empty text area)
- Buttons:** Add Log Entry, Replace All...
- Footer:** Save, Close, Advanced (dropdown), Preview

Open

Provides access to the library. Refer to [Manage Libraries](#), page 6-2 for information on managing libraries.



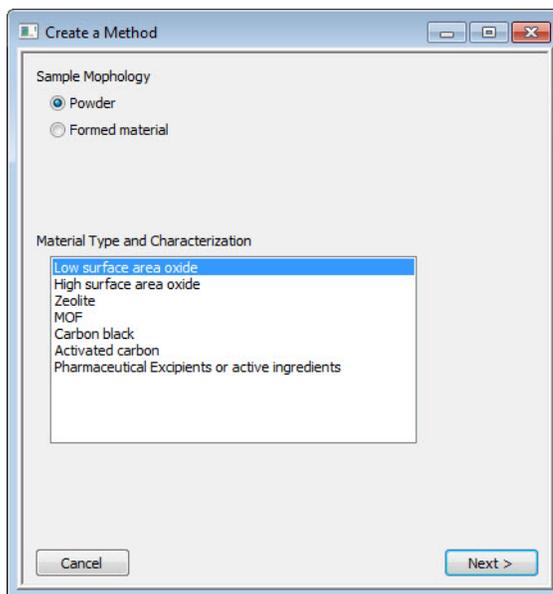
- **Sample Information files** - the **File name** text box contains the next sequential sample information file name generated by the program. The sample information file extension is .SMP.
- **Parameter files** - the **File name** text box contains an asterisk (*) and a default file extension depending on the type of parameter file selected. Default file extensions are:

*.ADP	Adsorptive Properties
*.ANC	Analysis Conditions
*.DEG	Degas Conditions
*.FPI	Fluid Properties
*.RPO	Report Options
*.STB	Sample Tube Properties
*.MTH	Method

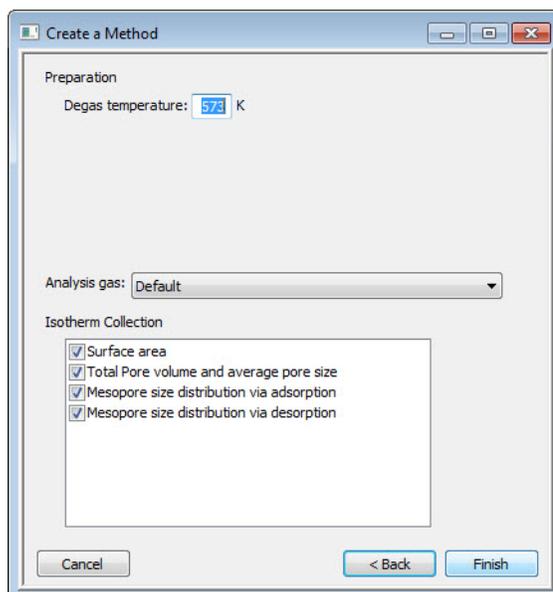
Create a New Method

File > New Method

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



2. Enter a **Degas temperature** then select an **Analysis gas** from the dropdown list.



3. 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.

- On the **Method** tab, if files created using this Method are to be saved in a file directory other than the default, select the **Use separate sample file directory** checkbox and click the **Browse** icon to select a directory. The **Browse** icon is enabled only when the **Use separate sample file directory** checkbox is selected. Select the new directory then click **OK** on the **Browse for Folder** window.

- If the file sequence numbers for this Method will differ from other Methods, select the **Use separate sequence number** checkbox and enter the new sequence number in the text box.
- In the **Sample file name** text box, enter a default form for file names. The \$ symbol is required and represents the position of the sequence number in the file name.
- Refer to [Sample Information Files](#), page **3-10** for details on completing the remainder of this window. The **Type of Data** group box is disabled when creating a Method.
- Click **Save**. The **Save as Sample Information File** window displays. Select **Methods** in the library and enter a file name for the Method in the **File name** text box.
- Click **Save**.

Sample Information Files

File > New Sample

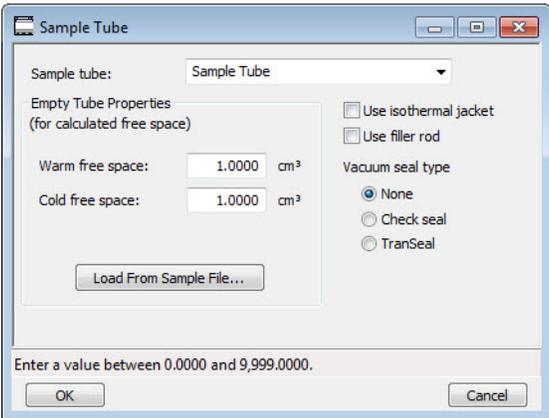
Parameter	Value
Parameter 1	0.000
Parameter 2	0.000
Parameter 3	0.000

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.

Parameter files allow repeated use of the file, for example, if the same analysis conditions exist for multiple analyses, create an Analysis Conditions file containing the recurring conditions. When the sample file is created, select the Analysis Conditions file for the analysis conditions. Once it becomes part of the new sample file, edit the new file as needed without affecting the original Analysis Conditions file. Sample Information files can be created or opened in Advanced, Basic, or Restricted format.



Specify or change the default format by selecting *Options > Option Presentation* or select *Basic / Advanced* from the dropdown list at the bottom of the window. Refer to [Editing the Default Method](#), page 2-3 for a description of the Advanced, Basic, and Restricted formats.

Field or Button	Description
<i>Method dropdown list</i>	Select a method to use for the sample information file. To select an ASTM method, click Browse and navigate to C:\3Flex\Data\Examples . Select an ASTM method from the list.
<i>Sample text box</i>	Enter a description of the sample.
<i>Operator / Submitter text boxes</i>	Enter identification information in the respective text boxes. Some text boxes may have been renamed or may not display if modified in Options > Default Methods .
<i>Bar Code text box</i>	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.
<i>Sample Tube dropdown list</i>	<ul style="list-style-type: none"> • Edit button - click to edit the sample tube parameters. To save the sample tube parameters as a file, go to File > Save As. Select .STB as the File Type and enter a File name.  <ul style="list-style-type: none"> • Sample tube - enter a description of the sample tube, such as the tube size.

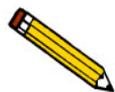
Field or Button	Description (<i>continued</i>)
<p><i>Sample Tube dropdown list (continued)</i></p>	<ul style="list-style-type: none"> • Empty Tube Properties group box - use for calculated free space, enter the values for the following or use the Load from Sample File button to import this information from a sample information file: <ul style="list-style-type: none"> – Warm free space - empty sample tube gas capacity measured at room temperature. – Cold free space - empty sample tube gas capacity measured with the Dewar raised. • Use isothermal jacket checkbox - 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. • Use filler rod checkbox - 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. • Vacuum seal type option - select the seal type to be used.
<p><i>Mass group box</i></p>	<p>If mass = 1, the reported surface area equals the total surface area but it is always shown as m²/g. If the actual mass is entered, the surface area is reported as m²/g. Choose whether to enter mass manually or have the system automatically calculate mass. Enter a value for sample mass and/or density. Both of these values may be edited at the time of analysis.</p> <ul style="list-style-type: none"> • 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: $\text{Mass}_{\text{sample}} = \text{Mass}_{\text{sample+tube}} - \text{Mass}_{\text{tube}}$ • Density - value is used for the Calculated free space method only. Use 0.000 for a blank analysis.

Field or Button	Description (<i>continued</i>)
<i>Type of Data group box</i>	<ul style="list-style-type: none"> • Automatically collected - select if the type of data will be automatically collected by the system while an analysis is running. • Manually entered - use to manually enter data collected from another source. If Manually entered is selected, the Isotherm Report becomes available in the Basic/Advanced dropdown list to allow you to paste or import data into the file. Refer to Manually Entering Isotherm Data in a Sample File, page 2-23.
<i>User Parameters group box</i>	<p>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.</p> <p>The entered parameters display on the Sample Description window, in Reports > SPC Report Options, and the Summary Report.</p> <p>Choose to hide or display these fields or modify the default alphanumeric field labels in Options > Default Method.</p>
<i>Comments text box</i>	Enter comments about the sample or analysis. Comments display in the report header.
<i>Add Log Entry button</i>	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.
<i>Replace All button</i>	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.
<i>Close button</i> <i>Preview button</i> <i>Save button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

Degas Conditions File



Use this option only when the SmartPrep Degasser is installed. To specify in situ degas, on the Analysis Conditions tab click the Preparation button.



The Degas Conditions tab displays only if enabled in *Options > Option Presentation > Show Degas Conditions*.

File > Open > [.DEG file] (or click the **Degas Conditions** tab when in Advanced format)

The **Degas Conditions** tab provides details for setting up the Degas Conditions parameter file. This information will be automatically applied during the degassing procedure if using the SmartPrep Degasser.

Field or Button	Description
<i>Degas Conditions dropdown list</i>	Use to browse for a .DEG file that contains degas condition parameters to be used in the analysis.

Field or Button	Description (<i>continued</i>)
<i>Heating Phase table</i>	Enter up to five stages of degas conditions. <ul style="list-style-type: none">• Temperature - soaking temperature with flowing gas.• Temperature Ramp Rate - rate at which the temperature is to change when advancing to the soak temperature.• Time - amount of time to soak the sample.
<i>Close button</i> <i>Preview button</i> <i>Save button</i> <i>Table buttons</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

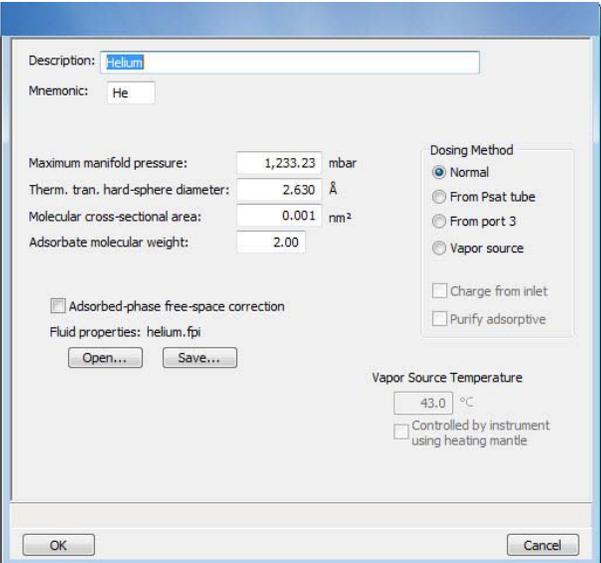
Analysis Conditions Files

File > Open > [.ANC file] (or click the **Analysis Conditions** tab when in Advanced format)

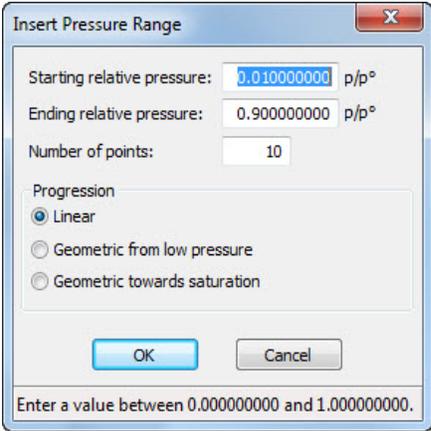
The **Analysis Conditions** tab provides details for setting up the sample analysis conditions file.

	Up to		Add a Point Every		Using	
	Relative Pressure (p/p°)	Relative Pressure (p/p°)	Dose Amount (cm³/g STP)	Equilibration Interval (s)	Relative Pressure (p/p°)	Equilibration Interval (s)
1	0.300000000	0.025000000		10		
2	0.600000000	0.050000000		10		
3	0.950000000		10.0000	10		
4	0.990000000	0.010000000	5.0000	10		
5	0.995000000			10		
6	1.000000000			10		
7	0.995000000			10		
8	0.990000000			10		

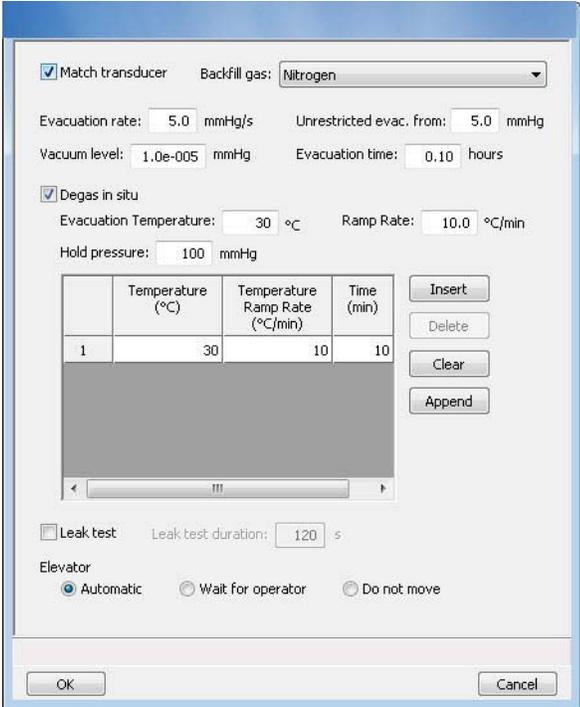
Field or Button	Description
<i>Run Conditions</i> dropdown list	Use to browse for a .ANC file that contains analysis condition parameters to be used in the analysis.

Field or Button	Description (<i>continued</i>)
<i>Adsorptive dropdown list</i>	<p>Displays a list of defined gases. Select the adsorptive to be used for the analysis. Click the Edit button to edit the adsorptive properties.</p> 

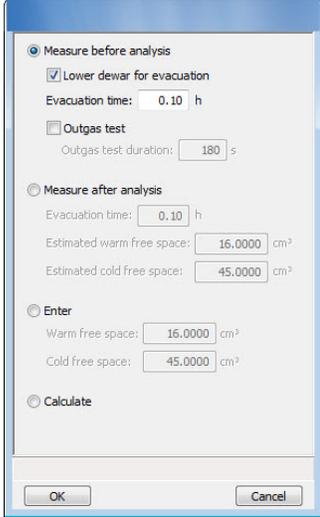
Field or Button	Description <i>(continued)</i>
<i>Adsorptive (continued)</i>	<ul style="list-style-type: none"> • Description text box - defaults to the adsorptive listed on the previous window. • Mnemonic - enter the mnemonic name for the adsorptive. • Maximum manifold pressure - the highest pressure that the manifold will be dosed to. To avoid damage to the instrument, this number is limited to 925 mmHg. Low pressure sources, such as vapors, will require lower numbers. • Therm. tran. hard-sphere diameter - an estimate of molecular size used in calculating the thermal transpiration correction. • 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. • Adsorbate molecular weight - the molecular weight is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot. • Adsorbed-phase free-space correction checkbox - use 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. It should be deselected for blank tube analyses. • Fluid properties - use to import parameters from a Fluid Properties file. Click Open to browse and select an .FPI file. Locate and select the file then click Open on the file selector screen. 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. • Dosing Method group box - select the dosing method to be used. <ul style="list-style-type: none"> – Normal - select for standard and high throughput analyses. Dose from a pressurized tank of gas attached to a gas inlet port. – From Psat tube - select if the Psat tube is filled with condensed adsorptive and dosed from the Psat tube. Select this option if using Krypton. – From Port 3 - select if the tube attached to sample port 3 is filled with condensed adsorptive and dosed from Port 3.

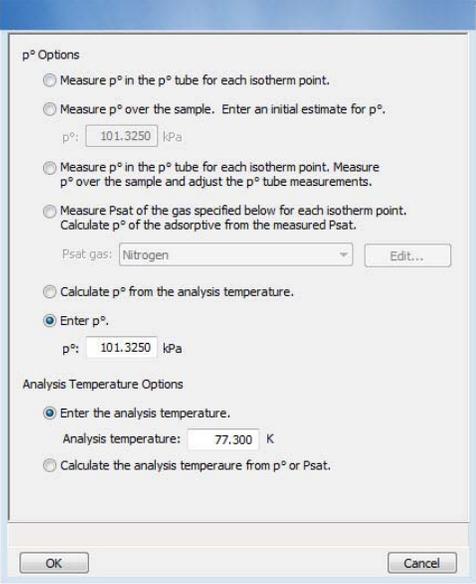
Field or Button	Description <i>(continued)</i>
<i>Adsorptive (continued)</i>	<ul style="list-style-type: none"> - Vapor Source - select if a container of condensed vapor is attached to the Psat port in place of the Psat tube, and is dosed from the Psat port. - Charge from inlet - use to have the tube automatically charged with condensate from a gas inlet port after the Dewar is raised. To import parameters from a Fluid Properties file, click Open to browse and select an .FPI file. Locate and select the file then click Open on the file selector screen. Click Save to save the changes made from the importing the .FPI file. - Purify adsorptive - 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 dosing 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. • Vapor Source Temperature checkbox - select if the vapor source temperature is to be controlled by the instrument. If the vapor source temperature is to be controlled by the operator, do not select this checkbox. This field is enabled only if Vapor source is selected.
<i>Insert Range button</i>	<p>To change the screen options to Absolute pressure, select the Absolute pressure dosing checkbox.</p> <p>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.</p> 

Field or Button	Description <i>(continued)</i>
<i>Insert Range button (continued)</i>	<ul style="list-style-type: none"> • Starting relative pressure field - enter the relative pressure at which data points will start to be taken. • Ending relative pressure field - enter the relative pressure at which data points will no longer be taken. • Number of points field - enter the number of points to be taken between the specified starting and ending relative pressures.
<i>Progression group box</i>	<ul style="list-style-type: none"> • Linear - use to insert evenly spaced points into the table. • Geometric from low pressure - use to insert geometrically spaced points from the low pressure range. For example, to insert 5 points with a 0.01 starting pressure and a 0.16 ending pressure, the following points are inserted into the table: <ul style="list-style-type: none"> 0.01 0.02 0.04 0.08 0.16 • Geometric towards saturation - use to insert geometrically spaced points from the saturation pressure. For example, to insert 5 points with a 0.99 starting pressure and a 0.84 ending pressure, the following points are inserted into the table: <ul style="list-style-type: none"> 0.99 0.98 0.96 0.92 0.84
<i>Absolute pressure dosing checkbox</i>	<p>Use 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, H₂ adsorption on carbon at liquid nitrogen temperature.</p> <p>If this option is selected, the Relative Pressure labels and entries change to Absolute Pressure in the selected pressure units.</p>

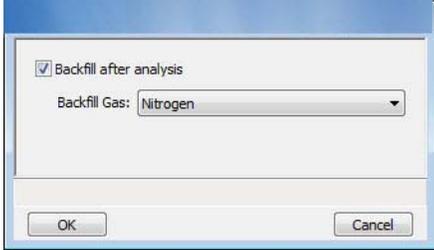
Field or Button	Description (<i>continued</i>)
<i>Preparation button</i>	<p>Use to enter analysis preparation details.</p>  <ul style="list-style-type: none"> • Match transducer checkbox - use to backfill the sample tube to 760 mmHg at the beginning of the analysis and to recalibrate the sample port pressure transducer scale to match the manifold pressure transducer. • Backfill gas dropdown list - select the backfill gas to be used. • Evacuation rate - enter the rate for restricted evacuation. • Unrestricted evac. from - enter the pressure at which unrestricted evacuation is to begin. • Vacuum level - enter the pressure for unrestricted evacuation. • Evacuation time - enter the length of time for preliminary evacuation which takes place prior to the free space measurement or sample analysis if free space is to be entered or calculated. The timer starts when the entered vacuum level is reached.

Field or Button	Description <i>(continued)</i>
Preparation button <i>(continued)</i>	<ul style="list-style-type: none"> • Degas in situ - use to degas the sample on the analysis port prior to analysis. <ul style="list-style-type: none"> – Evacuation Temperature - temperature of the gas during evacuation. – Ramp Rate - rate at which the temperature is to change when advancing to the hold pressure. – 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. • Leak test - select if a leak test is to be performed. • Leak test duration - enter the duration of the leak test. • Elevator - select the appropriate elevator control option. <ul style="list-style-type: none"> – Automatic - the elevator is raised and lowered automatically. – Wait for operator - the operator will be prompted to set the elevator or analysis bath to the desired 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 desired position, or the elevator must 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.

Field or Button	Description (<i>continued</i>)
<i>Free Space button</i>	<p>Use to enter the type of free space measurement and if it is to be measured, entered or calculated.</p>  <ul style="list-style-type: none"> • Measure before analysis - select if the free space is to be measured before the analysis begins. <ul style="list-style-type: none"> – Lower dewar for evacuation - select if the Dewar is to be lowered for evacuation. <p>Evacuation time - if the Dewar is to be lowered for evacuation, enter the length of time for evacuation after the free-space measurement.</p> – Outgas test - select if an outgas test is to be performed before analysis. <ul style="list-style-type: none"> – Outgas test duration - if an outgas test is to be performed, enter the duration of the outgas test. • Measure after analysis - select if free space is to be measured after analysis ends. Enter the evacuation time and the estimated warm and estimated cold free space or accept the defaults. • Enter - use to enter warm and cold free space manually and enter the amount in the text box. • Calculate - use to have the free space measurement calculated using the sample and tube parameters.

Field or Button	Description (<i>continued</i>)
<i>p⁰ and T</i> button	<p>Use to select options for obtaining the saturation pressure (p^0) and analysis bath temperature.</p>  <ul style="list-style-type: none"> • p⁰ Options - select one option indicating how p^0 is to be measured or calculated. <p>Psat Gas dropdown list - if choosing to measure the Psat for each isotherm port, select the Psat gas from the dropdown list and click the Edit button to edit the Psat adsorptive properties. Refer to <i>Adsorptive dropdown list</i> earlier in this table for details on editing this window.</p> <ul style="list-style-type: none"> • Analysis Temperature Options - select an option to manually enter analysis temperature or choose to have it automatically calculated from p^0 or Psat.

Field or Button	Description (<i>continued</i>)
<i>Dosing button</i>	<div data-bbox="727 300 1242 705" style="border: 1px solid gray; padding: 5px; margin-bottom: 10px;"> <p>Absolute pressure tolerance: <input type="text" value="5.000"/> mmHg</p> <p>Relative pressure tolerance: <input type="text" value="5.0"/> %</p> <p>Minimum equilibration delay at %s >= 0.995: <input type="text" value="600"/> s</p> <p>Low pressure equilibration delay:</p> <p>Minimum: <input type="text" value="0.00"/> h</p> <p>Maximum: <input type="text" value="999.00"/> h</p> <p style="font-size: small;">These delays will be used until the first pressure in the table is reached.</p> <p style="font-size: x-small;">Enter a value between 0.000 and 760.000.</p> <p style="text-align: center;"> <input type="button" value="OK"/> <input type="button" value="Cancel"/> </p> </div> <ul style="list-style-type: none"> • Absolute / Relative pressure tolerance - values used to determine how close the actual pressure must be to each target pressure from the pressure table. At lower pressures, the relative tolerance value is less. At higher pressures, the absolute tolerance value is less. For example: <ul style="list-style-type: none"> Experiment 1: You have 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 less 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: You have 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 less 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. <p>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.</p> • Minimum equilibration delay at $p/p^0 \geq 0.995$ - 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.

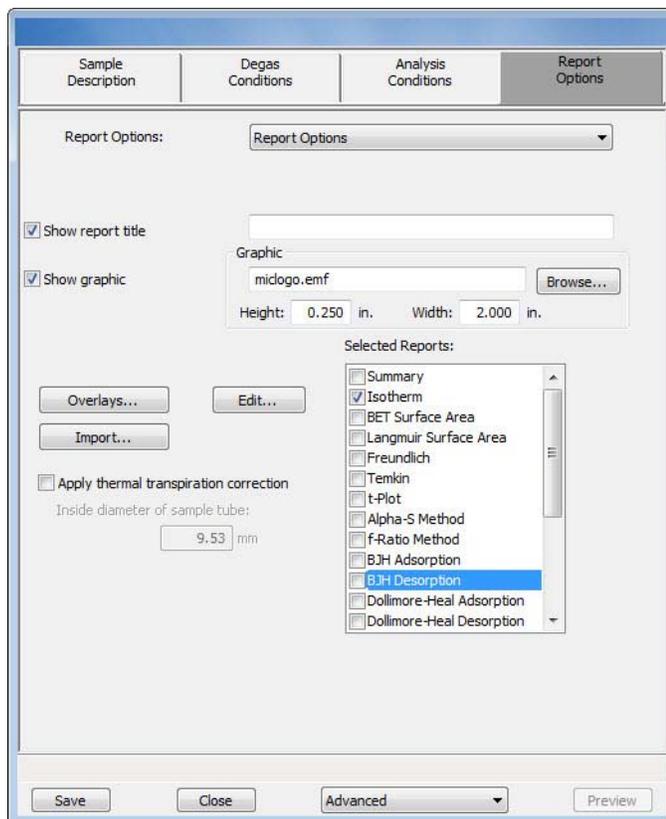
Field or Button	Description <i>(continued)</i>
<i>Termination button</i>	<p>Select if backfill is to be done after the analysis. Click the dropdown list to select the backfill gas to be used.</p> 
<i>Cancel button</i> <i>Close button</i> <i>OK button</i> <i>Preview button</i> <i>Save button</i> <i>Table buttons</i>	<p>Refer to Common Fields and Buttons - File Menu Options, page 3-1.</p>

Report Options Files

File > Open > [.RPO file] (or click the **Report Options** tab when in Advanced format)

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. Refer to **REPORTS MENU**, page 5-1.

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



Field or Button	Description
Report Options <i>dropdown list</i>	Use to browse for a .RPO file that contains report options parameters to be used in the report.
Show report title <i>text box</i>	Enter a report title to appear on the report header.
Show graphic <i>text box</i>	Use to show a graphic on the report header. Click the Browse button to locate the graphic. <ul style="list-style-type: none"> Height / Width - enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.

Field or Button	Description (<i>continued</i>)
<i>Overlays button</i>	Refer to Multiple Sample Overlays , page 2-61.
<i>Import button</i>	Use to import up to 25 pore distribution data files. These datasets are shown only in BJH and Dollimore-Heal reports.
<i>Apply thermal transpiration correction checkbox</i>	<p>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 1.0 mmHg. It is OK to use a sample tube filler rod and thermal transpiration if the filler rod has a bore.</p> <p>Always use thermal transpiration when performing micropore analyses. Refer to CALCULATIONS, page C-1 for additional information on thermal transpiration.</p> <ul style="list-style-type: none"> • Inside diameter of sample tube text box - Enabled when Apply thermal transpiration correction is selected. Enter the inside diameter of the sample tube used in the analysis.
<i>Selected Reports list box</i>	<p>Select the checkbox to the left of the report names to include in the report.</p> <p>For BJH reports, BJH pore dimension can be calculated in pore width (w), pore radius (R) or pore diameter (D). Go to <i>Options > Units</i> to specify default calculations.</p>
<i>Browse button</i> <i>Cancel button</i> <i>Close button</i> <i>Edit button</i> <i>OK button</i> <i>Preview button</i> <i>Save button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

Summary Report

The **Summary Report** provides a condensed listing of selected data results. In the **Selected Reports** list box, highlight **Summary**, then click **Edit**. Select the data types to include in the Summary report.

In the **Pore Volume** group box, if **Adsorption total** or **Desorption total** is selected, the **p/p⁰** field is enabled. Enter the relative pressure used to calculate the total pore volume.

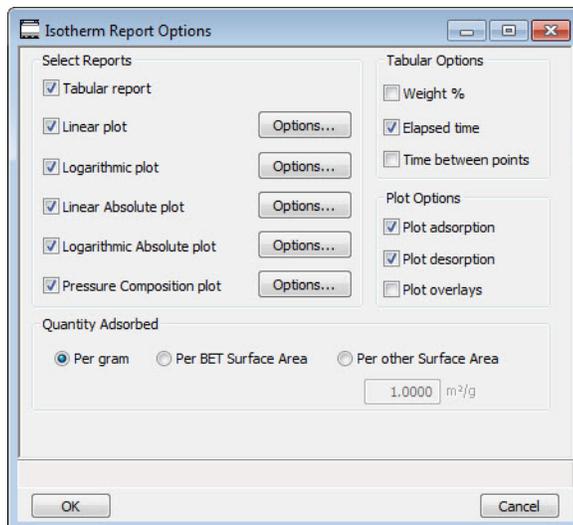
If **Use calculation assignments** is not selected on the **Collected Data** window, the isotherm is interpolated to the value in the **p/p⁰** fields and the point is used for the total pore volume calculation. Otherwise, the point selected with calculation assignment is used. Pass/Fail criteria can be specified for up to four parameters.

Field or Button	Description
<i>Select All / Deselect All buttons</i>	Selects (or deselects) all options.

Field or Button	Description (<i>continued</i>)
<p>Item [n]</p>	<p>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 the checkbox is selected, click the Pass/Fail button and select criteria options for pass/fail options.</p> <ul style="list-style-type: none"> • S A: Single-point BET checkbox - use to enable the Pass/Fail [n] button in the Item [n] group box. • Pass/Fail [n] button - select the S A: Single-point BET checkbox to enable this button. Click the Pass/Fail [n] button to display the Pass/Fail Options window for selection of pass/fail criteria. <div data-bbox="529 674 1344 1314" data-label="Image"> </div> <ul style="list-style-type: none"> • Upper/Lower options and text boxes - 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.
<p>Cancel button OK button</p>	<p>Refer to Common Fields and Buttons - File Menu Options, page 3-1.</p>

Isotherm Report Options

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. In the **Selected Reports** list box, highlight **Isotherm**, then click **Edit**.



Field or Button	Description
<i>Select Reports</i> <i>group box</i>	Select the checkbox to the left of each option to include on the final report.
<i>Options</i> buttons	<p>Click to display related linear plot options. All plot windows contain identical fields.</p> <div data-bbox="781 1245 1286 1537" data-label="Image"> </div> <ul style="list-style-type: none"> <li data-bbox="602 1598 1321 1629">• Plot curve / Plot points - use to plot curves and/or points. <li data-bbox="602 1667 1430 1772">• 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. <li data-bbox="602 1810 1468 1841">• Autoscale y-axis - the y-axis field shows the quantity of gas adsorbed.

Field or Button	Description (<i>continued</i>)
<i>Tabular Options</i> <i>group box</i>	Select the options to include on the report. <ul style="list-style-type: none">• Weight %• Elapsed time• Time between points
<i>Plot Options</i> <i>group box</i>	Select the types of isotherm to plot: <ul style="list-style-type: none">• adsorption• desorption• overlays
<i>Quantity Adsorbed</i> <i>group box</i>	Select how to report the quantity adsorbed. <ul style="list-style-type: none">• Per gram (cm³/g) STP• Per BET Surface Area (cm³/m²) STP or mmol/g• Per other Surface Area (cm³/m²) STP or mmol/m²
<i>Cancel button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1 .

BET/Langmuir Surface Area Report Options

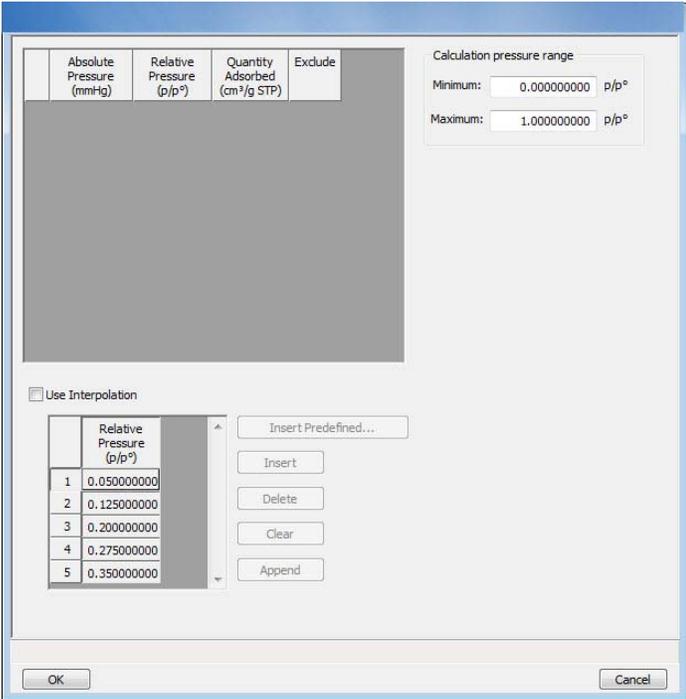
The Langmuir and BET Surface Area windows are identical unless otherwise specified. In the **Selected Reports** list box, highlight **BET (or Langmuir) Surface Area**, then click **Edit**.

- 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.
- 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.

Displays as Langmuir Surface Area Report Options if the Langmuir report is being edited.

Field or Button	Description
<i>Select Pressure Range for BET (or Langmuir) fit text boxes</i>	Enter values to indicate the fitted pressure range.
<i>Tabular report checkbox</i>	Use to have a table of measured and calculated values generated.

Field or Button	Description (<i>continued</i>)
<i>BET (or Langmuir) Transform plot</i>	<p>Use to generate a traditional BET (Langmuir) surface area plot used to determine monolayer volume and BET C constant.</p> <ul style="list-style-type: none">• Overlay samples checkbox - use to overlay sample files on the BET (or Langmuir) transform plot.• Autoscale x-axis - linear x-axes begin at zero. The x-axis field shows the relative pressure for BET and show absolute pressure for Langmuir.• Autoscale y-axis - the y-axis field shows BET (Langmuir) transformation.
<i>BET (or Langmuir) Isotherm plot</i>	<p>Uses the BET (Langmuir) monolayer volume and constant to produce an isotherm.</p> <ul style="list-style-type: none">• Overlay samples checkbox - use to overlay sample files on the BET (or Langmuir) isotherm plot.• Autoscale x-axis - linear x-axes begin at zero. The x-axis field shows the relative pressure for BET and show absolute pressure for Langmuir.• Autoscale y-axis - the y-axis field shows the quantity of gas adsorbed.

Field or Button	Description (<i>continued</i>)
Pressures button	<p>This option is not available if the active file has a status of <i>No Analysis</i>.</p> <p>Use to enter a range of pressure points or to modify table values for pressure points.</p>  <p>To exclude a point from the calculations used to generate the report, select the Exclude checkbox.</p> <ul style="list-style-type: none"> • Calculation pressure range group box - enter the minimum and maximum pressures to be used in the pressure table if not using the Use Interpolation option. • Use Interpolation checkbox - use to indicate if the system should use the table or entered data. • Insert Predefined button - click to insert a predefined (default) set of points into the report. Use Interpolation checkbox must be selected to enable this button. This button does not display for the Langmuir report. <p>Refer to Table buttons, page 3-4 for a description of the Insert, Delete, Clear and Append buttons.</p>

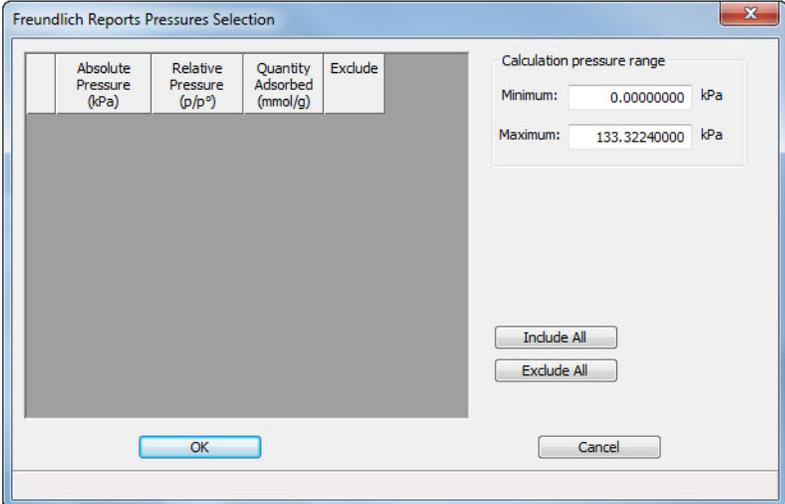
Field or Button	Description (<i>continued</i>)
<p><i>Cancel button</i></p> <p><i>Close button</i></p> <p><i>From / To text boxes</i></p> <p><i>OK button</i></p>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

Freundlich Report Options

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**.

Field or Button	Description
<i>Specify monolayer capacity checkbox and text box</i>	Select and enter the monolayer capacity of the sample.
<i>Tabular report checkbox</i>	Use to have a report of the pressure points generated.

Field or Button	Description (<i>continued</i>)
<i>Transform plot checkbox</i>	<p>Plots the log(P) vs log(Q) and the best fit.</p> <ul style="list-style-type: none"> • Overlay samples checkbox - 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.
<i>Freundlich Isotherm plot checkbox</i>	<p>Plots the absolute pressure vs quantity adsorbed. Shows best fit line.</p> <ul style="list-style-type: none"> • Overlay samples checkbox - 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.

Field or Button	Description (<i>continued</i>)
<p><i>Pressures button</i></p>	<p>This option is not available if the active file has a status of <i>No Analysis</i>.</p> <p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <p>To exclude a point from the calculations used to generate the report, select the Exclude checkbox. To include or exclude all points, click the Include All or Exclude All button.</p> <ul style="list-style-type: none"> • Calculation pressure range group box - enter the minimum and maximum pressures to be used in the pressure table. <p>Refer to Table buttons, page 3-4 for a description of the Insert, Delete, Clear and Append buttons.</p>
<p><i>Cancel button</i></p> <p><i>OK button</i></p>	<p>Refer to Common Fields and Buttons - File Menu Options, page 3-1.</p>

Temkin Isotherm Report Options

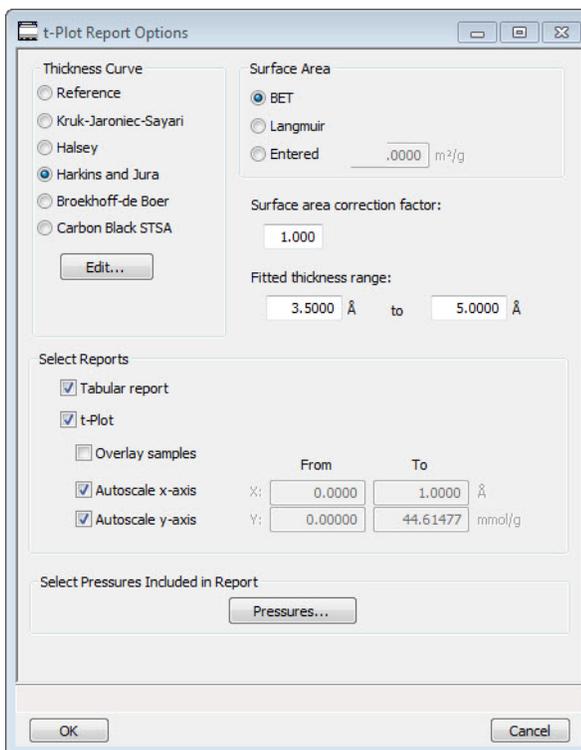
The Temkin isotherm is used to model adsorption data where the heat of adsorption drops linearly with increasing coverage. In the **Selected Reports** list box, highlight **Temkin**, then click **Edit**.

Field or Button	Description
<i>Specify monolayer capacity checkbox and text box</i>	Select and enter the monolayer capacity of the sample.
<i>Specify differential heat of adsorption checkbox and text box</i>	Select and enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all Temkin constants.
<i>Tabular report checkbox</i>	Use to have a report of the pressure points generated.
<i>Transform plot checkbox</i>	Plots a linear form of the Temkin transform plot. <ul style="list-style-type: none"> • Overlay samples checkbox - use to overlay sample files on the 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.

Field or Button	Description (<i>continued</i>)
<i>Temkin Isotherm plot checkbox</i>	Overlays the Temkin isotherm with the analysis data. <ul style="list-style-type: none">• Overlay samples checkbox - 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.
<i>Pressures button</i>	Refer to Pressures button , page 3-38 .
<i>Cancel button</i> <i>From / To text boxes</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1 .

t-Plot Report Options

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. In the **Selected Reports** list box, highlight **t-Plot**, then click **Edit**.



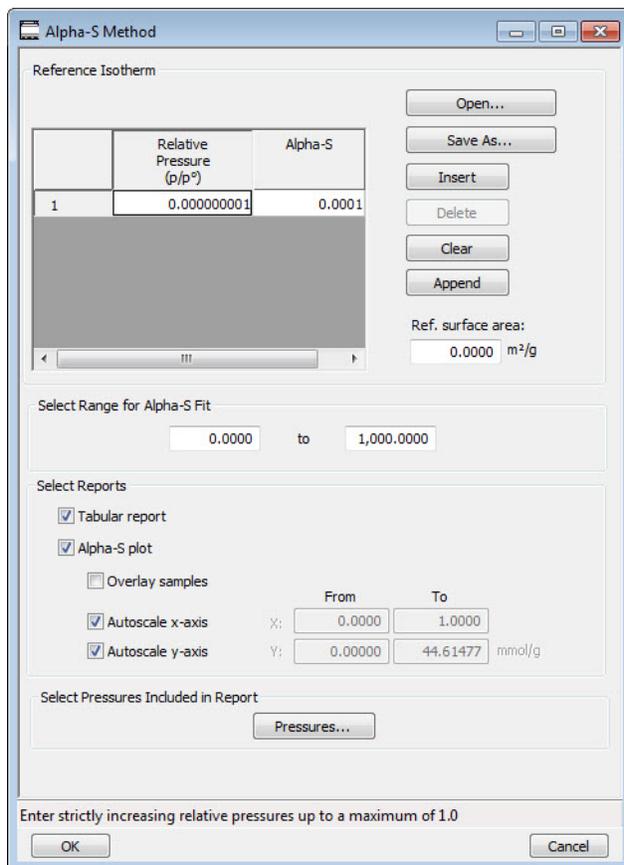
Field or Button	Description
<i>Thickness Curve group box</i>	<p>Select the thickness curve and click Edit to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option.</p> <p>Reference option - select Reference and click Edit to define a t-curve by entering the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the Reference directory.</p>

Field or Button	Description (continued)																																										
<p>Thickness Curve group box (continued)</p>	<div data-bbox="782 331 1256 928" data-label="Image"> <table border="1"> <thead> <tr> <th></th> <th>Relative Pressure (p/p²)</th> <th>Thickness (Å)</th> </tr> </thead> <tbody> <tr><td>1</td><td>0.00000583</td><td>0.0042</td></tr> <tr><td>2</td><td>0.00001269</td><td>0.0082</td></tr> <tr><td>3</td><td>0.00002467</td><td>0.0123</td></tr> <tr><td>4</td><td>0.00004337</td><td>0.0164</td></tr> <tr><td>5</td><td>0.00006996</td><td>0.0205</td></tr> <tr><td>6</td><td>0.00010668</td><td>0.0246</td></tr> <tr><td>7</td><td>0.00015595</td><td>0.0287</td></tr> <tr><td>8</td><td>0.00022070</td><td>0.0328</td></tr> <tr><td>9</td><td>0.00030356</td><td>0.0369</td></tr> <tr><td>10</td><td>0.00040910</td><td>0.0410</td></tr> <tr><td>11</td><td>0.00054173</td><td>0.0450</td></tr> <tr><td>12</td><td>0.00070642</td><td>0.0491</td></tr> <tr><td>13</td><td>0.00091180</td><td>0.0531</td></tr> </tbody> </table> </div> <p>To import values from an existing thickness curve (.THK file), click Open and 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.</p> <p>Refer to Table buttons, page 3-4 for a description of the Insert, Delete, Clear, and Append buttons.</p> <p>Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA - select the thickness curve option and click Edit. Modify the equation for the selected curve as needed.</p> <div data-bbox="764 1419 1224 1724" data-label="Image"> $t = \left(\frac{60.6500}{0.03071 \cdot -\log(p/p^2)} \right) \cdot 0.3968$ <p>Enter a value between 0.00001 and 9,999.00000.</p> </div>		Relative Pressure (p/p ²)	Thickness (Å)	1	0.00000583	0.0042	2	0.00001269	0.0082	3	0.00002467	0.0123	4	0.00004337	0.0164	5	0.00006996	0.0205	6	0.00010668	0.0246	7	0.00015595	0.0287	8	0.00022070	0.0328	9	0.00030356	0.0369	10	0.00040910	0.0410	11	0.00054173	0.0450	12	0.00070642	0.0491	13	0.00091180	0.0531
	Relative Pressure (p/p ²)	Thickness (Å)																																									
1	0.00000583	0.0042																																									
2	0.00001269	0.0082																																									
3	0.00002467	0.0123																																									
4	0.00004337	0.0164																																									
5	0.00006996	0.0205																																									
6	0.00010668	0.0246																																									
7	0.00015595	0.0287																																									
8	0.00022070	0.0328																																									
9	0.00030356	0.0369																																									
10	0.00040910	0.0410																																									
11	0.00054173	0.0450																																									
12	0.00070642	0.0491																																									
13	0.00091180	0.0531																																									
<p>Surface Area group box</p>	<p>Select the surface area value used for thickness calculations. BET is the most commonly used option.</p>																																										

Field or Button	Description <i>(continued)</i>
<i>Surface area correction factor text box</i>	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.
<i>Fitted thickness range text boxes</i>	Enter the minimum and maximum thicknesses (in angstroms or nanometers) to include in the thickness curve. Go to <i>Options > Units</i> to specify default units. Refer to UNIT MENU , page 4-1 .
<i>Tabular report checkbox</i>	Use to have a tabular report of data generated.
<i>t-Plot checkbox</i>	Use to have a graphical representation of data generated. <ul style="list-style-type: none"> • Overlay samples checkbox - 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.
<i>Pressures button</i>	Refer to Pressures button , page 3-38 .
<i>Cancel button</i> <i>From / To text boxes</i> <i>OK button</i> <i>Table buttons</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1 .

Alpha-S Method

The Alpha-S plot converts the standard adsorption isotherm into a dimensionless isotherm using the quantity adsorbed at a relative pressure of 0.4. In the **Selected Reports** list box, highlight **Alpha-S Method**, then click **Edit**.



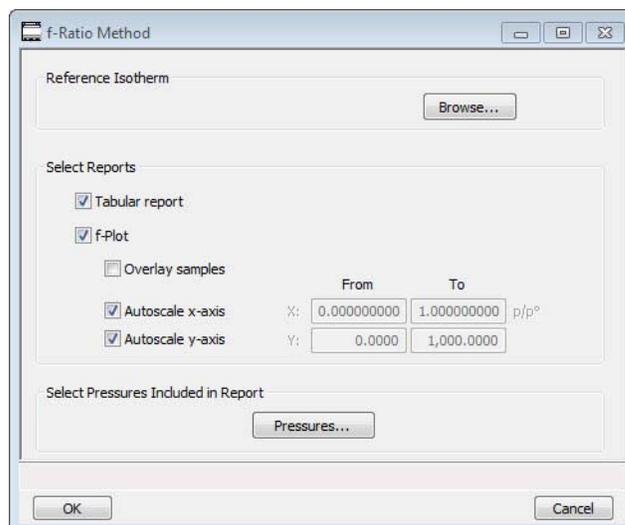
One predefined curve is shipped with the analysis program and is located in the Reference directory. Use the table to enter relative pressure and the alpha-s values.

Field or Button	Description
<i>Open button</i>	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.
<i>Insert / Delete / Clear / Append buttons</i>	Refer to Table buttons , page 3-4 for a description of the Insert , Delete , Clear , and Append buttons.

Field or Button	Description (<i>continued</i>)
<i>Ref. surface area text box</i>	Enter the surface area from the reference curve. This value is used to calculate the sample surface area.
<i>Select Range for Alpha-S Fit text boxes</i>	Enter minimum and maximum relative pressures to determine the fit.
<i>Tabular report checkbox</i>	Use to have a tabular report of data generated.
<i>Alpha-S plot checkbox</i>	<p>Use to plot data in graph format.</p> <ul style="list-style-type: none"> • Overlay samples checkbox - 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.
<i>Pressures button</i>	Refer to Pressures button , page 3-38 .
<i>Browse button</i> <i>Cancel button</i> <i>From / To buttons</i> <i>OK button</i> <i>Save As button</i> <i>Table buttons</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1 .

f-Ratio Method

The f-Ratio report uses the measured isotherm and normalizes it using a reference isotherm. In the **Selected Reports** list box, highlight **f-Ratio Method**, then click **Edit**.



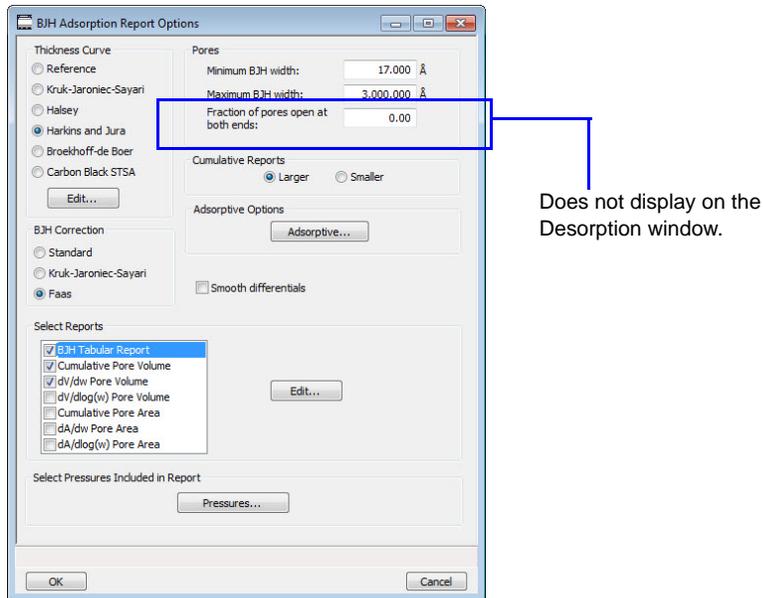
Field or Button	Description
<i>Reference isotherm</i>	Click Browse to select a sample file to use as a reference for the isotherm. Select a file containing an isotherm measured from a non-porous sample of the same material as the current sample. When the referenced file is selected, the file name appears to the left of the Browse button.
<i>Tabular report checkbox</i>	Select to have a tabular report of data generated.
<i>f-Plot checkbox</i>	Use to generate a normalized isotherm. <ul style="list-style-type: none"> • Overlay samples checkbox - 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.
<i>Pressures button</i>	Refer to Pressures button , page 3-38 .
<i>Browse button</i> <i>Cancel button</i> <i>From / To text boxes</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1 .

BJH Adsorption/Desorption Report Options

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 report options are identical unless otherwise specified.

In the **Selected Reports** list box, highlight **BJH Adsorption** (or **BJH Desorption**), then click **Edit**.



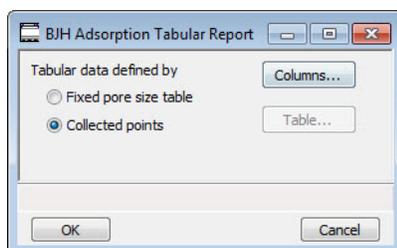
Field or Button	Description
<i>Thickness Curve group box</i>	Refer to Thickness Curve group box , page 3-41.
<i>BJH Correction group box</i>	Select the type of correction to apply to calculations. The selected type displays in the report header. <ul style="list-style-type: none"> • Standard - uses original BJH models • Kruk-Jaroniec-Sayari - good for reference thickness curves • Faas - good for statistical thickness curves

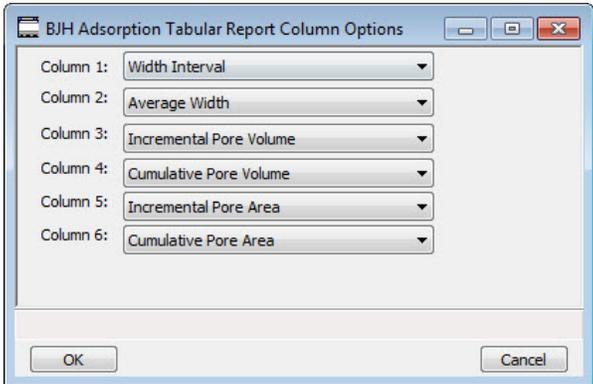
Field or Button	Description (<i>continued</i>)																		
<i>Pores group box</i>	<p>Enter the minimum and maximum diameter (radius or width) of pores to include in the BJH reports.</p> <ul style="list-style-type: none"> • Fraction of pores open at both ends - This field is not available for the BJH Desorption Report Options window. <p>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.</p>																		
<i>Cumulative Reports options</i>	<ul style="list-style-type: none"> • 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. 																		
<i>Adsorptive button</i>	<p>Displays the BJH Adsorptive Options window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.</p> <div data-bbox="691 1050 1195 1583" data-label="Image"> <table border="1"> <thead> <tr> <th>Adsorptive</th> <th>Adsorbate Property Factor (Å)</th> </tr> </thead> <tbody> <tr> <td>1: N2</td> <td>9.53000</td> </tr> <tr> <td>2: Ar</td> <td>10.44000</td> </tr> <tr> <td>3:</td> <td>0.00000</td> </tr> <tr> <td>4:</td> <td>0.00000</td> </tr> <tr> <td>5:</td> <td>0.00000</td> </tr> <tr> <td>6:</td> <td>0.00000</td> </tr> <tr> <td>7:</td> <td>0.00000</td> </tr> <tr> <td>8:</td> <td>0.00000</td> </tr> </tbody> </table> </div>	Adsorptive	Adsorbate Property Factor (Å)	1: N2	9.53000	2: Ar	10.44000	3:	0.00000	4:	0.00000	5:	0.00000	6:	0.00000	7:	0.00000	8:	0.00000
Adsorptive	Adsorbate Property Factor (Å)																		
1: N2	9.53000																		
2: Ar	10.44000																		
3:	0.00000																		
4:	0.00000																		
5:	0.00000																		
6:	0.00000																		
7:	0.00000																		
8:	0.00000																		
<i>Smooth differentials checkbox</i>	<p>Use to smooth all differential calculations thus eliminating variations in the differential computation caused by noise in the input data.</p>																		
<i>Selected Reports list box</i>	<p>Select the checkbox to the left of the report names to include in the report. Highlight the report name and click the Edit button to modify report parameters.</p>																		

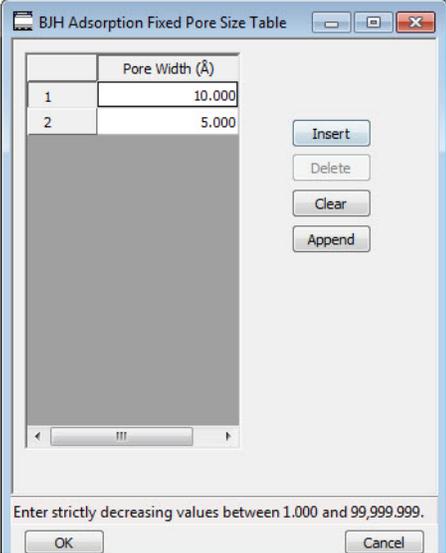
Field or Button	Description (<i>continued</i>)
<i>Pressures button</i>	Refer to Pressures button , page 3-38.
<i>Cancel button</i> <i>Edit button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

Tabular Report Options

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

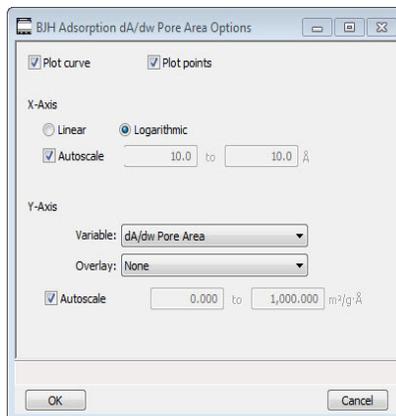


Field or Button	Description
<i>Fixed pore size table</i>	Use to specify exact pore sizes for volume or area data. Click the Table button to modify the fixed pore size table. Refer to Table and Columns below for information on the use of these buttons.
<i>Collected points</i>	Use to include all relative pressure points collected. Refer to the Columns button shown below.
<i>Columns button</i>	Select the data types to include in the report. Column [n] indicates the column order and data contents for the report. 

Field or Button	Description (<i>continued</i>)
<p>Table button</p>	<p>The fixed pore size table must contain a minimum of two points. The points must be strictly decreasing. Enabled only when Fixed pore size table is selected.</p> 
<p>Cancel button OK button Table buttons</p>	<p>Refer to Common Fields and Buttons - File Menu Options, page 3-1.</p>

Plot Options

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 and click **Edit**.



Field or Button	Description
<i>Plot curve / Plot points checkboxes</i>	Refer to Isotherm Report Options , page 3-31 .
<i>X-Axis</i>	Use to have the x-axis on a logarithmic or linear scale.
<i>Y-Axis</i>	<ul style="list-style-type: none"> • Variable dropdown list - select a variable. • Overlay dropdown list - select an option to overlay on the current report.
<i>Autoscale checkbox</i> <i>Cancel button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1 .

Dollimore-Heal Adsorption/Desorption Report Options

This report option generates Dollimore-Heal reports from both adsorption and desorption data. In the **Selected Reports** list box, highlight **Dollimore-Heal Adsorption** (or **Dollimore-Heal Desorption**), then click **Edit**.

Dollimore-Heal Adsorption/Desorption fields and buttons are identical to the **BJH Adsorption/Desorption Report Options**, page 3-47.

Or Dollimore-Heal
Desorption Report
Options

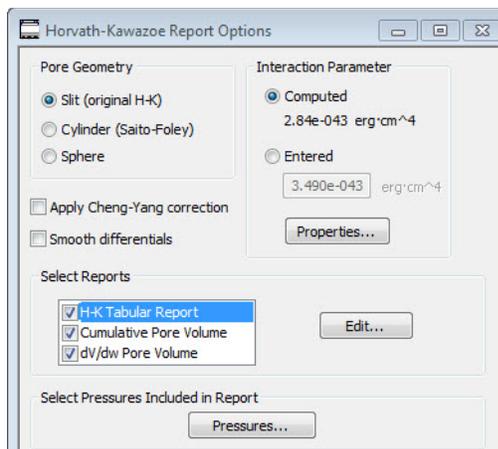
The screenshot shows the 'Dollimore-Heal Adsorption Options' dialog box. It is divided into several sections:

- Thickness Curve:** Radio buttons for Reference, Kruk-Jaroniec-Sayari, Halsey, Harkins and Jura (selected), Broekhoff-de Boer, and Carbon Black STSA. An 'Edit...' button is below.
- Pores:** Input fields for Minimum Pore width (17,000 Å) and Maximum Pore width (3,000,000 Å).
- Cumulative Reports:** Radio buttons for Larger (selected) and Smaller.
- Adsorptive Options:** An 'Adsorptive...' button.
- Smooth differentials:** A checkbox that is currently unchecked.
- Select Reports:** A list box with the following items:
 - Dollimore-Heal Tabular Report
 - Cumulative Pore Volume
 - dV/dw Pore Volume
 - dV/dlog(w) Pore Volume
 - Cumulative Pore Area
 - dA/dw Pore Area
 - dA/dlog(w) Pore AreaAn 'Edit...' button is to the right of the list.
- Select Pressures Included in Report:** A 'Pressures...' button.

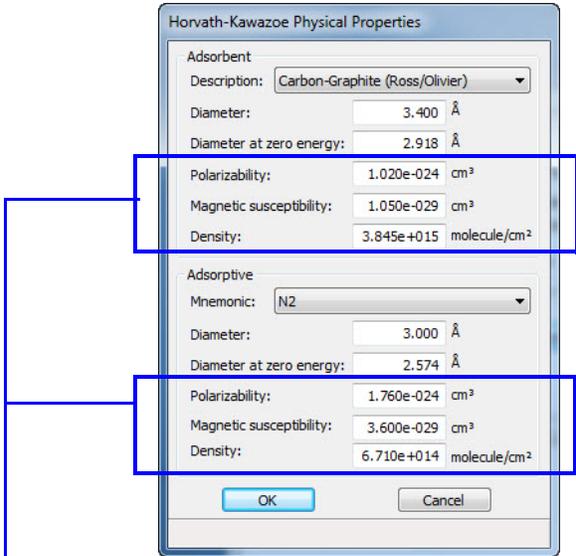
At the bottom of the dialog are 'OK' and 'Cancel' buttons.

Horvath-Kawazoe Report Options

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. In the **Selected Reports** list box, highlight **Horvath-Kawazoe**, then click **Edit**.



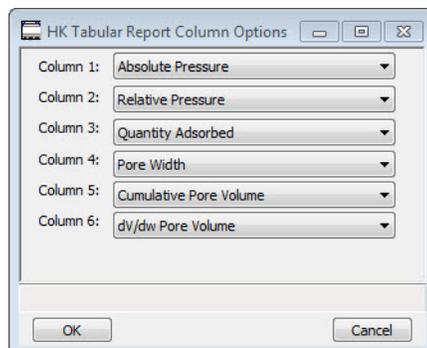
Field or Button	Description
<i>Pore Geometry</i> group box	Select the option that best represents the physical geometry of the micropores in the sample material. When Sphere is selected, options in the Interaction Parameter group box are disabled.
<i>Interaction Parameter</i> group box	Use to determine which interaction parameter will be used in the report. These options are disabled if Sphere is selected in the Pore Geometry group box. <ul style="list-style-type: none"> • Computed - use to calculate using the parameters on the Horvath-Kawazoe Physical Properties window (click the Properties button to display the Physical Properties window). The interaction parameter is recalculated each time a parameter in the Physical Properties window is edited. • Entered - select to calculate using the value entered in the text box.

Field or Button	Description (<i>continued</i>)
Properties button	<p>Click to view or edit the constants describing the physical properties of the adsorbent and adsorptive.</p>  <p>These options are disabled if Entered is selected in the Interaction Parameter group box.</p> <p>Adsorbent group box:</p> <p>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.</p> <ul style="list-style-type: none"> • 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: $(2/5)^{1/6} \times \text{diameter}$. • 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.

Field or Button	Description (<i>continued</i>)
<i>Properties button</i> (<i>continued</i>)	<p>Adsorptive group box:</p> <p>Contains the parameters for the adsorptives (provided with the software and/or user-defined). 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.</p> <ul style="list-style-type: none"> • 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: $(2/5)^{1/6} \times \text{diameter}$. • 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.
<i>Apply Cheng-Yang correction checkbox</i>	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.
<i>Smooth differentials checkbox</i>	Refer to BJH Adsorption/Desorption Report Options , page 3-47 .
<i>Selected Reports list box</i>	Select the types of reports to generate.
<i>Cancel button</i> <i>Edit button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1 .

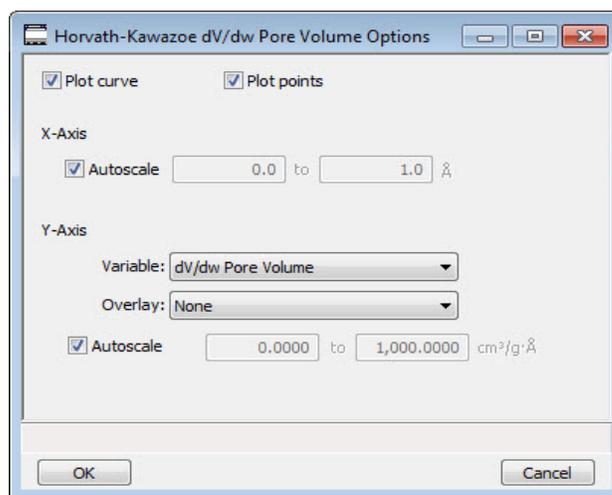
Tabular Report Options

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



Plot Options

Highlight a plot option in the **Selected Reports** list box in the **Horvath-Kawazoe Report Options** window and click **Edit** to customize the plotting method.

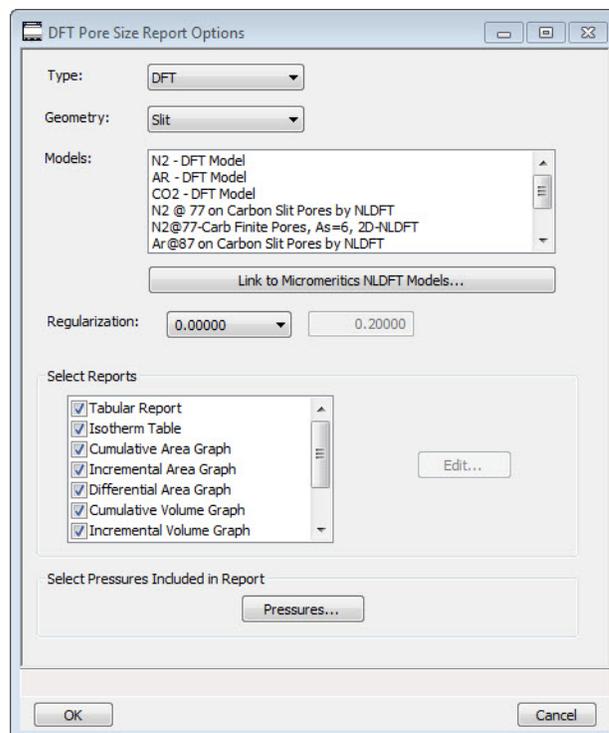


Field or Button	Description
<i>Plot curve / Plot points checkboxes</i>	Use to plot curves and/or points.

Field or Button	Description (<i>continued</i>)
<i>X-Axis / Y-Axis</i>	<ul style="list-style-type: none">• X-Axis - the x-axis field shows pore radius or diameter in angstroms or nanometers.• Y-Axis - the y-axis field shows the quantity adsorbed.• Variable dropdown list - select a y-axis variable for the report.• Overlay dropdown list - select an option to overlay on the current report.
<i>Autoscale checkbox</i> <i>Cancel button</i> <i>Edit button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1

DFT Pore Size Report Options

The Dubinin method provides pore volume distributions for microporous materials by making use of an expression for the adsorption potential. In the **Selected Reports** list box, highlight **Dubinin**, then click **Edit**



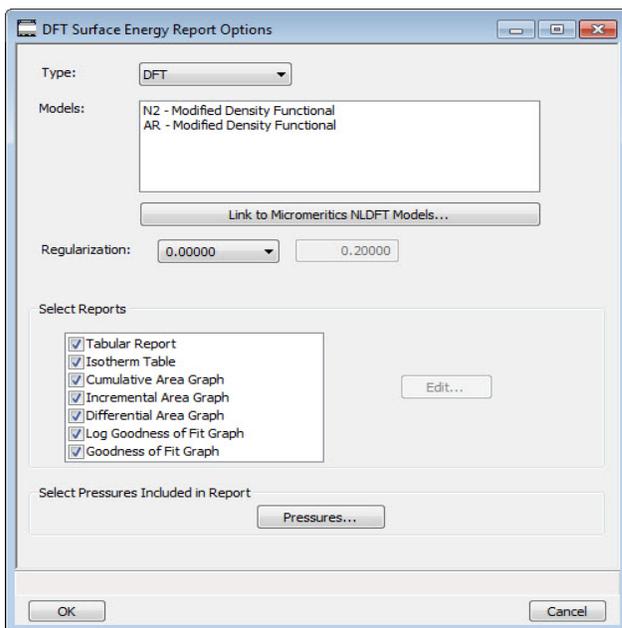
Field or Button	Description
<i>Type dropdown list</i>	<ul style="list-style-type: none"> • DFT - model based on the density functional theory. • Classical - model based on the Kelvin equation and thickness for determining the pore size distribution. <p>Refer to DFT MODELS, page F-1 for further discussion on models.</p>
<i>Geometry dropdown list</i>	Select the pore shape.
<i>Models list box</i>	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.
<i>Link to Micromeritics NLDFT Models button</i>	Click to hyperlink to the NLDFT Model Table on the Micromeritics web page.

Field or Button	Description (<i>continued</i>)
<p>Regularization dropdown list and text box</p>	<p>Select the extent of smoothing to apply to the data.</p> <p>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.</p>
<p>Selected Reports group box</p>	<p>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.</p> <div data-bbox="891 611 1318 1031" data-label="Image"> </div> <ul style="list-style-type: none"> • Plot Type group box - select the method for data display. • Autoscale Options group box - use to autoscale the x-axis and/or y-axes. • Overlay dropdown list - select an overlay for the report. • Axis Range group box - From / To fields are enabled when Autoscale options are not selected. Enter the starting and ending values for the x- and/or y-axes. <ul style="list-style-type: none"> – X-axis - shows the pore size. – Y-axis - shows the area.
<p>Pressures button</p>	<p>Refer to Pressures button, page 3-38.</p>
<p>Cancel button From / To text boxes OK button</p>	<p>Refer to Common Fields and Buttons - File Menu Options, page 3-1.</p>

DFT Surface Energy Report Options

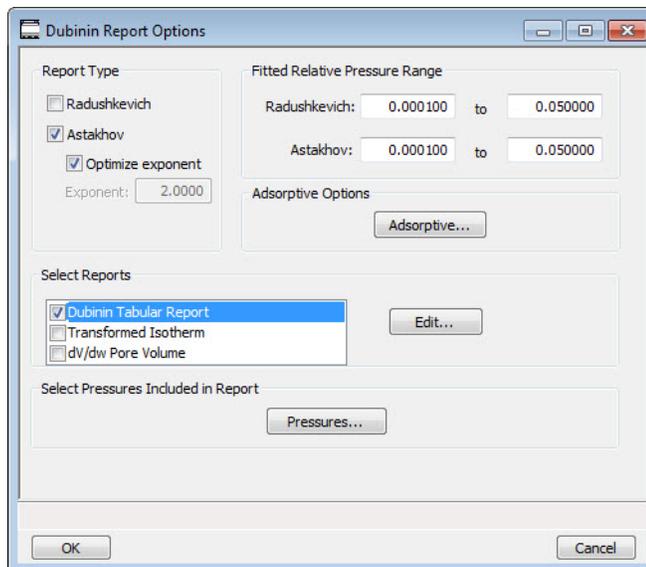
The DFT Surface Energy report contains the results of surface energy distribution analyses. In the **Selected Reports** list box, highlight **DFT Surface Energy**, then click **Edit**.

DFT Surface Energy Report Options fields and buttons are identical to the **DFT Pore Size Report Options**, page 3-58.



Dubinin Report Options

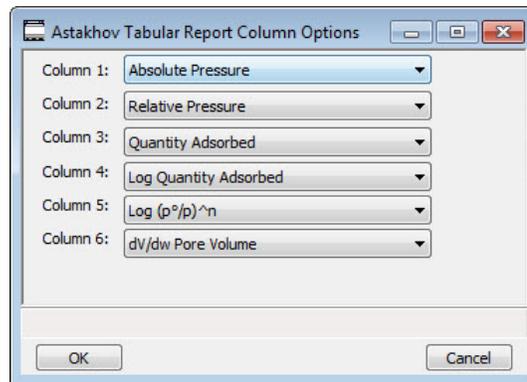
The Dubinin method provides pore volume distributions for microporous materials by making use of an expression for the adsorption potential. In the **Selected Reports** list box, highlight **Dubinin**, then click **Edit**.



Field or Button	Description
<i>Report Type group box</i>	Select report types. If Astakhov is selected, either select the Optimize exponent checkbox or enter an appropriate exponent value in the text box.
<i>Fitted Relative Pressure Range text boxes</i>	Enter the minimum and maximum limits for Radushkevich or Astakhov relative pressures included in the line fit.
<i>Selected Reports list box</i>	Select the reports to generate. Highlight the report and click the Edit button to modify report options.
<i>Pressures button</i> <i>Adsorptive button</i>	Refer to BJH Adsorption/Desorption Report Options , page 3-47.
<i>Cancel button</i> <i>Edit button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

Tabular Report Options

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

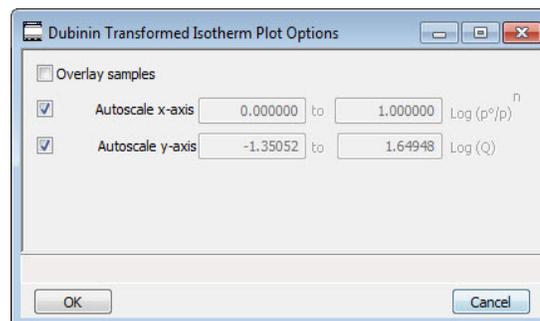


Log (p°/p)ⁿ - the value for **[n]** is the optimized exponent if **Optimized exponent** is selected on the **Dubinin Report Options** window. If not, then the value for **[n]** is the entered exponent value.

Transformed Isotherm Plot Options

Highlight **Transformed Isotherm** in the **Selected Reports** list box in the **Dubinin Report Options** window and 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.



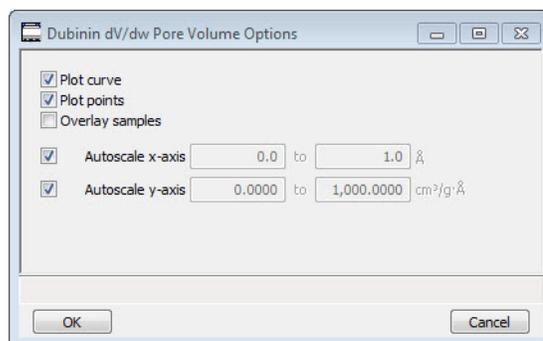
Field or Button	Description
<i>Overlay samples</i> checkbox	Use to overlay sample files on the plot.

Field or Button	Description (<i>continued</i>)
<i>Autoscale x-axis / Autoscale y-axis checkboxes</i>	<p>Select an option to have the axis scaled automatically. Both axes begin at 0; the system uses the maximum values collected during analysis as the ending points for axis ranges.</p> <p>To enter beginning and ending values manually, deselect these checkboxes.</p> <p>Autoscale x-axis - shows the quantity of gas adsorbed at standard temperature and pressure.</p> <p>Autoscale y-axis - shows the log of relative pressure.</p>
<i>Cancel button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

Pore Volume Options

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

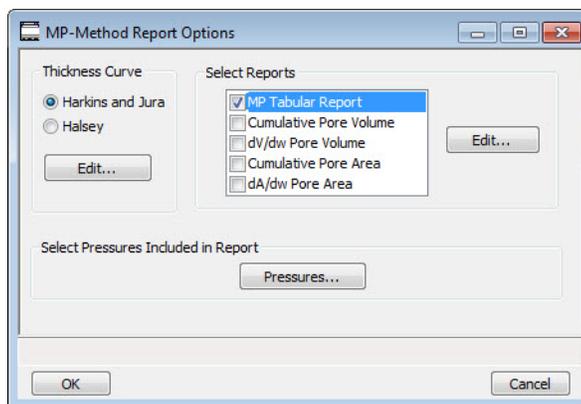
This option plots differential pore volume as a function of pore width.



Field or Button	Description
<i>Plot curve / Plot points checkboxes</i>	Use to plot curves and/or points.
<i>Overlay samples checkbox</i>	Use to overlay sample files on the plot.
<i>Autoscale x-axis / Autoscale y-axis checkboxes</i>	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. To enter beginning and ending values manually, deselect these checkboxes.
<i>Cancel button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

MP-Method Report Options

The MP-Method 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. In the **Selected Reports** list box, highlight **MP-Method**, then click the **Edit** button.

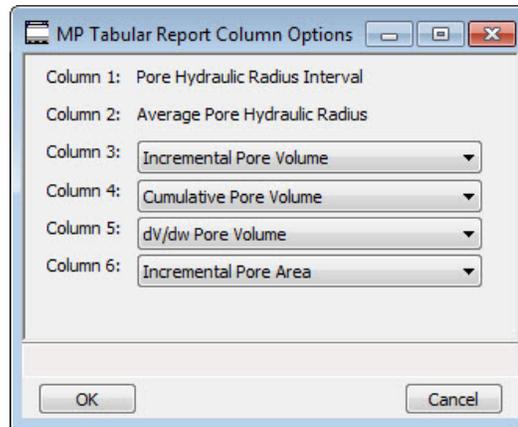


Pore size can be expressed in angstroms or nanometers. Go to *Options > Units* to specify the unit. Refer to **UNIT MENU**, page 4-1.

Field or Button	Description
<i>Thickness Curve group box</i>	Refer to Thickness Curve group box , page 3-41.
<i>Selected Reports list box</i>	Select the reports to generate. Highlight the report and click the Edit button to modify report options.
<i>Pressures button</i>	Refer to Pressures button , page 3-38.
<i>Cancel button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

Tabular Report Options

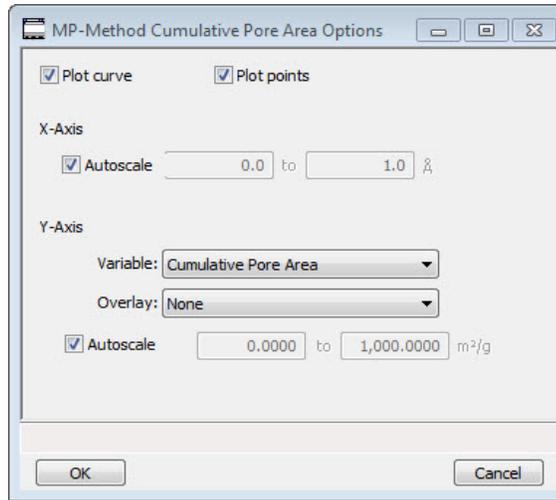
In the **MP-Method Report Options** window, highlight **MP Tabular Report** in the **Selected Reports** list box and click **Edit**. **Column [n]** indicates the column order and data contents for the report.



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

Plot Options

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

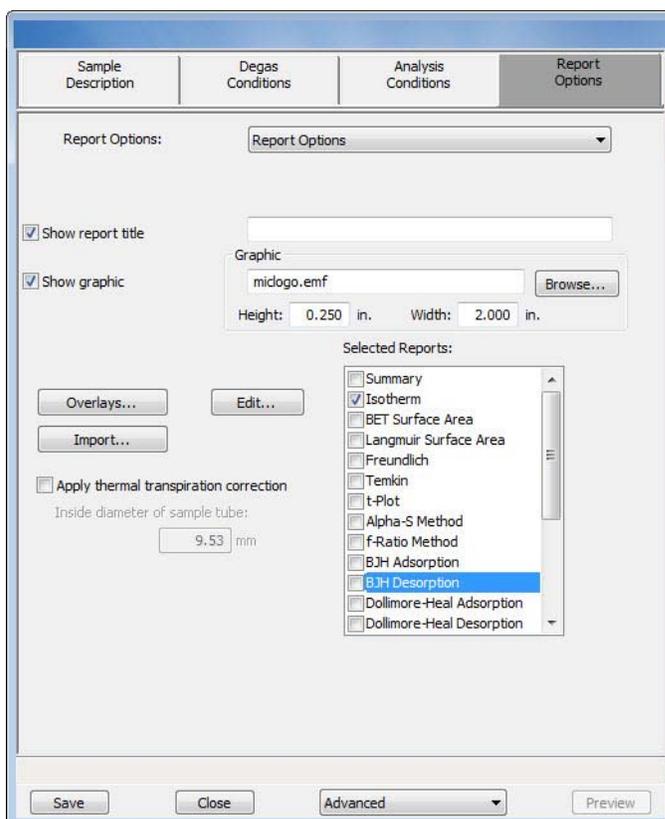


Field or Button	Description
<i>Plot curve / Plot points checkboxes</i>	Use to plot a curve and/or points.
<i>X-Axis</i>	Use to have the x-axis autoscaled or enter beginning and ending values.
<i>Y-Axis</i>	<ul style="list-style-type: none"> • Variable dropdown list - select a variable. • Overlay dropdown list - select an option to overlay on the current report. • Autoscale checkbox - use to have the y-axis autoscaled or enter beginning and ending values.
<i>Cancel button</i> <i>OK button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

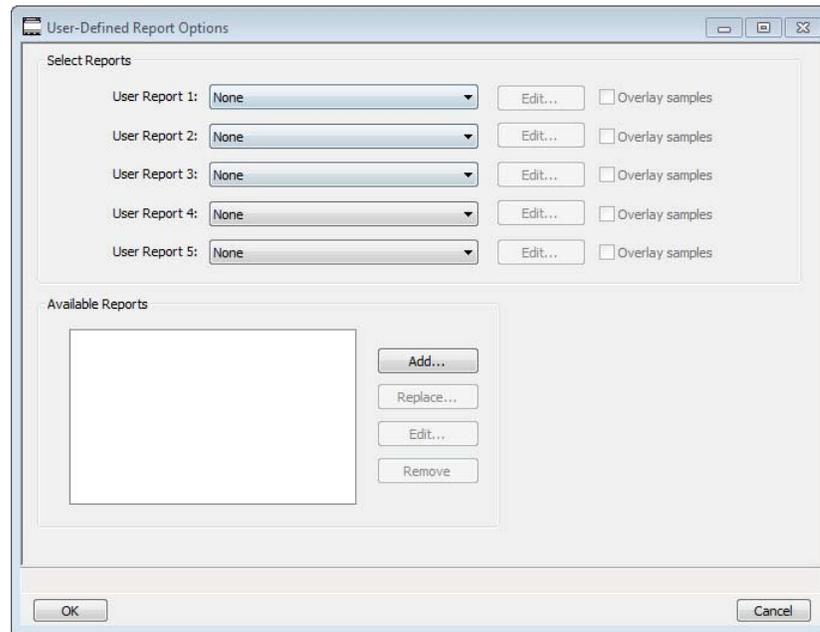
User-Defined Reports

Up to five user-defined reports may be created, each with up to 10 summary reports, 10 tabular reports, and 10 graphical reports. In order to use this feature you must create a file containing a Python script that imports a "mic" python module. Appendix G provides an example of python script and provides functions for the "mic" python Module.

Once python scripts have been created for user-defined reports, you can select the reports from the **Reports Options** window.



Click **User-Defined**, then click **Edit**. The **User-Defined Report Options** window is displayed.



Field or Button	Description
<i>User Report 1 through User Report 5</i>	Use the dropdown lists to select currently-defined functions used to define the report calculations and output.
<i>Edit button</i>	Use to edit a function.
<i>Overlay samples checkbox</i>	Use to overlay samples as defined by the function.
<i>Available Reports group box</i>	Lists the available reports and allows you to replace, edit or remove reports.
<i>Add button</i> <i>Cancel button</i> <i>Edit button</i> <i>OK button</i> <i>Remove button</i> <i>Replace button</i>	Refer to Common Fields and Buttons - File Menu Options , page 3-1.

Options Report

The Options report lists the conditions used to perform the analysis. It contains analysis information including:

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

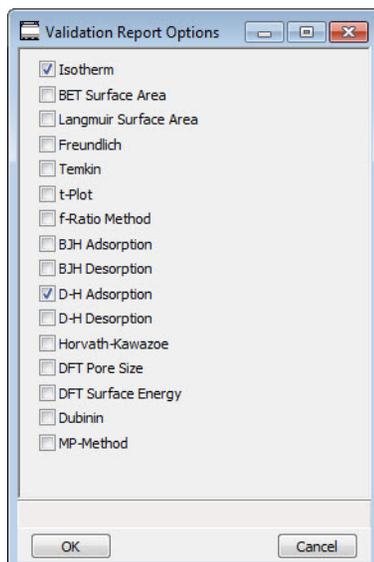
Sample Log Report

In the **Selected Reports** list box, select **Sample Log**. This report provides the following information:

- 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

Validation Report

In the **Selected Reports** list box, highlight **Validation**, then 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.



4. UNIT MENU

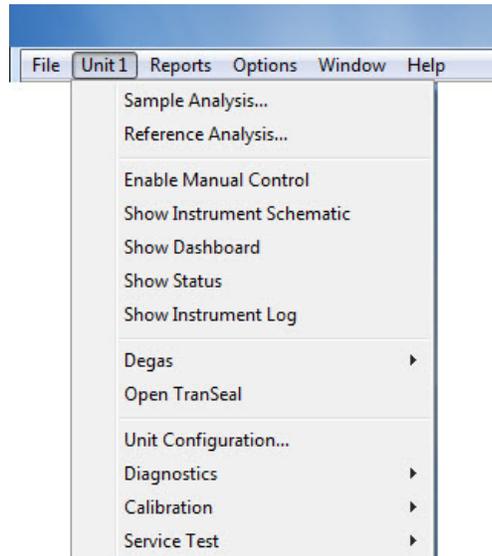
Introduction

This chapter contains information specific to the Unit menu options. These options are used to run analyses on one or more units attached to a controlling computer. This chapter provides details of Unit menu options, and descriptions of commonly used functions and buttons.

The title bar of the main window displays as *Unit [n]* for each attached unit. Each unit has a status window displayed in different colors.



Common field and button descriptions are listed in a Common table at the beginning of their respective chapters. Field and button descriptions not listed in the Common table are listed in their appropriate heading.



Common Fields and Buttons - Unit Menu Options

The following fields and buttons are common to many of the Unit menu options. References are made to these fields and buttons throughout this manual.

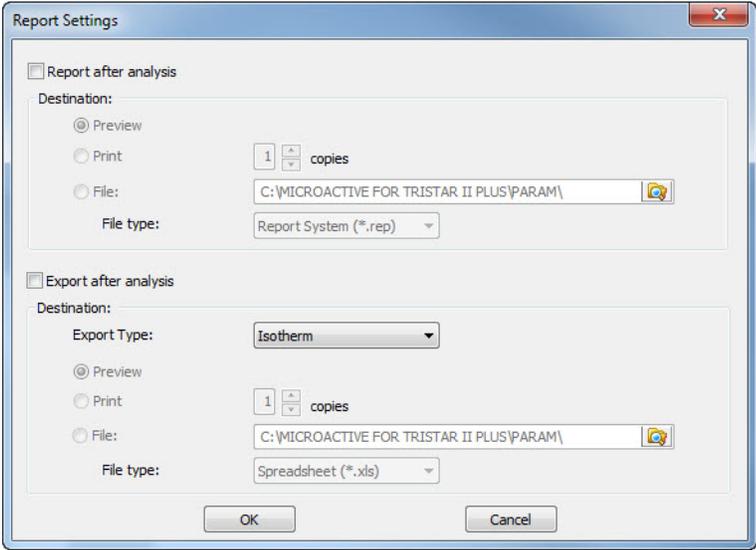
Field or Button	Description
<i>Browse button</i>	Click to search for a file. Select a file from either the Name column or from the library and click Open or double click the file name to open (or import) the file.
<i>Cancel button</i>	Discards any changes made to the screen or cancels the current process. On the Analysis window, at the prompt, choose the port(s) to cancel.
<i>Close button</i>	Click to close the active window.
<i>Edit button</i>	On the analysis window, click to edit the sample information file. Refer to Sample Information Files , page 3-10 .
<i>OK button</i>	Click to save and close the active window.
<i>Start button</i>	Click to start an analysis or calibration procedure.

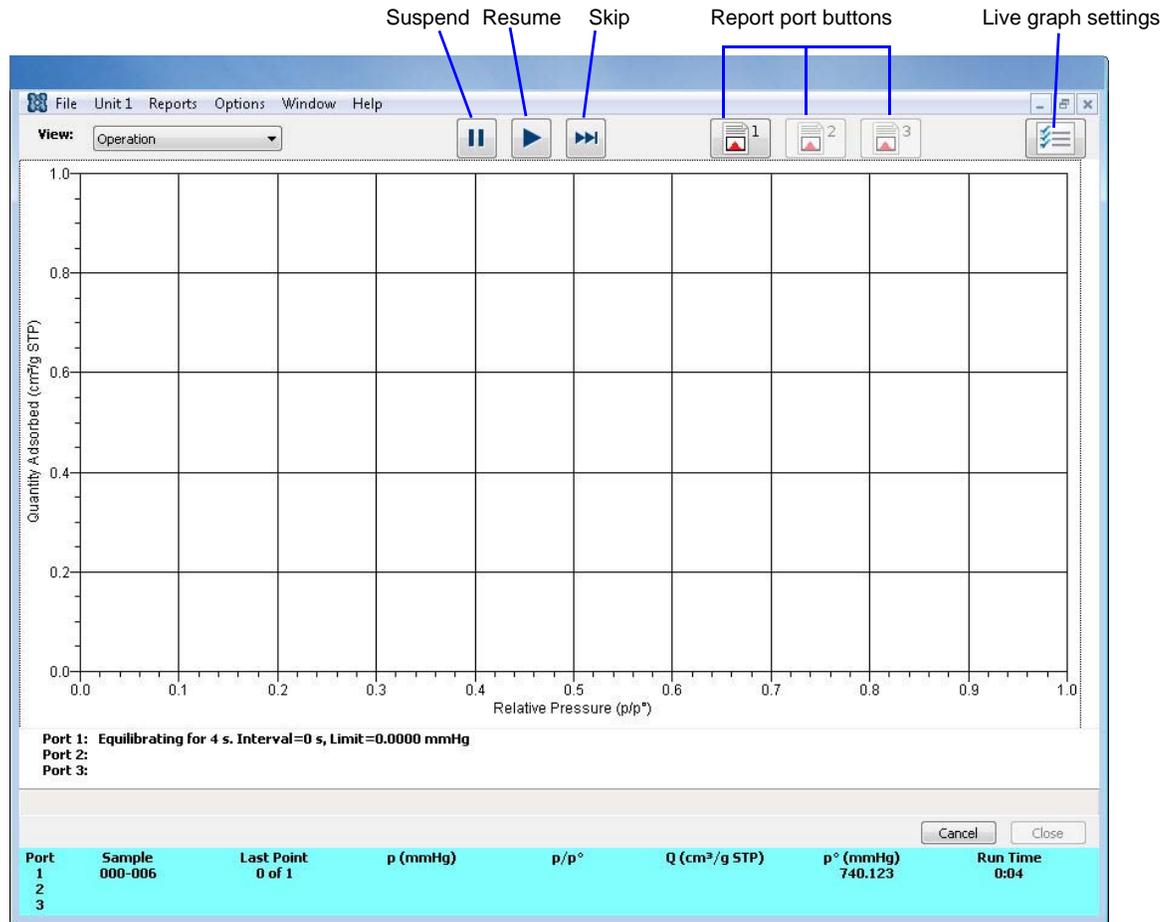
Sample Analysis

Unit [n] > Sample Analysis

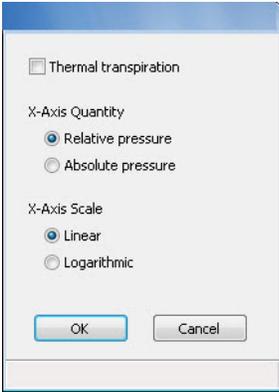
Use to schedule up to three analyses with different analysis conditions and/or report options. Sample files can be loaded into ports, 1, 2, and 3 in the order they appear on the screen.

Field or Button	Description
<i>View dropdown list</i>	<ul style="list-style-type: none"> • Operation - use to display the current mode of operation. • Instrument Log - use to display recent analyses, calibrations, errors or messages. Refer to Show Instrument Log, page 4-20. • Instrument Schematic - use to display a schematic of the analyzer system. Refer to Show Instrument Schematic, page 4-15.
<i>Close Valves button</i>	Click to close all valves on the unit.
<i>New button</i>	Click to create a new sample information file.

Field or Button	Description (<i>continued</i>)
Browse button	Click to select a sample file to be used for analysis on the associated port. On Port 1, you can select up to three sample files. The files will be loaded into ports 1, 2, and 3 in the order they appear in the file selector.
Edit button	Click to edit the selected sample file.
Clear button	Click to clear all fields for this port.
Density / Mass / Sample + Tube / Empty Tube text boxes	Enter default values for the sample's mass and density. Refer to Mass group box , page 3-12.
p^0 text box	Enabled if Entered is selected for the P_0 measurement for at least one file.
Bath temperature text box	Enter the temperature for the analysis bath.
Report after analysis button	<p>Click to select a print destination.</p>  <ul style="list-style-type: none"> • Report after analysis checkbox - use to send the report to the screen, printer, or file in either ASCII, .XLS, or REP format. • Export after analysis checkbox - use to export Isotherm data to the screen, printer, or file in either ASCII, or .XLS format. <p>Refer to Destination group box in New Sample, page 3-6.</p>



Field or Button	Description
<i>Suspend</i> button	Click to suspend an analysis in progress. Select the ports containing the analysis to suspend.
<i>Resume</i> button	Click to resume an analysis. Select the ports containing the analysis to resume.
<i>Skip</i> button	Click to skip to the next step. This button is visible only when an analysis is in progress. Select the ports containing the step to skip.
<i>Report Port [n]</i> button	Click to generate reports on data being collected on the respective port. The reports are printed to the screen only.

Field or Button	Description (<i>continued</i>)
<i>Live Graph Settings button</i>	<p>Allows you to select Thermal transpiration, X-axis Quantity (relative or absolute pressure) and the X-Axis Scale (linear or logarithmic).</p> 
<i>Status window</i>	<p>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, $0.1 \leq P < 10$: 4 digits, $P \geq 10$: 2 digits. Relative pressure will show 3 more digits than absolute pressure.</p>
<i>Browse button</i> <i>Cancel button</i> <i>OK button</i>	<p>Refer to Common Fields and Buttons - Unit Menu Options, page 4-2.</p>

Reference Analysis

Unit [n] > Reference Analysis

A reference analysis is used to verify that the instrument is operating properly and producing optimum results. These methods provide specifications for critical report quantities and reporting of whether the quantities are in or out of specification. Predefined SPC reports are provided that include all sample files in a specified directory for a given reference and show SPC results for the critical report quantities.

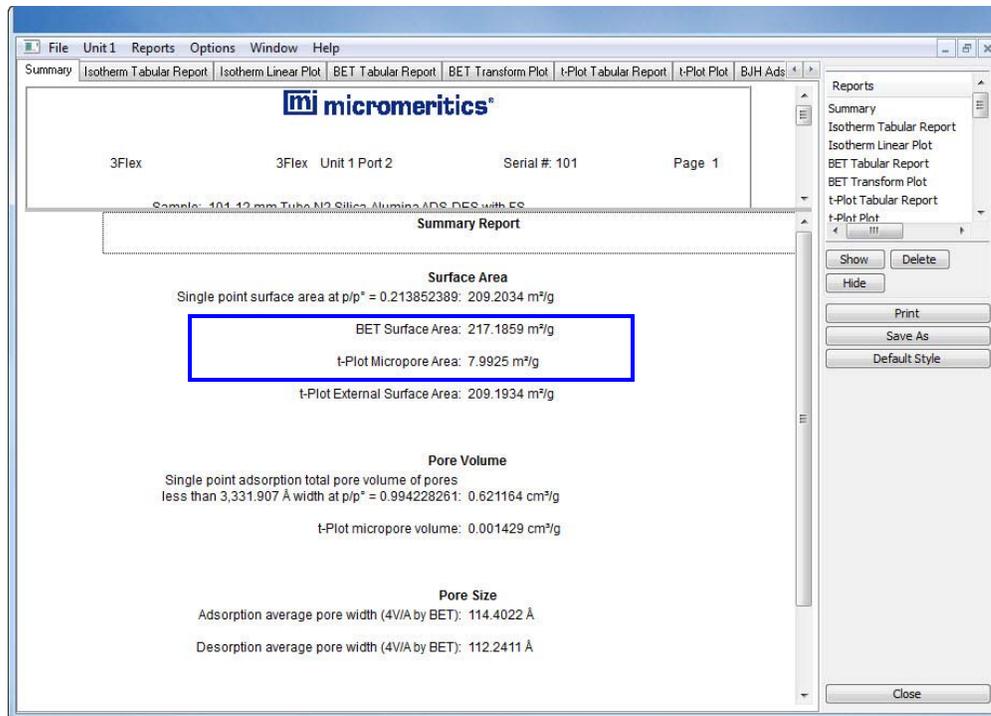
When running a reference analysis, use the Silica Alumina reference material provided in the accessories kit to perform this analysis. The results should match those shown on the label of the reference material bottle, within the tolerance level.

Refer to [Defining Sample Information Files](#), page 2-6 for instructions on creating a sample file.

1. Go to *Unit [n] > Reference Analysis*.
2. Click **Browse** from the **Method** dropdown list and select a method.
3. To manually close all instrument valves, click the **Close Valves** button.

4. Enter Sample Mass for each sample. Verify the information populated into the remaining fields is correct and modify if necessary. This information is pulled from the selected file. The density value is applicable only if using the **Calculate** method for the free space determination.
5. Edit the **p°** and **Bath temperature** fields, if necessary.

6. Click **Report after analysis** to automatically generate reports when the analysis is complete. On the **Report Settings** window, select the report destination. Click **OK**.
7. 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 screen changes from the Idle state.
8. 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 for Ports 2 and 3.



- If the results are within tolerance, the instrument is operating properly. If the BET Surface Areas match, click **Close**.
- If the results are not within tolerance, refer to the following table for possible causes and actions. After performing the action, perform the reference analysis again

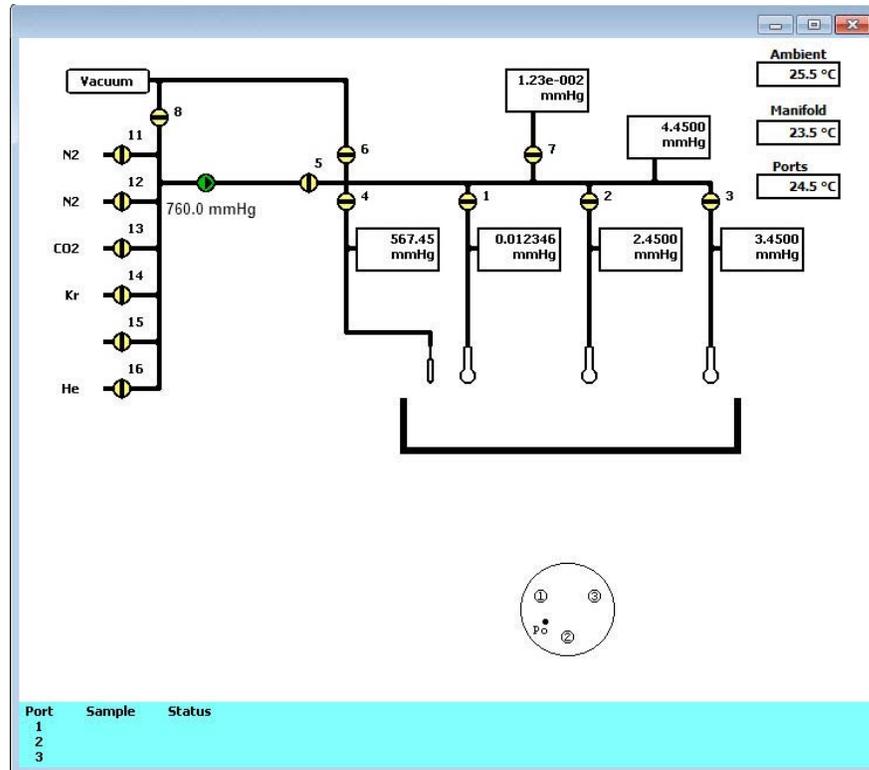
Cause	Action
The sample was not degassed properly.	Degas the sample again.
The gas lines are not clean.	Perform the procedure for cleaning and verifying gas lines, then try again.
The measured free space is too high. This indicates the helium is not pure enough.	Use helium that is 99.999% pure, then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

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 the menu item.

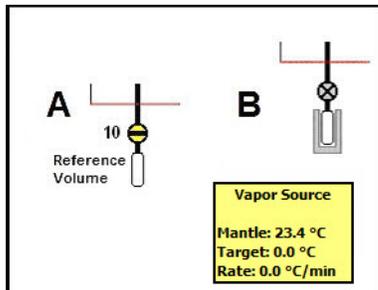
If the following instrument schematic is not immediately visible, go to *Unit [n] > Show Instrument Schematic*.



Alternate Schematic Icons

P^o Port

The following icons will display instead of the p^o port icon under the conditions described below.



A - Reference volume icon displays when a reference volume is attached to the instrument (Service test mode only).

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

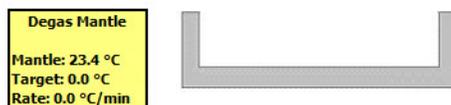
Transducers

The schematic shows the transducers present in the system.

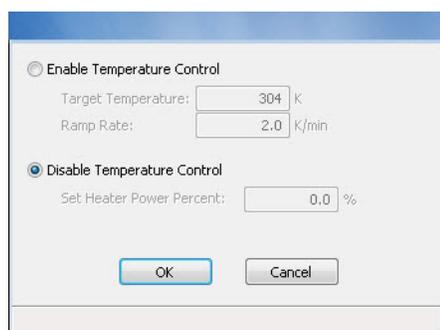
- All systems have 1000 mmHg transducers on the manifold and the analysis ports.
- Optional 10 mmHg transducers on the manifold and one or more analysis ports may be present.
- Optional 0.1 mmHg transducers on one or more analysis ports may be present.

Heating Mantle

When samples are being degassed on the analysis ports, the following icons appear beneath the elevator. When the target temperature is reached, the sample tube icons turn red.



Right-click on the Degas Mantle box to change the target temperature or the rate of temperature increase.



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

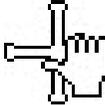
System Valves

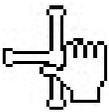
Go to *Unit [n] > Show Instrument Schematic* to display the instrument schematic.

Valves	Description
1 - 3	Sample ports
4	Po port
5	Servo Isolation Valve
6	Manifold Vacuum
7	Vacuum Gauge
8	Inlet Vacuum
10	Reference Volume (shown in Service Test mode only)
11 - 16	Inlet Valves

Instrument Schematic Shortcut Menus

Each manually controlled component has a shortcut menu displaying the operations available for that particular component, such as, Open, Close, Pulse. To access the shortcut menu, hover the mouse cursor over the component and right click.

Shortcut Icon	Description
	<p>When the cursor changes to this icon, right click to display options for the selected component:</p> <ul style="list-style-type: none"> For Valves 1-8, 11-16 <ul style="list-style-type: none"> Open - opens the selected valve. The valve symbol changes to green. An alternate method is to either double click the valve or select the valve and press the keyboard spacebar to turn it off/on. Close - closes the selected valve. The valve symbol changes to yellow. An alternate method is to either double click the valve or select the valve and press the keyboard spacebar to turn it off/on. Pulse - use to quickly turn the valve on and off allowing the operation to proceed in small increments. For Servo Valve: <ul style="list-style-type: none"> Set - use to set the servo valve target pressure and to Dose or Evacuate. <div data-bbox="764 1073 1279 1688" data-label="Image"> </div> <ul style="list-style-type: none"> Open - opens the servo valve. The valve symbol changes to green. Close - closes the servo valve. The valve symbol changes to solid black.

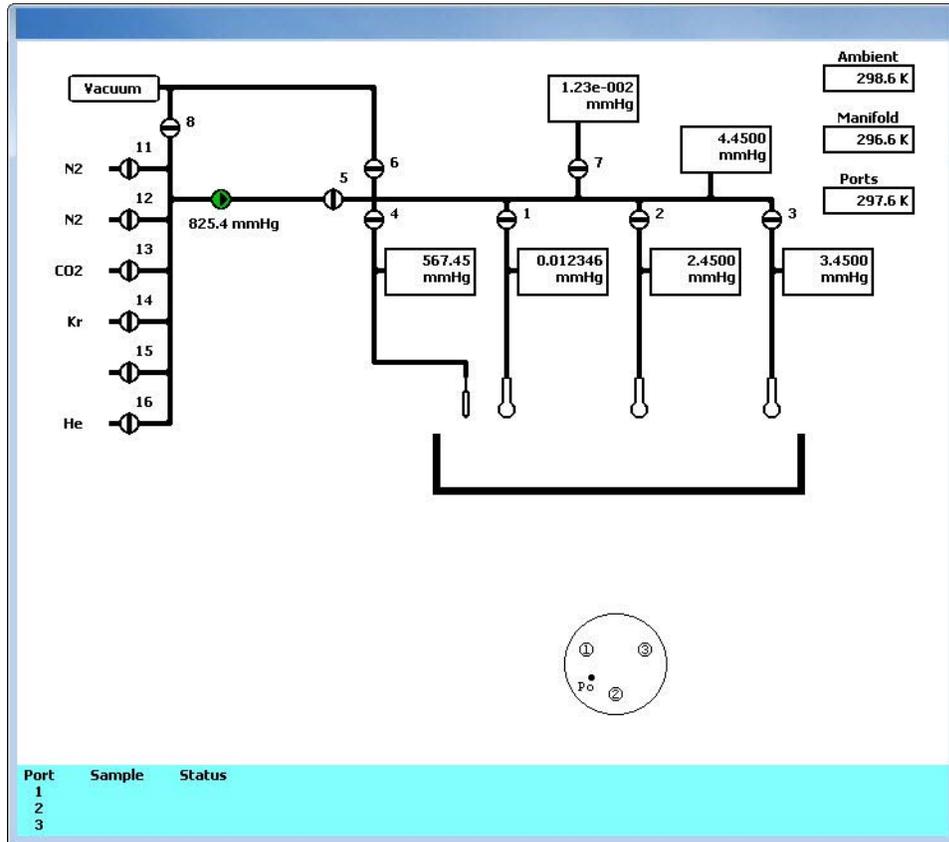
Shortcut Icon	Description (<i>continued</i>)
	<p>Automatic - automatically operates the servo valve during dosing or evacuation. Enter the target pressure.</p> <p>Direct - used in Service Test mode only under the direction of a Micromeritics service representative.</p>
	<p>When the cursor changes to this icon, right click to display options for the elevator:</p> <ul style="list-style-type: none"> • Raise - raises the elevator. Select Raise and press the keyboard space bar to raise the elevator. • Lower - lowers the elevator. Select Lower and press the keyboard space bar to lower the elevator. • Stop - use to stop the elevator from raising or lowering.
	<p>When the cursor changes to this icon, right click to enable or disable the temperature control.</p> <p>Heating Mantle - Set Heating Mantle</p> <div data-bbox="708 1056 1148 1381" style="border: 1px solid #ccc; padding: 10px; margin: 10px auto; width: fit-content;"> <p><input checked="" type="radio"/> Enable Temperature Control</p> <p>Target Temperature: <input type="text" value="31"/> °C</p> <p>Ramp Rate: <input type="text" value="2.0"/> °C/min</p> <p><input type="radio"/> Disable Temperature Control</p> <p>Set Heater Power Percent: <input type="text" value="0.0"/> %</p> <p style="text-align: center;"> <input type="button" value="OK"/> <input type="button" value="Cancel"/> </p> </div> <p>Refer to Heating Mantle, page 4-12 for field descriptions.</p>

Show Instrument Schematic

Unit [n] - Show Instrument Schematic

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

Refer to [Instrument Schematic Components](#), page 4-10 for details on this schematic.



Valve State	Description
	Green indicates an open valve.
	Yellow indicates a closed valve.

Show Dashboard

Unit [n] > Show Dashboard

The dashboard displays the following:

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

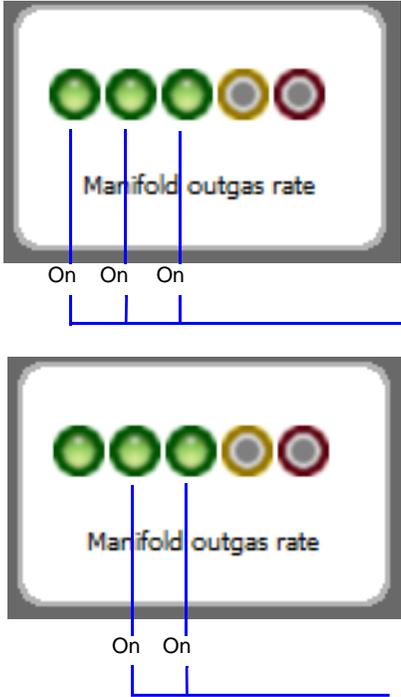
Data for the dashboard comes from the logged diagnostic data. The dashboard is automatically kept current as the relevant diagnostic data are updated. The gauges will be updated even if the dashboard window is not open.

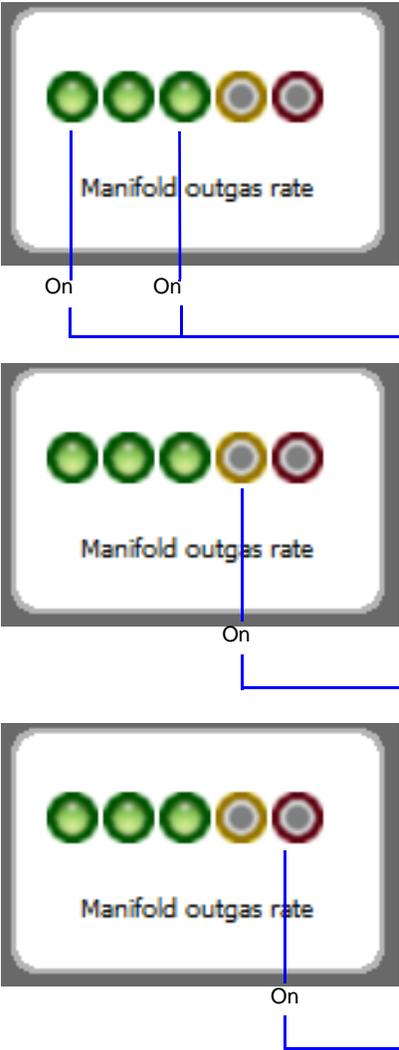


Red numbers on the dashboard require attention.

To reset the dashboard numbers, right-click on the dashboard setting and click Reset.

Gauge	Description
<p><i>Analyses completed/started</i></p> 	<p>Displays <i>N/M</i> where <i>N</i> is the number of analysis that have finished data collection and <i>M</i> 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.</p>

Gauge	Description (<i>continued</i>)
<p><i>Days until roughing-pump service is due</i></p> 	<p>Yearly maintenance is recommended. The number of days until the anniversary of the last pump maintenance are 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.</p>
<p><i>Manifold outgas rate</i></p>	<p>Provides the qualitative indication of the outgas rate in the dosing manifold. LED images constitute a bidirectional bar graph of the outgas rate.</p> <p>The gauge is updated after each outgas rate measurement.</p>  <p>The three green LEDs are lit if the outgas rate is below 30% of the outgas rate limit.</p> <p>At 30%, the left LED turns off.</p>

Gauge	Description (<i>continued</i>)
<p>Manifold outgas rate (<i>continued</i>)</p>	 <p>The diagrams show the gauge's LED indicators at different outgas rate levels:</p> <ul style="list-style-type: none"> 60%: The center green LED turns off. Two other green LEDs remain on. 90%: Three green LEDs turn off and the yellow LED is turned on. 110% and above: Only the red LED is lit and attention is required.
<p>Manifold temperature</p>  <p>26.25±0.05 26.02/26.85 Manifold temperature (°C) mean ± 2s, min/max</p>	<p>Displays the statistics of the manifold temperature reading. The mean, the value at two standard deviations, the minimum, and the maximum display.</p>

Gauge	Description (<i>continued</i>)
<p>Nitrogen Po</p> <div style="border: 1px solid black; padding: 5px; width: fit-content; margin: 10px auto;"> <p style="font-size: 1.2em; margin: 0;">736.50 ± 0.00</p> <p style="font-size: 1.2em; margin: 0;">736.50/736.50</p> <p style="font-size: 0.8em; margin: 0;">Nitrogen p^o (mmHg) mean ± 2σ, min/max</p> </div>	<p>Displays statistics of the saturation pressures measured with nitrogen gas at liquid nitrogen temperatures. The mean, two-sigma, minimum, and maximum values display.</p> <p>The gauge is updated when a Po is logged with nitrogen as the adsorptive and a bath temperature of 77±2 K.</p>

Show Status

Unit [n] > Show Status

Use to show the current status for each port.

1:	Preliminary	Analysis					Termination
Sample:							
Stage	Last Point	p (mmHg)	p/p*	Q (cm ³ /g STP)	p* (mmHg)	Run Time	
Analysis	24 of 30	564.000000	0.917000000	37.0000	774.000	4:43	
Details:	<input type="text"/>						
2:	Preliminary	Analysis			Free Space	Termination	
Sample:							
Stage	Last Point	p (mmHg)	p/p*	Q (cm ³ /g STP)	p* (mmHg)	Run Time	
Analysis	24 of 30	564.000000	0.917000000	37.0000	774.000	4:43	
Details:	<input type="text"/>						
3:	Preliminary	Analysis				Termination	
Sample:							
Stage	Last Point	p (mmHg)	p/p*	Q (cm ³ /g STP)	p* (mmHg)	Run Time	
Analysis	29 of 30	569.000000	0.967000000	42.0000	779.000	5:33	
Details:	<input type="text"/>						

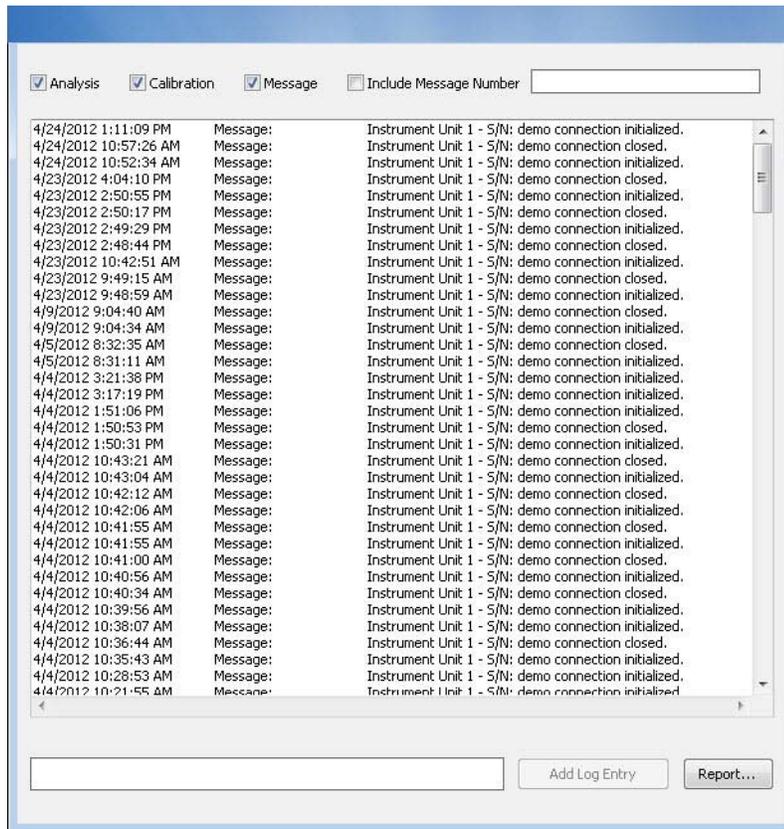
The yellow Post Analysis Free Space displays when applicable and if **Free Space After Analysis** is selected.

If there are multiple units attached to the computer. Select **Show Status** on each unit menu and have the status for all units displayed at one time.

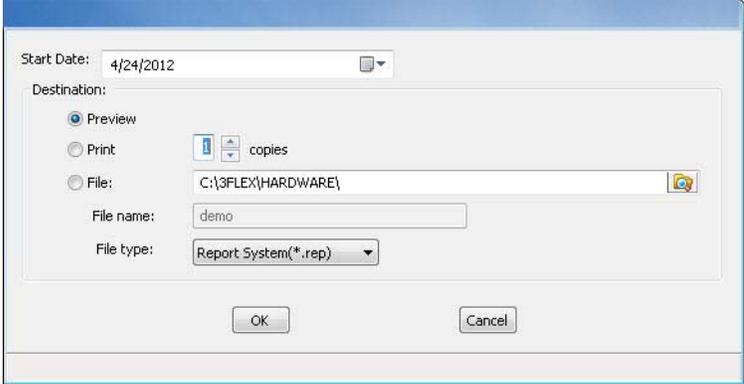
Show Instrument Log

Unit [n] > Show Instrument Log

Use to display a log of recent analyses, calibrations, errors or messages. This information is logged for a 7-day period for analyses and a 30-day period for messages and calibrations.



Field or Button	Description
<p><i>Analysis</i></p> <p><i>Calibration</i></p> <p><i>Message</i></p>	Select which logs to display.
<p><i>Include Message Number</i></p>	Use to generate a log report containing all the instances of one message number making it easier to look at the history of a particular quantity.
<p><i>Add Log Entry button</i></p>	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.

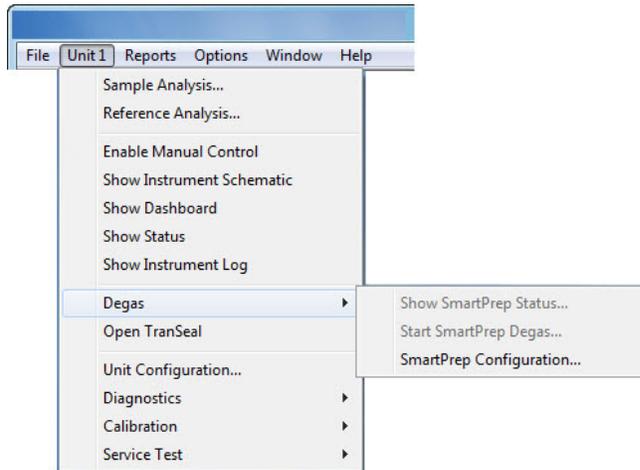
Field or Button	Description (<i>continued</i>)
Report button	<p>Click to display the Instrument Log Report Settings window to specify report output options.</p>  <ul style="list-style-type: none"> • Start Date - click to display a calendar to select the start date for the report. • Destination group box: <ul style="list-style-type: none"> Preview - sends the report to the screen. Click Print on the report screen to send the file to the printer. 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 - saves the report as a file. <ul style="list-style-type: none"> Click the Browse icon to the right of the text field to select the directory where the new report file will be stored. Enter the new file name in the File name text box. File Type - use to save the new file with a .TXT, .XLS or .REP file extension. This field is only enabled when File is selected. <ul style="list-style-type: none"> – .REP (Report system) - saves the report in a format that can be opened with any MicroActive program. – .TXT (ASCII text) - saves the report as a common machine language file. – .XLS (Spreadsheet file) - saves the report in a format that can be opened within a spreadsheet program.

Degas



If a SmartPrep is not connected to the analyzer, the menu options in this section are disabled. Refer to [Analysis Conditions Files](#), page 3-16 for in situ degassing.

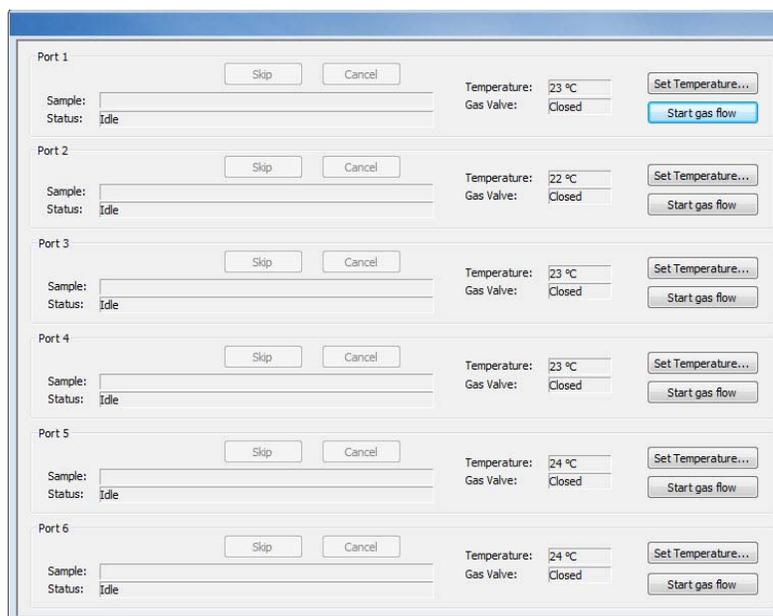
Unit > Degas



Show SmartPrep Status

Unit > Degas > Show SmartPrep Statu

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



Field or Button	Description
<i>Skip button</i>	Use to skip the degassing of the selected sample.
<i>Cancel button</i>	Use to cancel the degassing of the selected sample.
<i>Set Temperature button</i>	Use to set the temperature of the selected port. <div data-bbox="873 1348 1195 1562" data-label="Image"> </div>
<i>Stop Gas Flow button</i>	Stops the gas flow to the selected port.

Start SmartPrep Degas

Unit > Degas > Start SmartPrep Degas

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

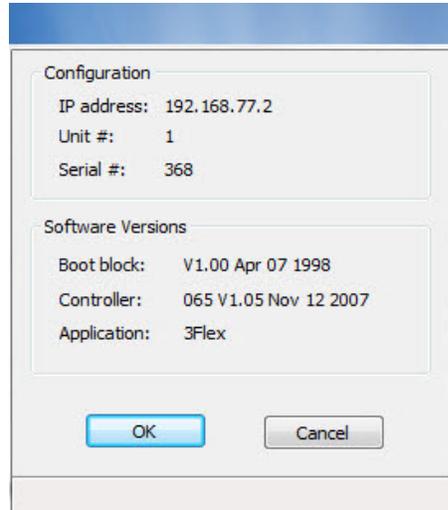
The screenshot shows a software window titled "Automatic Degas" with a blue header. It contains six rows, each representing a heating station. Each row has a "Sample:" label, a "Browse..." button, a "Degas conditions:" label, a dropdown menu currently set to "Degas Conditions", and a "Clear" button. At the bottom of the window, there is a "Start" button and a "Cancel" button. Below the buttons, a small text box reads: "After selecting the parameters and making sure the sample is properly installed on the degas port, press the start button to begin the automatic degas."

Field or Button	Description
<i>Browse button</i>	Use to locate a sample file to degas.
<i>Clear button</i>	Clears the entry of the selected sample.
<i>Start button</i>	Starts the degas process for all samples.
<i>Cancel button</i>	Cancel the degassing process for all samples.

SmartPrep Configuration

Unit [n] > Degas > SmartPrep Configuration

Displays the SmartPrep configuration and software versions.



Open TranSeal

Unit > Open TranSeal

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



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

Field or Button	Description
<i>Sample ports to open</i> checkboxes	Select the ports to open during analysis.
<i>Samples are NOT under vacuum</i> option	Select if samples are not under vacuum and specify the amount of backfill and adsorptive to be used prior to opening TranSeals.
<i>Samples ARE under vacuum</i> option	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 instrument damage may occur.
<i>Start</i>	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 Log is recorded in the Instrument Log with the port pressure before and after opening the TranSeal.
<i>Cancel</i>	Cancels the process.

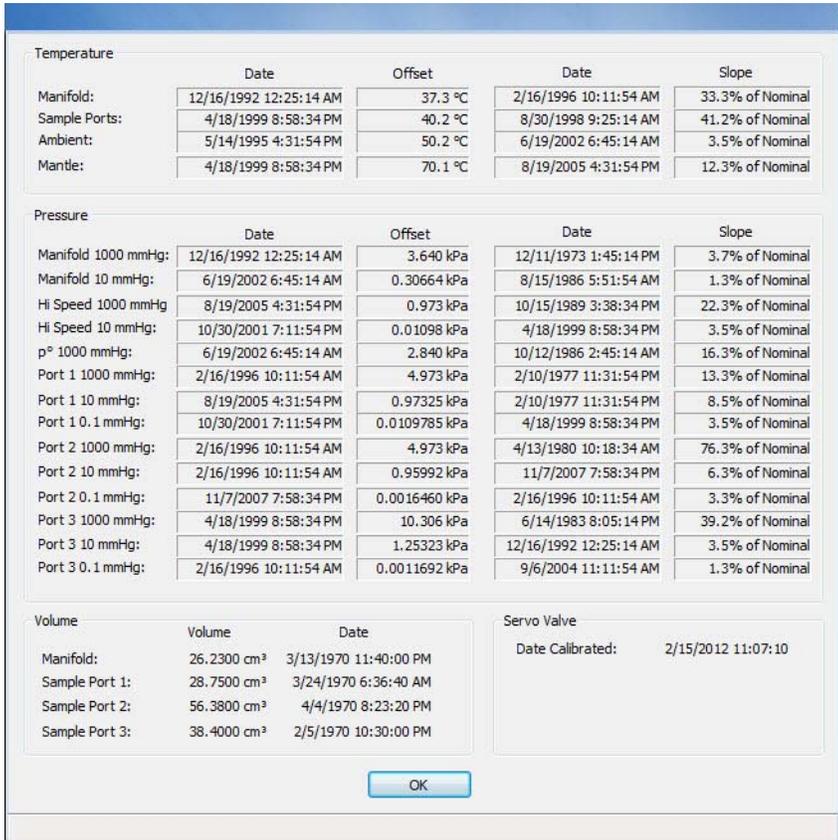
Unit Configuration

Unit [n] > Unit Configuration

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

If the auxiliary gas inlet manifold is present, a second column displays.

Field or Button	Description
<i>Configuration group box</i>	<p>Displays the IP address used by the analysis program and the serial number of the selected analyzer.</p> <ul style="list-style-type: none"> • 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. The parameters on this window cannot be edited.
<i>Software Versions group box</i>	Displays the software versions of the MIC BIOS, controller, and analysis program.
<i>Gas Selections group box</i>	In the text boxes, enter the mnemonics for the analysis gases attached to inlet valves.
<i>Options</i>	Displays options installed on the instrument.

Field or Button	Description (<i>continued</i>)
<i>Calibrations button</i>	<p>Displays calibration information for instrument components.</p>  <p>The screenshot shows a 'Calibrations' dialog box with three main sections: Temperature, Pressure, and Volume. Each section contains a table of calibration data with columns for the component name, Date, Offset, and Slope. The Temperature section includes Manifold, Sample Ports, Ambient, and Mantle. The Pressure section lists various ports and speeds (Manifold 1000 mmHg, Manifold 10 mmHg, Hi Speed 1000 mmHg, Hi Speed 10 mmHg, p^o 1000 mmHg, Port 1 1000 mmHg, Port 1 10 mmHg, Port 1 0.1 mmHg, Port 2 1000 mmHg, Port 2 10 mmHg, Port 2 0.1 mmHg, Port 3 1000 mmHg, Port 3 10 mmHg, Port 3 0.1 mmHg). The Volume section includes Manifold and Sample Port 1, 2, and 3. A 'Servo Valve' section shows the 'Date Calibrated' as 2/15/2012 11:07:10. An 'OK' button is at the bottom.</p>

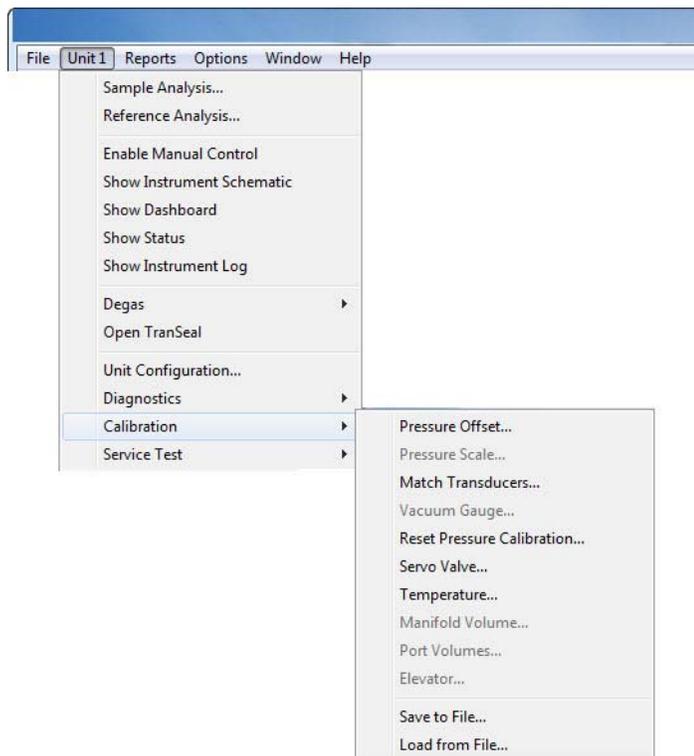
Diagnostics

Refer to **DIAGNOSTICS**, page 7-1.

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.



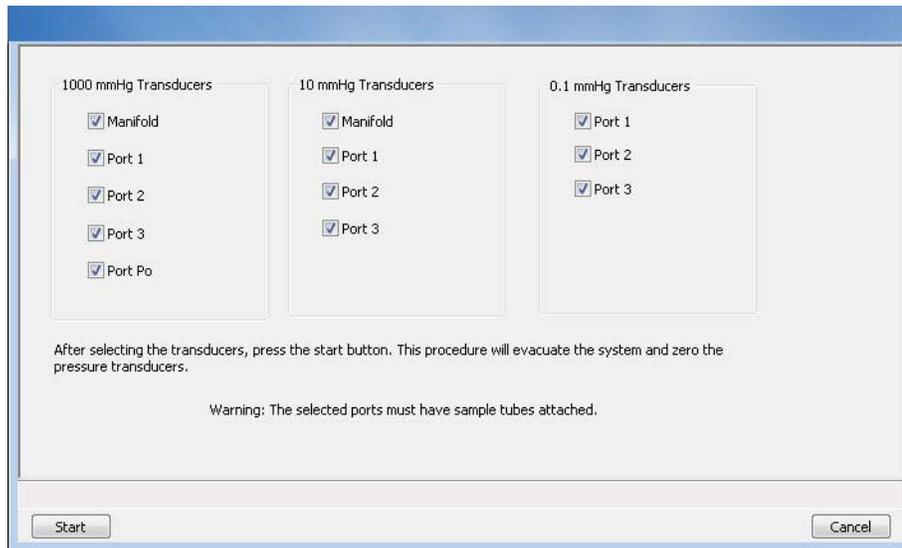
Information on the following items are not available in this manual. They are enabled when in Service Test Mode only.

- Pressure Scale
- Vacuum Gauge
- Manifold Volume
- Port Volumes
- Elevator

Pressure Offset

Unit [n] > Calibration > Pressure Offset

This procedure evacuates the system and zeros the selected pressure transducers. In order to perform this procedure, sample tubes must be attached to each port.

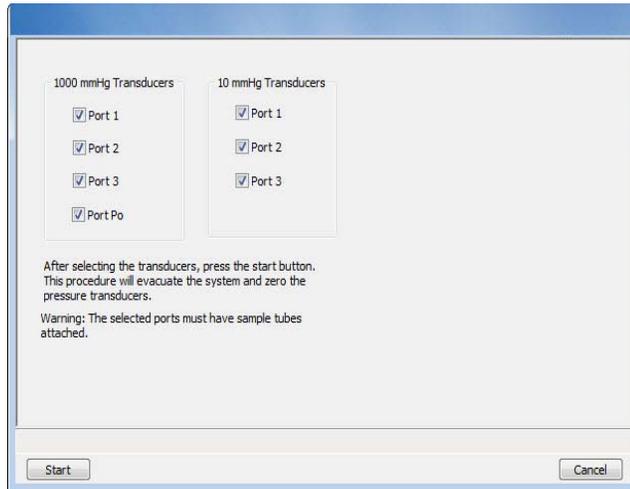


Field or Button	Description
<i>1000 mmHg Transducers checkboxes</i>	Select the manifold and/or ports.
<i>10 mmHg Transducers checkboxes</i>	Select the manifold and/or ports. Enabled only for the manifold and ports with 10 mmHg transducers present.
<i>0.1 mmHg Transducers checkboxes</i>	Select the ports. Enabled only for ports with 0.1 mmHg transducers present.
<i>Start button</i> <i>Cancel button</i>	Refer to Common Fields and Buttons - Unit Menu Options , page 4-2.

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. In order to perform this procedure, sample tubes must be attached to each port.

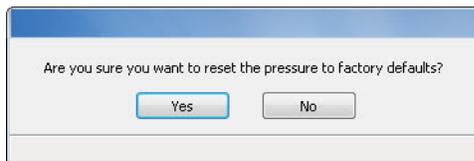


Field or Button	Description
<i>1000 mmHg Transducers checkboxes</i>	Select the ports.
<i>10 mmHg Transducers checkboxes</i>	Select the ports. Enabled only for ports with 10 mmHg transducers present.
<i>Start button</i> <i>Cancel button</i>	Refer to Common Fields and Buttons - Unit Menu Options , page 4-2.

Reset Pressure Calibration

Unit [n] > Calibration > Reset Pressure Calibration

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

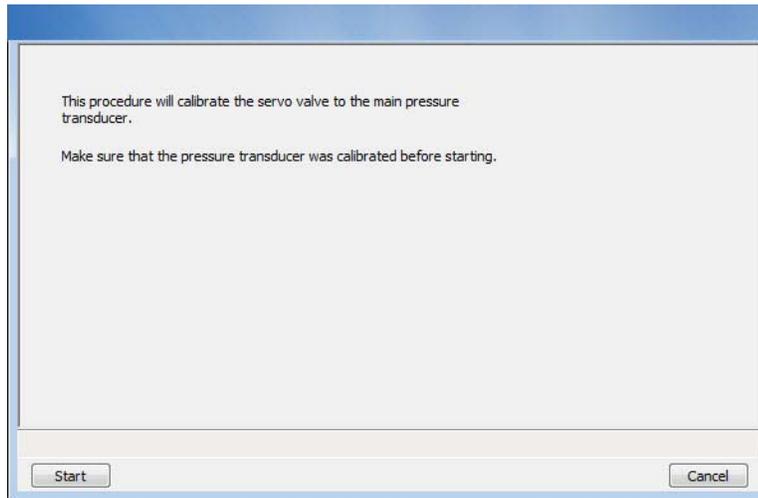


Click **Yes** to reset the pressure calibration.

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. Refer to [Servo Valve](#), page 8-24.



Field or Button	Description
<i>Start button</i>	Refer to Common Fields and Buttons - Unit Menu Options , page 4-2.
<i>Cancel button</i>	

Temperature

Unit [n] > Calibration > Temperature



Changing the calibration information will affect the performance of the instrument. Only qualified personnel should do this.

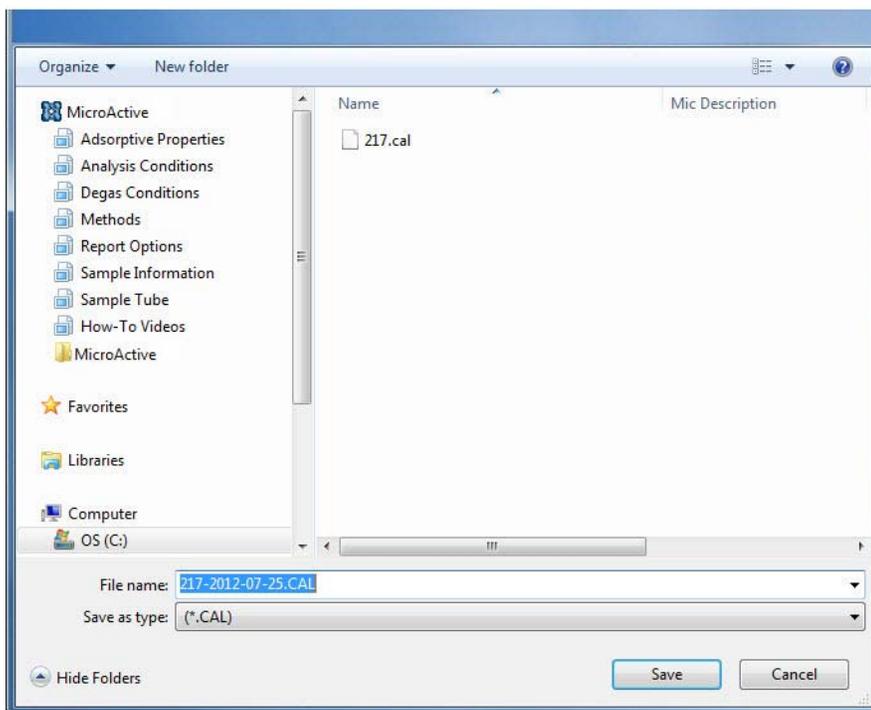
The allowable low calibration range is from 0.0 °C to 40.0 °C. The allowable high calibration range will be from 41.0 °C to 400.0 °C.

	Manifold	Ports	Ambient	Mantle
Low calibration:	Uncalibrated °C	Uncalibrated °C	Uncalibrated °C	Uncalibrated °C
	Calibrate...	Calibrate...	Calibrate...	Calibrate...
High calibration:	Uncalibrated °C	Uncalibrated °C	Uncalibrated °C	Uncalibrated °C
	Calibrate...	Calibrate...	Calibrate...	Calibrate...
	Done			

Save 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.



Field or Button	Description
<p>File name text box</p>	<p>The default file naming convention for calibration files can be used or the filename can be changed. The default file name is interpreted as:</p> <p style="text-align: center;">0217 - 2012-04-25.CAL</p> <div style="text-align: center;"> </div>

Load from File

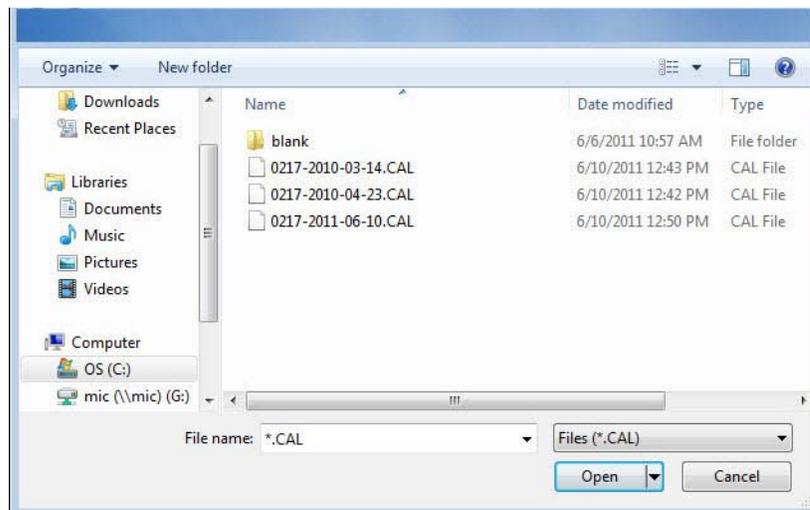
Unit [n] > Calibration > Load from File

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 instrument's performance.



Service Test

Unit [n] > Service Test

Use for service tests performed only with the assistance of a trained Micromeritics service representative. These tests provide the service representative with troubleshooting tools and readouts.

This option is enabled only when the analysis program is operating in Service Test mode. Refer to [Service Test Mode](#), page 6-4.

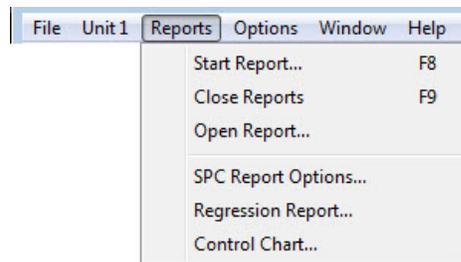
5. REPORTS MENU

Introduction

This chapter contains information specific to the Reports menu options used to customize and run reports. This chapter provides details of Reports menu options, commonly used functions and buttons, field-by-field descriptions, and sample reports.

Reports can be generated for data:

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



Common Fields and Buttons - Reports Menu Options

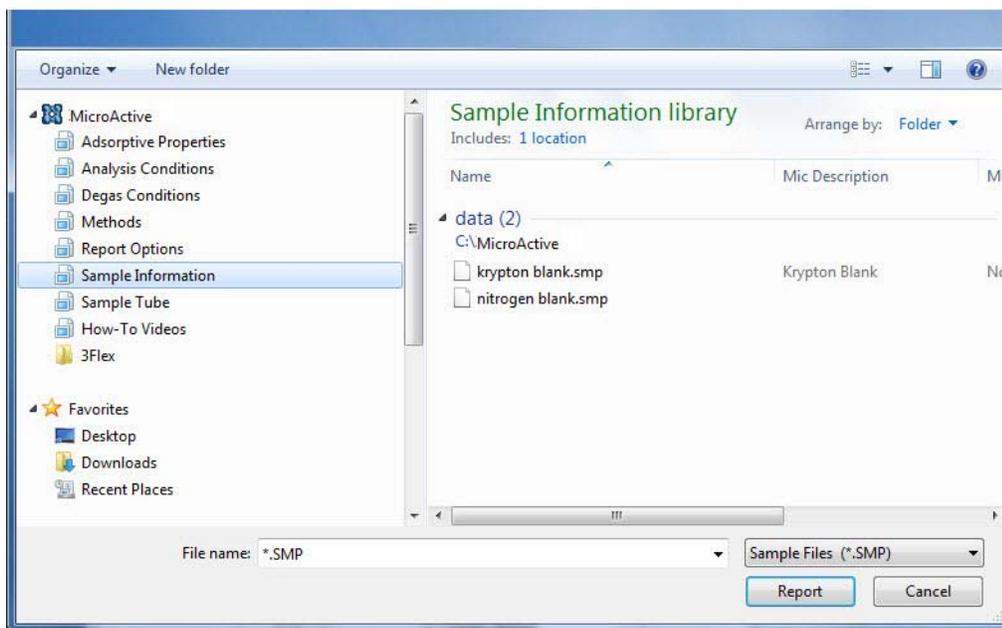
The following fields and buttons are common to many of the report files. References are made to these fields and buttons throughout this chapter.

Field or Button	Description
<i>Autoscale checkbox</i>	When enabled on report parameters screens, 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.
<i>Axis Range</i>	On report parameters screens, 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.
<i>Browse button</i>	Click to locate another file.
<i>Cancel button</i>	Closes the window and cancels any unsaved changes.

Field or Button	Description <i>(continued)</i>
<i>Destination group box</i>	<ul style="list-style-type: none"> • Preview - sends the file to the screen. Click Print on the report screen to send the file to the printer. • Print - sends the file to the default printer. • Copies - select the number of copies to print. This field is only enabled when Print is selected.
<i>Destination group box (continued)</i>	<ul style="list-style-type: none"> • File Type - use to save the new file with a .TXT, .XLS or .REP file extension. This field is only enabled when File is selected. <ul style="list-style-type: none"> – .REP (Report system) - saves the report in a format that can be opened with any MicroActive program. – .TXT (ASCII text) - saves the report as a common machine language file. – .XLS (Spreadsheet file) - saves the report in a format that can be opened within a spreadsheet program.
<i>File name text box</i>	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. Multiple files can be selected by holding down the Ctrl key on the keyboard while selecting multiple files.
<i>From / To text boxes</i>	Enter the From and To range for autoscaling the x- and/or y-axes.
<i>Name column</i>	Displays a list of files in the selected directory.
<i>OK button</i>	Click to save and close the active window.
<i>Open button</i>	Select a file from either the Name column or from the library. Click Open to open the file.
<i>Report button</i>	Click to generate the report.
<i>Save button</i>	Saves the active file.

Start Report

Reports > *Start Report* (or use the **F8** keyboard shortcut)



Use to generate a report on a sample analysis.

Close Reports

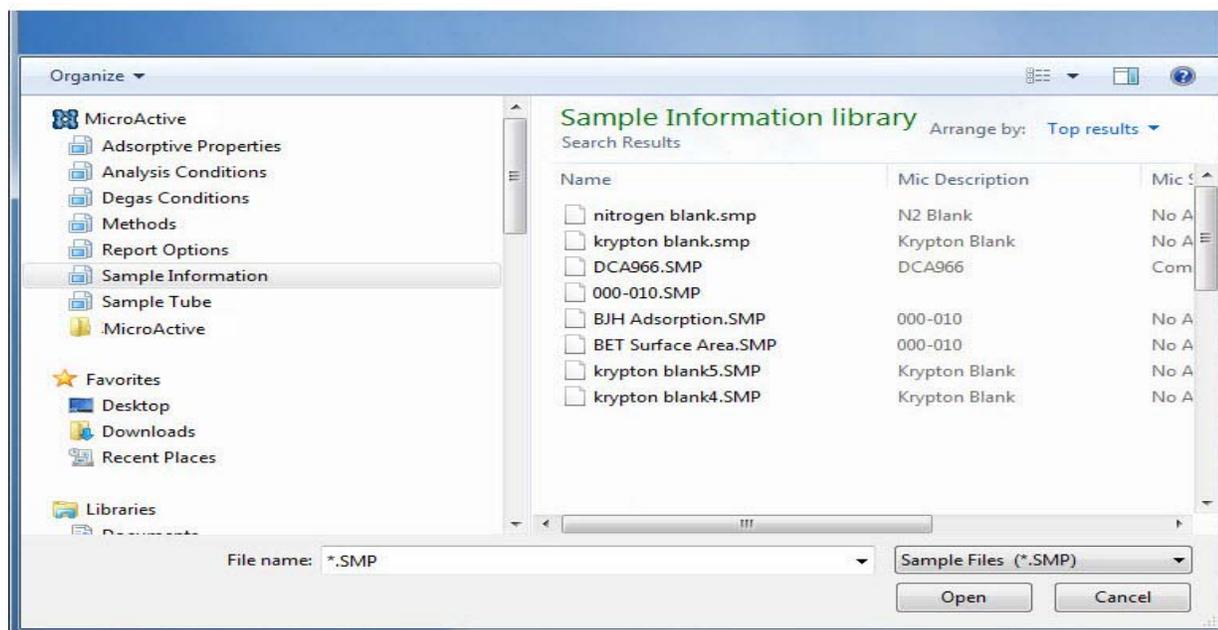
Reports > *Close Reports* (or use the **F9** keyboard shortcut)

Use to close all open report windows. This option is unavailable if reports are being generated.

Open Report

Reports > Open Report > [file]

Use to open a saved report.



SPC Report Options

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 appear as options on the *Reports > Regression Report* screen as selections in the dropdown boxes and are used in graph selection in *Reports > Control Chart*.

The dialog box contains the following options:

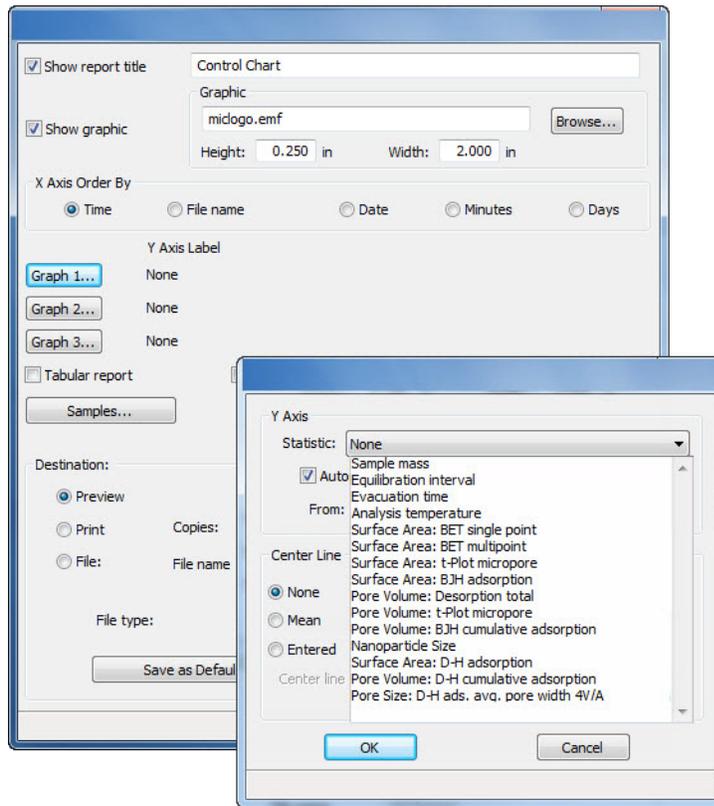
- Analysis Options:**
 - Sample mass
 - Equilibration interval
 - Evacuation time
 - Analysis temperature
 - Saturation pressure
 - Warm free space
 - Cold free space
 - Parameter 1
 - Parameter 2
 - Parameter 3
- Surface Area:**
 - Single-point BET
 - Multi-point BET
 - Langmuir
 - t-Plot micropore
 - t-Plot external
 - BJH adsorption
 - BJH desorption
 - D-H adsorption
 - D-H desorption
- Pore Volume:**
 - Adsorption total
 - Desorption total
 - t-Plot micropore
 - BJH cumulative adsorption
 - BJH cumulative desorption
 - D-H cumulative adsorption
 - D-H cumulative desorption
- Pore Size:**
 - BJH ads. avg. pore width 4V/A
 - BJH des. avg. pore width 4V/A
 - D-H ads. avg. pore width 4V/A
 - D-H des. avg. pore width 4V/A
 - Nanoparticle Size

If additional report options are required, click the **More** button.

The dialog box contains the following options:

- BET:**
 - C value
 - Monolayer volume
 - Correlation coefficient
- Dubinin-Astakhov:**
 - Micropore surface area
 - Limiting micropore volume
- Alpha-S:**
 - Slope
 - Y-Intercept
- Dubinin-Radushkevich:**
 - Micropore surface area
 - Monolayer capacity
- MP-Method:**
 - Cumulative surface area
 - Cumulative pore volume
 - Average pore width
- DFT Pore Size:**
 - Total pore area
 - Total pore volume
- Langmuir:**
 - B value
 - Monolayer volume
 - Correlation coefficient
- DFT Surface Area:**
 - Total surface area
- Horvath-Kawazoe:**
 - Maximum pore volume
 - Median pore width

The selected items also appear as options on the *Reports > Control Chart* screen. (Click the **Graph [n]** button, then click the **Statistic** dropdown arrow.)

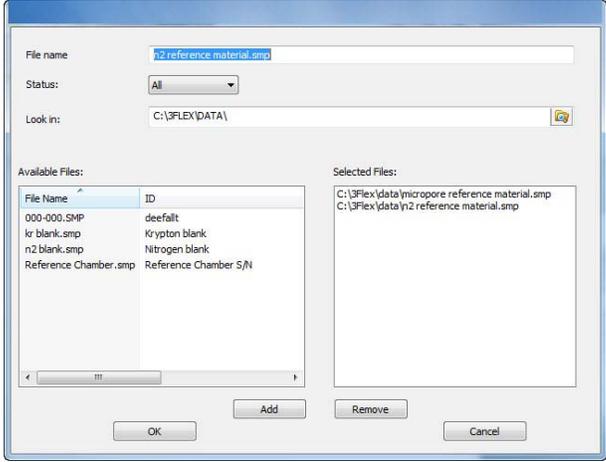


Regression Report

Reports > Regression Report

Use to generate an SPC (Statistical Process Control) 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.

Field or Button	Description
<i>Show report title text box</i>	Select and enter a report title to appear on the report header.
<i>Show graphic text box</i>	Use to show a graphic on the report header. Click the Browse button to locate the graphic. <ul style="list-style-type: none"> Height / Width - enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
<i>X- and Y-Axis Variable dropdown lists</i>	Use to designate the x- and y-axes variables. The variables in the dropdown 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.
<i>Axis Range text boxes</i>	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if Autoscale is selected.
<i>Autoscale checkboxes</i>	When enabled, allows the x- and y-axes to be scaled automatically.

Field or Button	Description (<i>continued</i>)
<i>Tabular report checkbox</i>	Use to generate a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
<i>Recalculate archived SPC results checkbox</i>	Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. Selecting this option lengthens the time required to generate the report.
<i>Label data checkbox</i>	Use to label the points on the plot to correspond with the values in the sample files.
<i>Samples button</i>	<p>Click to select sample files for report generation. Multiple files can be selected by holding down the Ctrl key on the keyboard while selecting the files.</p>  <ul style="list-style-type: none"> • 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 buttons - select a file in the Available Files list box and click Add to move the file to the Selected Files list box. Or select a file in the Selected Files list box and 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.
<i>Report button</i>	Click to view the report for the items selected.
<i>Save as Default button</i>	Click to save selected report options as default report settings.

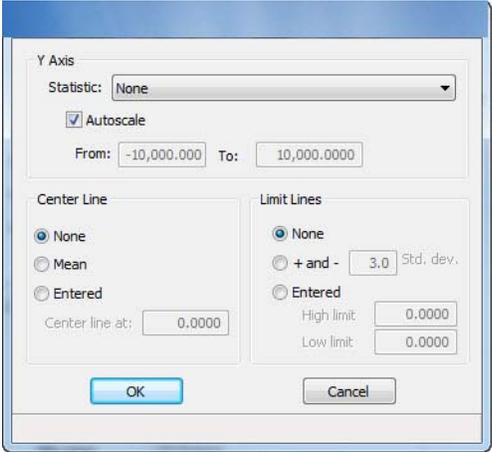
Field or Button	Description (<i>continued</i>)
<i>Browse button</i> <i>Cancel button</i> <i>Destination group box</i> <i>From / To text boxes</i> <i>OK button</i>	Refer to Common Fields and Buttons - Reports Menu Options , page 5-1 .

Control Chart

Reports > Control Chart

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

Field or Button	Description
<i>Show report title text box</i>	Select and enter a report title to appear on the report header.
<i>Show graphic text box</i>	Use to show a graphic on the report header. Click the Browse button to locate the graphic in either .BMP or .EMF format. <ul style="list-style-type: none"> • Height / Width - enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.

Field or Button	Description (<i>continued</i>)
<i>X Axis Order by group box</i>	<p>Select the order in which x-axis statistics are placed.</p> <ul style="list-style-type: none"> • Time - sorts by the time the files were analyzed. • File name - sorts in alphanumeric order. • Date - sorts by the date the files were analyzed. • Minutes - sorts by the minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. • Days - sorts by the number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.
<i>Graph [n] buttons</i>	<p>Click to define the y-axis of each graph.</p>  <ul style="list-style-type: none"> • Y-Axis group box - <ul style="list-style-type: none"> Statistic dropdown list - displays the SPC variables selected at <i>Reports > SPC Report Options</i> window. The selected variable will be plotted against time. This selection also becomes the y-axis label. Autoscale checkbox - 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 group box - 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.

Field or Button	Description (<i>continued</i>)
<i>Tabular report checkbox</i> <i>Samples button</i> <i>Save as Default button</i>	Refer to Regression Report , page 5-7 .
<i>Browse button</i> <i>Destination group box</i> <i>Cancel button</i> <i>Report button</i>	Refer to Common Fields and Buttons - Reports Menu Options , page 5-1 .

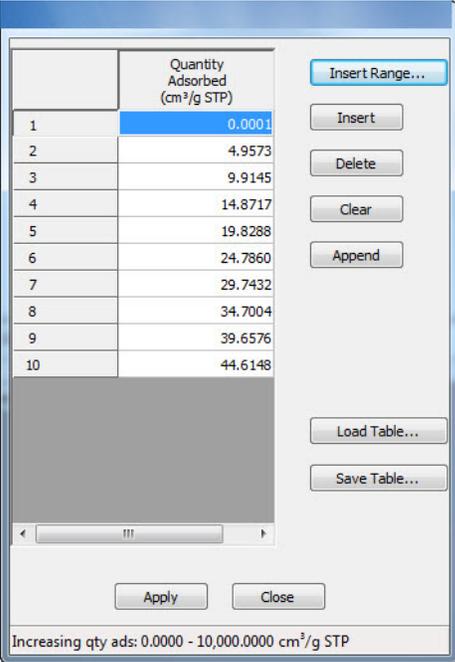
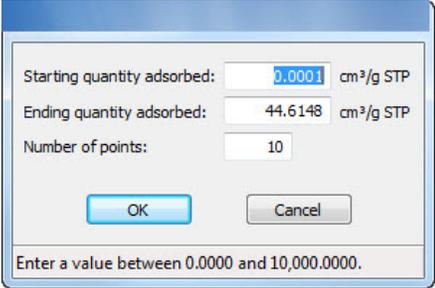
Heat of Adsorption Report

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.

Sample	Temp. (K)	0.00000
1 000-007	77.080	<input checked="" type="checkbox"/>
2 101-12 mm Tube N2.Silica-Alumi...	77.092	<input checked="" type="checkbox"/>
3 000-009	77.300	<input checked="" type="checkbox"/>

Field or Button	Description
<i>Table</i>	Contains files added by using the Add Samples button and provides the quantity adsorbed.
<i>Add Samples button</i>	Click to add a sample file to the table. <ol style="list-style-type: none"> 1. Click the Add Samples button. 2. Double-click the file in the Name column or select the file name and click Open.
<i>Remove Sample button</i>	Click to remove the selected sample from the list.

Field or Button	Description (<i>continued</i>)
<i>Clear Samples</i> button	Click to remove all entries from the table.
<i>Edit Quantities</i> button	<p>Use to specify the range of surface coverage to include in the report.</p>  <ul style="list-style-type: none"> • Insert Range button - click to specify the starting and ending quantities adsorbed and number of points to insert.  <ul style="list-style-type: none"> • Insert button - insert a row above the selected row. • Delete button - deletes the selected row. • Clear button - clears the entire table of all entries except one. • Append button - inserts one row at the end of the table. • Load Table button - click to import values from another file. • Save Table button - save the current table as a QNT file. • Apply button - click to apply all table changes. • Close button - click to close the table without saving changes.

Field or Button	Description (<i>continued</i>)
Tabular report checkbox	Refer to Regression Report , page 5-7.
Isostere plot checkbox	Select to generate a graph showing quantities of gas adsorbed versus the temperature.
Heat of adsorption plot checkbox	Select to generate the Heat of Adsorption data in a graphical format.
Open button	Click to select and open a Heat of Adsorption file.
Browse button Cancel button OK button Report button Save button	Refer to Common Fields and Buttons - Reports Menu Options , page 5-1.

Report Features and Shortcuts

The screenshot shows a software window titled "Micromeritics Gas Adsorption" with several report tabs at the top: Summary Report, Isotherm Tabular Report, Isotherm Linear Plot, Isotherm Log Plot, D-H Desorption Reports, and Horvath-Kawazoe Reports. The "Isotherm Tabular Report" tab is active, displaying a table of data. A "Reports List Box" is open on the right side of the window, listing the same report types. A "Tool Bar" is located at the bottom right of the window.

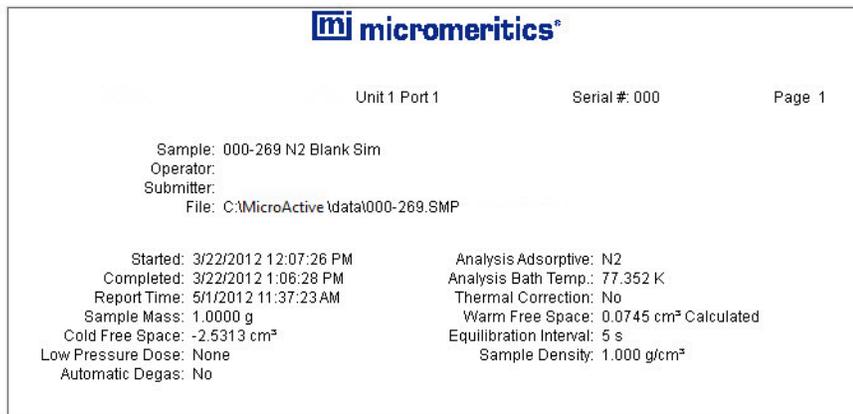
Labels with arrows point to the following features:

- Generated Report Tabs
- Header
- Data display (either graphical or text)
- Reports List Box
- Tool Bar

Relative Pressure (p/p [*])	Absolute Pressure (mmHg)	Quantity Adsorbed (cm ³ /g STP)	Elapsed Time (h:min)	Saturation Pressure (mmHg)
0.052299499	39.059860	6.5979	00:36	747.113342
0.072309507	54.012386	6.9404	00:45	746.849609
0.099184110	74.073235	7.3240	00:49	746.825623
0.124162079	92.734947	7.6476	00:51	746.886230
0.149525088	111.622742	7.9589	00:53	746.515137
0.174594599	130.398071	8.2596	00:55	746.862000
0.199964849	149.267670	8.5615	00:57	746.469543
0.225008249	167.975525	8.8633	00:58	746.530518
0.250152895	186.726059	9.1722	01:00	746.447754
0.275191746	205.496429	9.4863	01:02	746.739075
0.300574455	224.416641	9.8090	01:04	746.625793

Report Header

All reports contain a header displaying file statistics.



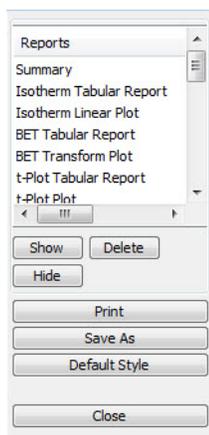
The screenshot shows a report header for a Micromeritics instrument. At the top center is the Micromeritics logo. Below it, the text 'Unit 1 Port 1' is on the left, 'Serial #: 000' is in the middle, and 'Page 1' is on the right. The main body of the header is divided into two columns of text. The left column contains sample and operator information, while the right column contains analysis parameters and results.

micromeritics®		
Unit 1 Port 1	Serial #: 000	Page 1
Sample: 000-269 N2 Blank Sim		
Operator:		
Submitter:		
File: C:\MicroActive\data\000-269.SMP		
Started: 3/22/2012 12:07:26 PM	Analysis Adsorptive: N2	
Completed: 3/22/2012 1:06:28 PM	Analysis Bath Temp.: 77.352 K	
Report Time: 5/1/2012 11:37:23 AM	Thermal Correction: No	
Sample Mass: 1.0000 g	Warm Free Space: 0.0745 cm ³ Calculated	
Cold Free Space: -2.5313 cm ³	Equilibration Interval: 5 s	
Low Pressure Dose: None	Sample Density: 1.000 g/cm ³	
Automatic Degas: No		

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

- Tabular and graphical reports contain sample and instrument 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.

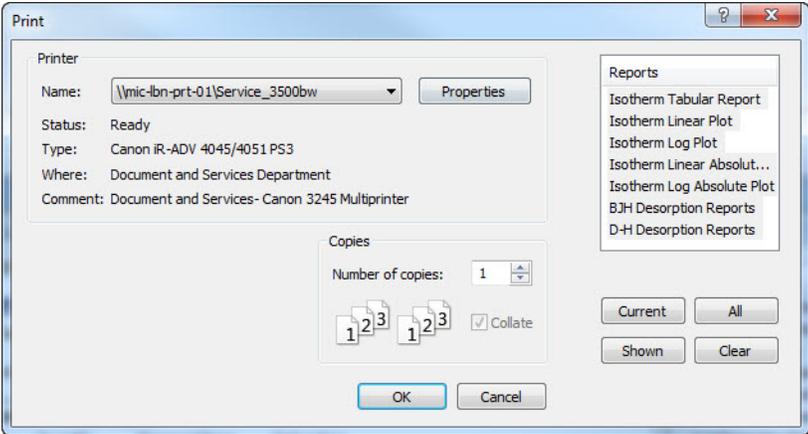
Report Tool Bar



The **Report** window has a tool bar and selectable tabs across the top of the report header. To view a specific report, select its tab or select the report in the Reports list box and click **Show**.

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

Field or Button	Description
<i>Reports list box</i>	Contains a list of all generated reports. The same reports display as tabs across the top of the report header unless the report has been hidden using the Hide button.
<i>Show button</i>	Jumps to the selected report in the Reports list box (or select the report tab to show the report). If the report tab has been hidden using the Hide button, click Show to display the report and tab.
<i>Delete button</i>	Deletes the selected report. Deleted reports will have to be regenerated if deleted in error.
<i>Hide button</i>	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the Reports list box. To redisplay the tab, select the report in the Reports list box and click Show .

Field or Button	Description (<i>continued</i>)
Print button	<p>Displays the Print window for report output.</p>  <ul style="list-style-type: none"> • Name dropdown list and Properties button - select the printer and click the Properties button to change printer setup, etc. • Copies group box - select the number of copies and collate option. • Current button - selects the active report (or selected tab). • All button - selects all reports in the Reports list box. • Shown button - selects only the reports not hidden. • Clear button - clears all selections. • OK button - prints the selected report to the printer indicated. • Cancel button - closes the Print window.
Save button	Saves all reports of the active file using the sample file name with a .REP file extension.
Save As button	<p>Saves all selected reports to the indicated file format:</p> <ul style="list-style-type: none"> • .REP (Report system) - saves the report in a format that can be opened with any MicroActive program. • .TXT (ASCII text) - saves the report as a common machine language file. • .XLS (Spreadsheet file) - saves the report in a format that can be opened within a spreadsheet program.

Field or Button	Description (<i>continued</i>)
<p>Default Style button</p>	<p>Click to specify default report parameters for fonts and curve properties.</p> <div data-bbox="659 359 1338 772" data-label="Image"> </div> <ul style="list-style-type: none"> • Font group box - <p>Font Type list box - allows font type and attributes to be edited for the selected item. Select an item in the list, click Edit, and select from various font options. Click OK when done.</p> <div data-bbox="717 1014 1289 1465" data-label="Image"> </div>

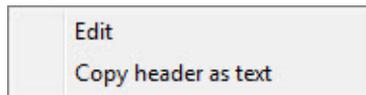
Field or Button	Description (<i>continued</i>)
<i>Default Style button (continued)</i>	<ul style="list-style-type: none"> • Curve group box - <ul style="list-style-type: none"> Thickness text box - enter a thickness number for the curve. Histogram Fill dropdown list - select a histogram fill option from the list. • Graph border line thickness text box - enter a thickness number for the graph border. • Load button - click to load the last saved default settings. • Save button - click to save the changes as the new default settings. • Close button - click to close the window and save the changes for the current report.

Report Shortcut Menus

Shortcut menus are accessed by right-clicking on the report header or the report body displayed on the screen.

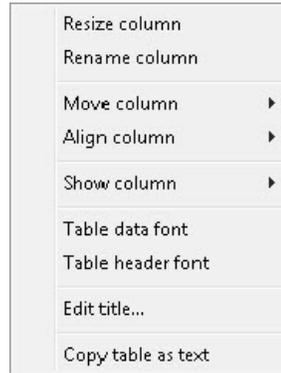
Report Header Shortcuts

Display header shortcuts by right-clicking in the report header.



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

Tabular Reports Shortcuts

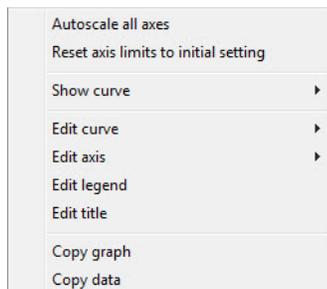


Display tabular report shortcuts by right-clicking in the body of the tabular report. Column shortcuts require right-clicking on the column to be modified.

Option	Description
<i>Resize column</i>	Right-click on the column to be resized. Select Resize Column on the shortcut menu and enter the new column width in inches.
<i>Rename column</i>	Right-click on the column to be renamed. Select Rename Column on the shortcut menu and enter the new column name.
<i>Move column</i>	Right-click on the column to be moved. Select Move Column on the shortcut menu and select Left or Right for the move.
<i>Align column</i>	Right-click on the column to be aligned. Select Align Column on the shortcut menu and select Left , Right or Center .
<i>Show column</i>	Displays a list of all columns. Click a column to add a checkmark and show the column or remove the checkmark to hide the column.
<i>Table data font</i>	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, select the Use default font checkbox.
<i>Table header font</i>	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 header. To return to the default fonts, select the Use default font checkbox.
<i>Edit title</i>	Use to edit the report title and/or title font attributes.

Option	Description (<i>continued</i>)
<i>Copy table as text</i>	Use to copy the report contents to the clipboard as tab delimited text. It can then be pasted into another document.

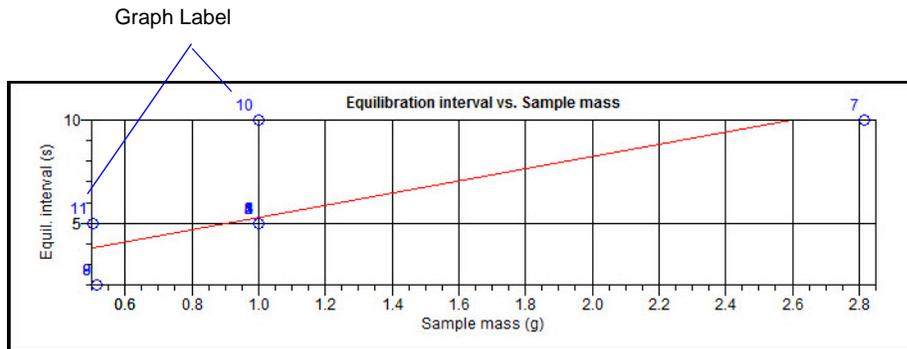
Graph Shortcuts

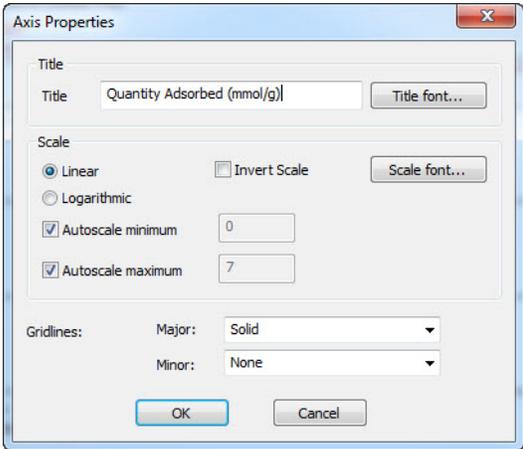


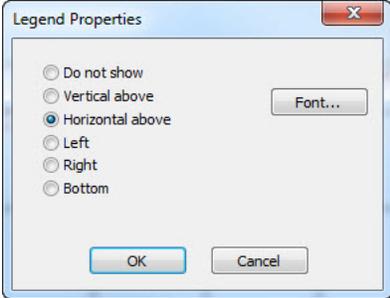
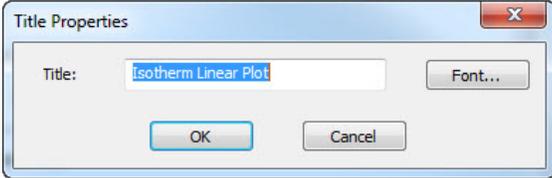
Display graph report shortcuts by right-clicking in the body of the graph report.

Option	Description
<i>Autoscale all axes</i>	Returns the report to full view after using the zoom feature.
<i>Reset axis limits to initial setting</i>	Removes the cross-hair and returns the graph back to the initial setting.
<i>Show curve</i>	Displays a list of all curves. Click a column to add a checkmark and show the curve or remove the checkmark to hide the curve.
<i>Edit curve</i>	Use to edit selected curve properties. <div data-bbox="649 1302 1201 1785" data-label="Image"> </div> <ul style="list-style-type: none"> • Title text box - use to change the title of the selected curve.

Option	Description (<i>continued</i>)
<i>Edit curve (continued)</i>	<ul style="list-style-type: none"> • Style dropdown list - use to select another style for the collected data curve. • Curve group box - options are disabled if Histogram is selected in the Style dropdown list. Use to change the interpolation, point style and pen style for the selected curve. <p>Color button - click to change the curve color.</p> <p>Use default thickness checkbox - select to use the default curve thickness. Deselect the checkbox and enter a new thickness number in the Thickness text box.</p> <ul style="list-style-type: none"> • Histogram group box - enabled only if Histogram is selected in the Style dropdown list. Use to specify the type of fill, fill color and label position for the selected curve. <p>Label dropdown list - select where the graph point labels will display (left, right, center, etc.) on the SPC report.</p>



Option	Description (<i>continued</i>)
<i>Edit axis</i>	<p>Use to edit the selected axis properties.</p>  <ul style="list-style-type: none"> • Title group box - use to edit the selected axis label. <ul style="list-style-type: none"> Title text box - use to modify the label of the selected axis. Title font button - use to modify the font for the selected axis label. Deselect the Use default font to enable font options. Select the font attributes and click OK. • Scale group box - use to change the graph display. <ul style="list-style-type: none"> Linear / Logarithmic - select the option to scale the graph as linear or logarithmic. Autoscale minimum / maximum - select the Autoscale checkbox to enable the option. To manually specify minimum / maximum, deselect the Autoscale checkbox and enter the new amount in the text box. Invert scale checkbox - use to invert the scale. Scale font button - use to modify the font for the scale label. Deselect the Use default font to enable font options. Select the font attributes and click OK. Gridlines Major / Minor dropdown lists - use to change how to display major / minor gridlines.

Option	Description (<i>continued</i>)
<i>Edit legend</i>	<p>Use to change the legend location and font. Click Font to modify legend fonts. Deselect the Use default font to enable font options.</p> 
<i>Edit title</i>	<p>Use to change the graph title and font. Deselect the Use default font to enable font options.</p> 
<i>Copy Graph</i>	<p>Copies the graph to the clipboard. It can then be pasted into other applications.</p>
<i>Copy Data</i>	<p>Copies the report data to the clipboard. It can then be pasted into other applications as tab-delimited columns of text.</p>

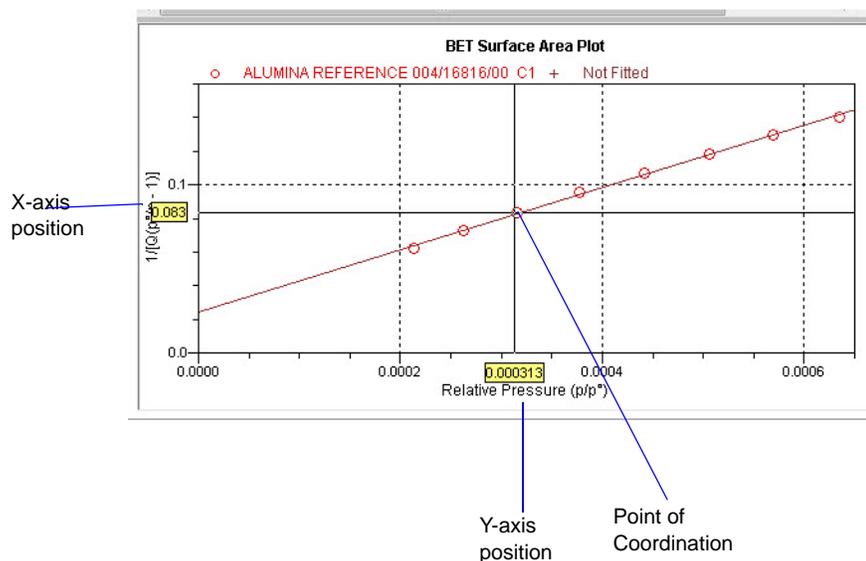
Other On-Screen Features

Zoom Feature

Use the zoom feature to closer examine graph details. To use this feature:

1. Open the graph.
2. Hold down the left mouse button and drag the cursor 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 and select **Autoscale all axes** or **Reset all axes to initial setting** on the shortcut menu.

Axis Cross-Hair



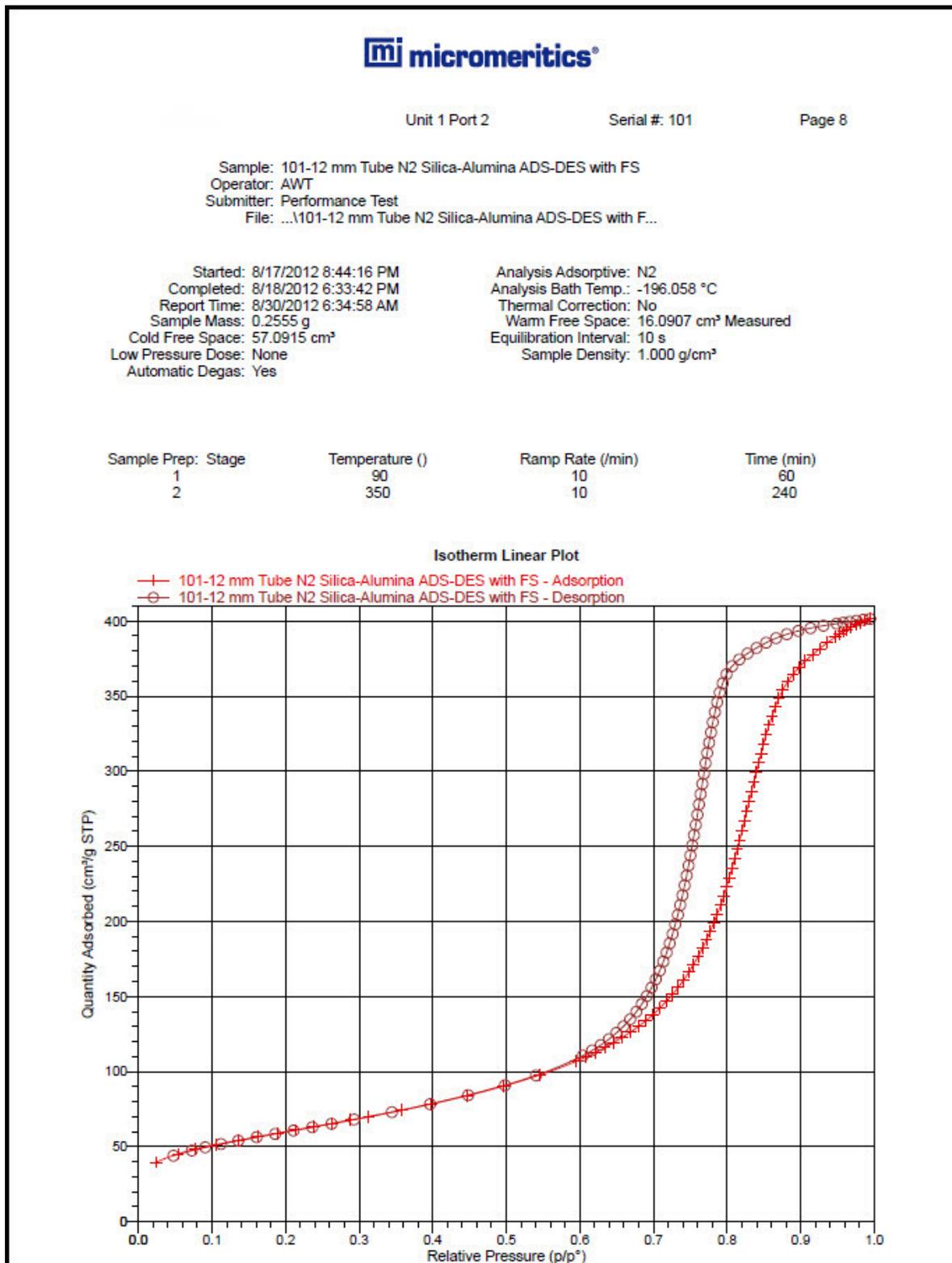
The cross-hair feature displays axis coordinates. To use this feature:

1. Click the left mouse button on the graph to view the cross-hair coordinates.
2. To remove the cross-hair, right-click in the graph area and select **Autoscale all axes** or **Reset all axes to initial setting** from the shortcut menu.

Report Examples

This section of the manual contains samples of some of the available reports. Most of the reports can be customized.

Isotherm Linear Plot



BET Surface Area Report



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 cm ³ Measured
Cold Free Space: 57.0915 cm ³	Equilibration Interval: 10 s
Low Pressure Dose: None	Sample Density: 1.000 g/cm ³
Automatic Degas: Yes	

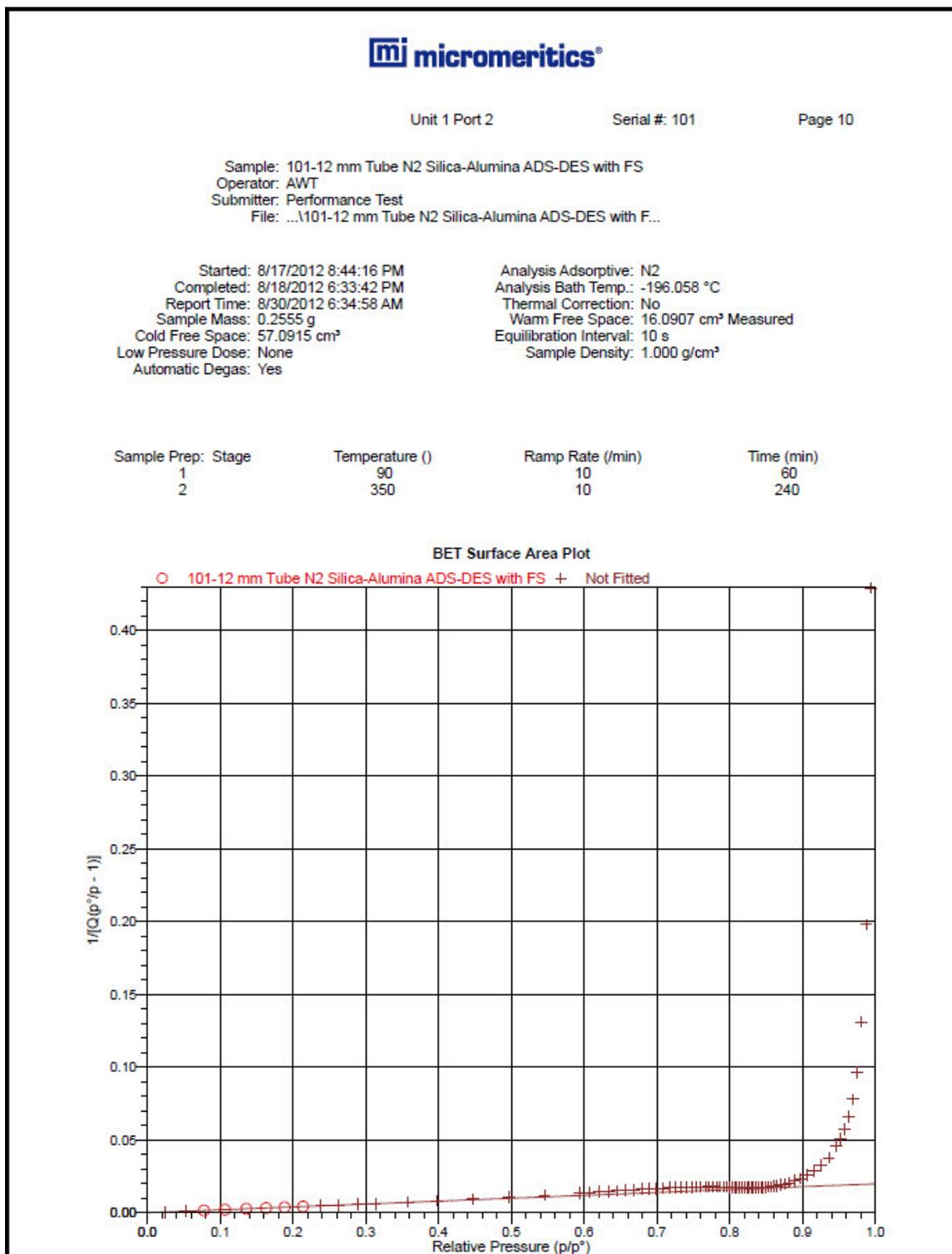
Sample Prep: Stage	Temperature (°)	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240

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 ⁰)	Quantity Adsorbed (cm ³ /g STP)	1/[Q(p ⁰ /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



t-Plot Report



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 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

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

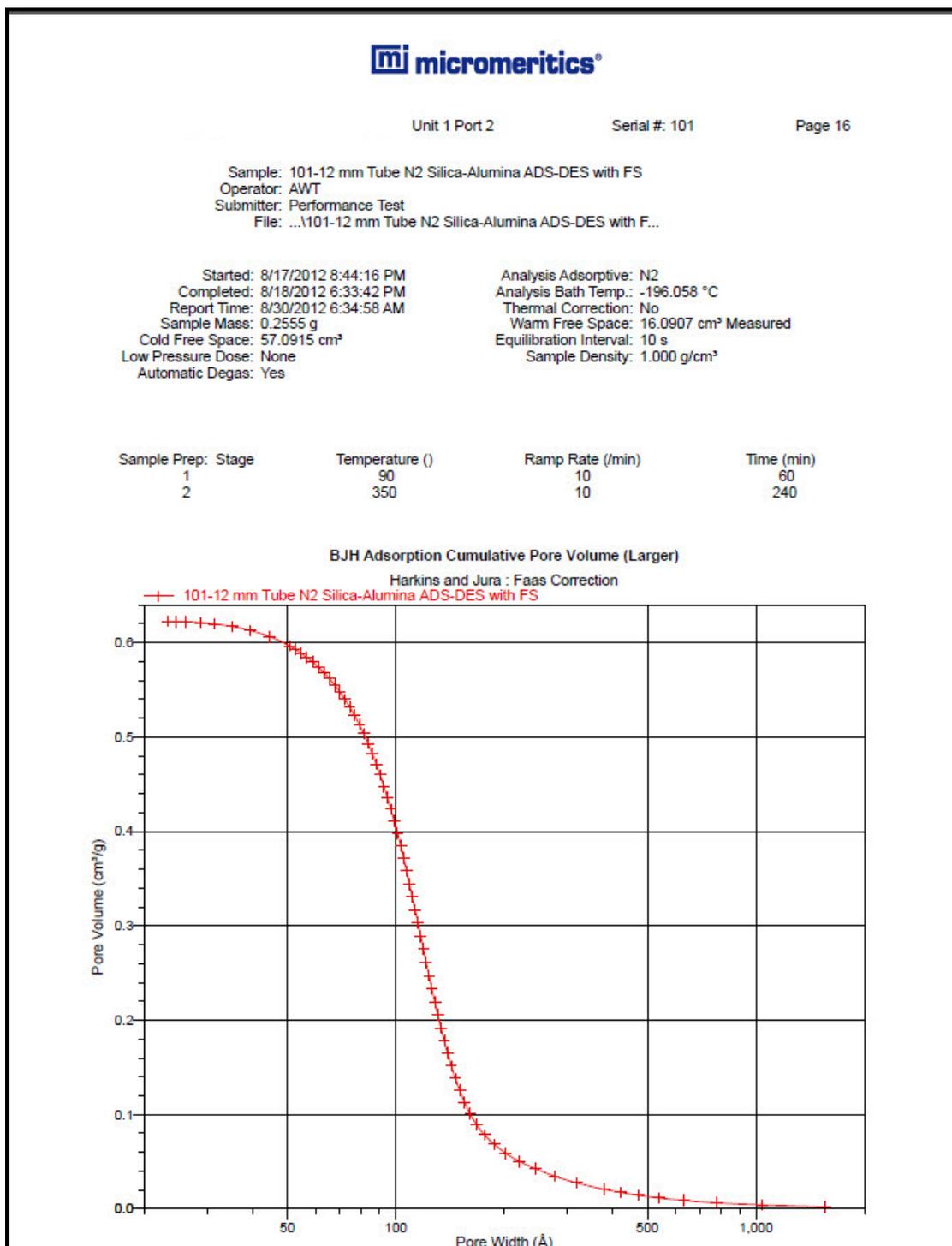
Thickness Curve

$$t = [13.99 / (0.034 - \log(p/p^*))] ^ 0.5$$

t-Plot Report - Data

Relative Pressure (p/p [*])	Statistical Thickness (Å)	Quantity Adsorbed (cm ³ /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	

BJH Adsorption: Cumulative Pore Volume



BJH Desorption: Cumulative Pore Volume



Unit 1 Port 2

Serial #: 101

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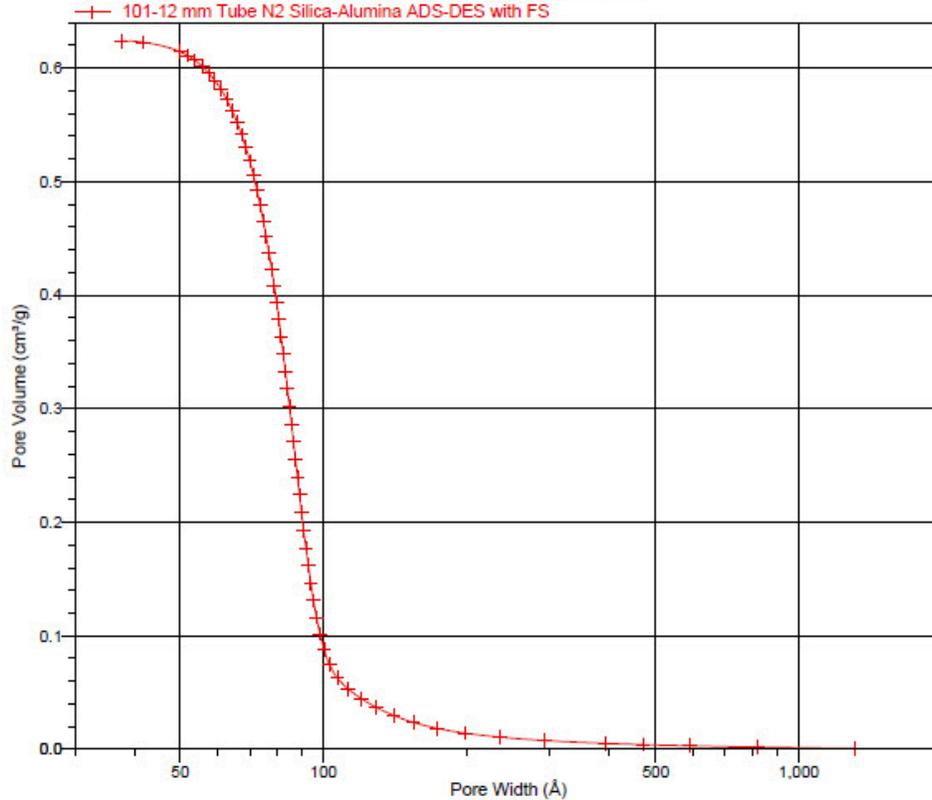
Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
 Operator: AWT
 Submitter: Performance Test
 File: ...101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

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 cm ³ Measured
Cold Free Space: 57.0915 cm ³	Equilibration Interval: 10 s
Low Pressure Dose: None	Sample Density: 1.000 g/cm ³
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



6. OPTIONS MENU

Introduction

This chapter contains information specific to the Options menu selections used to configure the system by setting defaults for sample and parameter files.

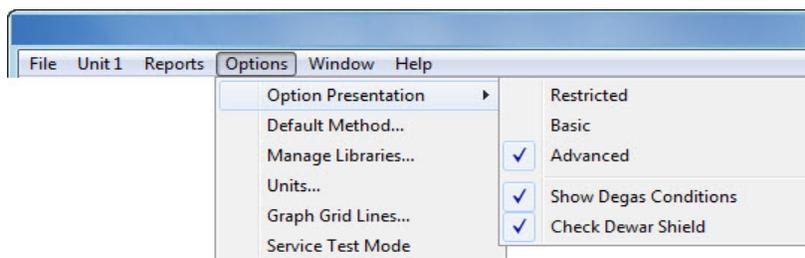
This chapter contains information on:

- changing the default presentation format - Restricted, Basic, or Advanced.
- specifying default parameters for sample information files and report option files.
- specifying how units appear on application windows and reports.

Option Presentation

Options > Option Presentation

Use to change the default editing format of sample files - Restricted, Basic, or Advanced. Each format type displays sample information and options differently. For descriptions of the presentation types, refer to [Defining Sample Information Files](#), page 2-6. When using **Restricted** format, a password is required to change to **Advanced** format.



- **Show Degas Conditions** - when enabled, displays a Degas Conditions tab in Advanced format.
- **Check Dewar Shield** - when enabled, checks to ensure the Dewar shield is in place prior to starting an analysis. An entry is made in the instrument log regardless of operator choice. If this option is selected and the Dewar shield is not in place prior to starting an analysis, a warning message displays. on the instrument schematic screen.

**Dewar Shield
Removed**

Default Method

Options > Default Method

Use to specify default parameters for sample information files.

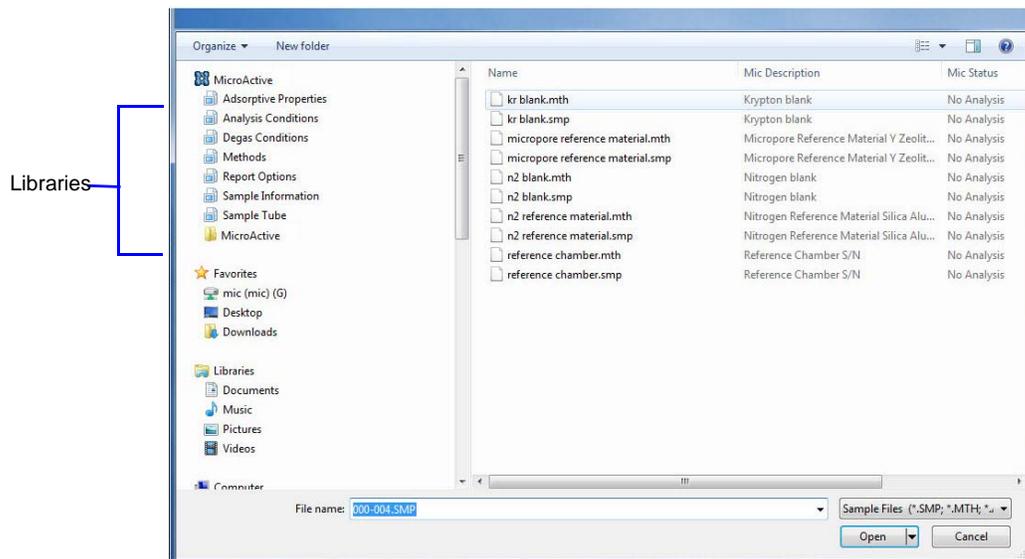
Manage Libraries

Options > Manage Libraries

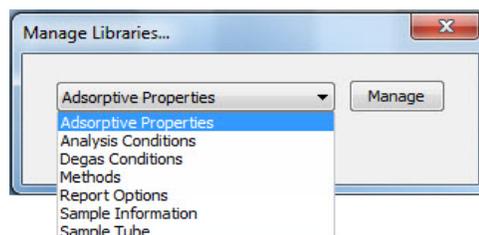


This feature is available only for Windows 7 operating system. It is greyed out if running Vista or Windows XP.

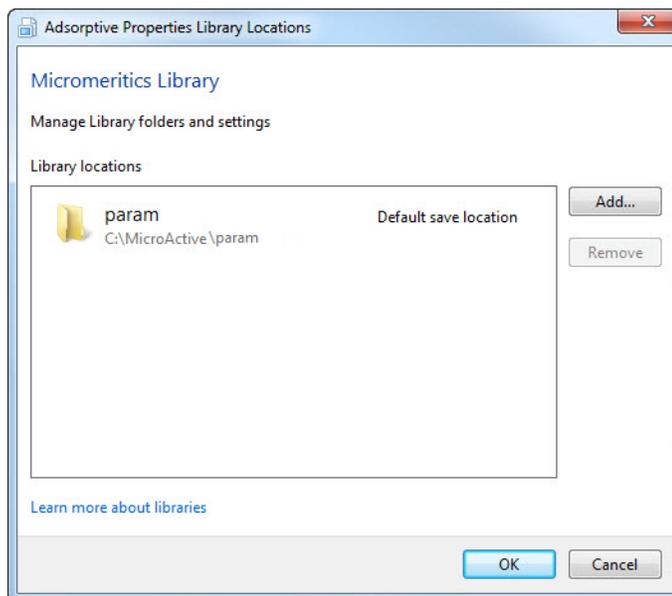
The library allows access to sample and parameter files. The library gathers files that are stored in several locations. Folders can be added or removed from each library.



1. To add or remove folders from a library, go to *Options > Manage Libraries*. Select the library to modify then click the **Manage** button.



- To add a folder, click **Add** to browse and locate a folder. Select the folder and click the **Include Folder** button.

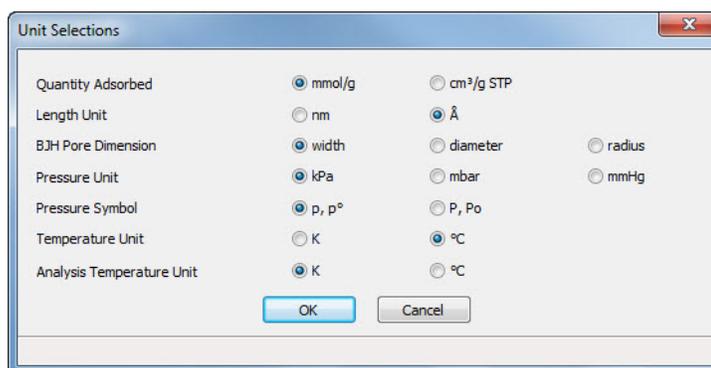


- To remove a folder, select the folder to be removed from the **Library locations** box and click the **Remove** button.
- Click **OK** when done.

Units

Options > Units

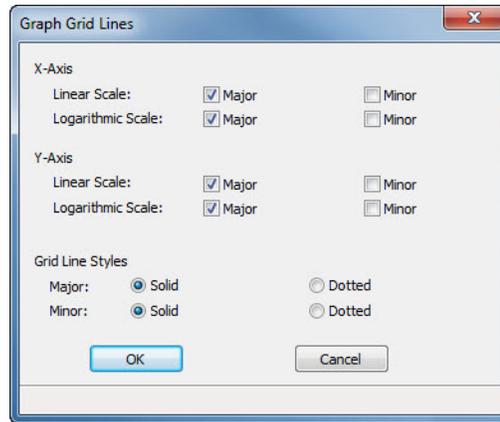
Use to specify how data should appear on the application windows and reports. This menu option is not available if using Restricted format.



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 format.



Field or Button	Description
<i>X-Axis / Y-Axis options</i>	Select major and/or minor lines to display in reports for the logarithmic and linear scales. To remove the gridlines, deselect these options.
<i>Grid Line Styles options</i>	Select if the major and/or minor grid lines should appear as solid or dotted lines.

Service Test Mode

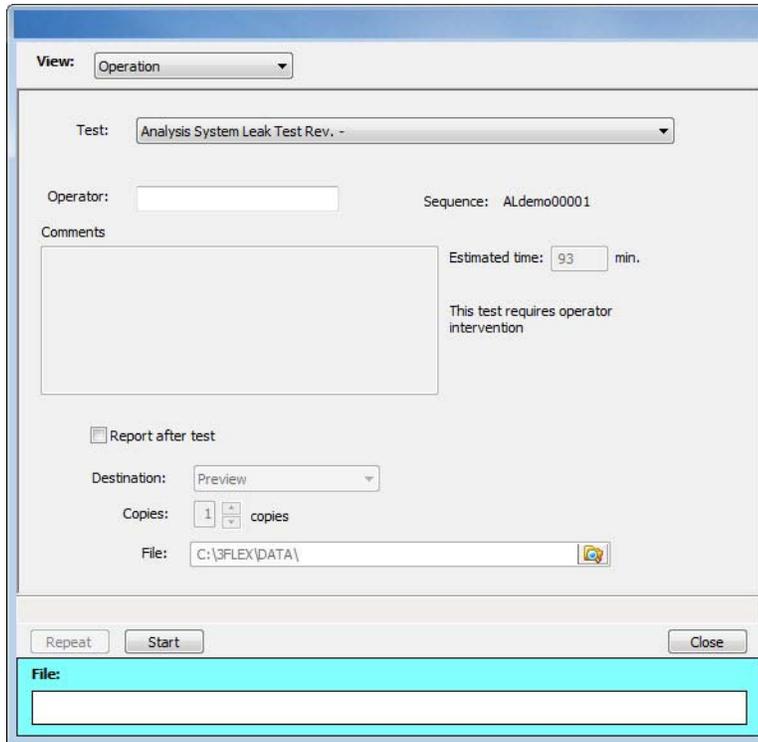
Options > Service Test Mode

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. After Service Test Mode has been enabled, the tests are accessible from the Unit menu (*Unit > Service Test*).

Start Diagnostic Test

Unit [n] > Diagnostics > Start Diagnostic Test

Provides a method to start a diagnostic test immediately.



Field or Button	Description
<i>View dropdown list</i>	<ul style="list-style-type: none"> • Operation - use to display the current mode of operation. • Instrument Log - use to display recent analyses, calibrations, errors or messages. Refer to Show Instrument Log, page 6-14. • Instrument Schematic - use to display a schematic of the analyzer system. Show Instrument Schematic, page 6-12.
<i>Test dropdown list</i>	<p>Select the diagnostic test to be performed.</p> <ul style="list-style-type: none"> • Degas Heat Test Rev. [n] • Clean and Verify Gas Line # Test Rev. [n] • Manifold Heat Test Rev. [n] • Ports Leak Test Rev. [n] • System Operation Verification Rev. C
<i>Operator</i>	Enter information to identify the person running the service test.

Field or Button	Description (<i>continued</i>)
<i>Comments text box</i>	Displays comments from the selected diagnostic test.
<i>Estimated time (min.)</i>	Approximate time for test completion.
<i>Report after test</i>	Select to automatically generate reports to the selected destination when the test is complete.
<i>Repeat button</i>	Repeats the selected diagnostic test.
<i>Next button</i>	Starts the next test.
<i>Start button</i>	Starts the selected diagnostic test.
<i>Close button</i>	Closes the window.

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 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.



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



The Po 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.

Test Frequency

Displays options for scheduling unattended diagnostic tests.

File Identification	Est. Time (min.)	Intervention Re
Clean and Leak Test Re...	813	✓
Degas Heat Test Rev. -	60	X
Clean and Verify Gas Li...	60	X

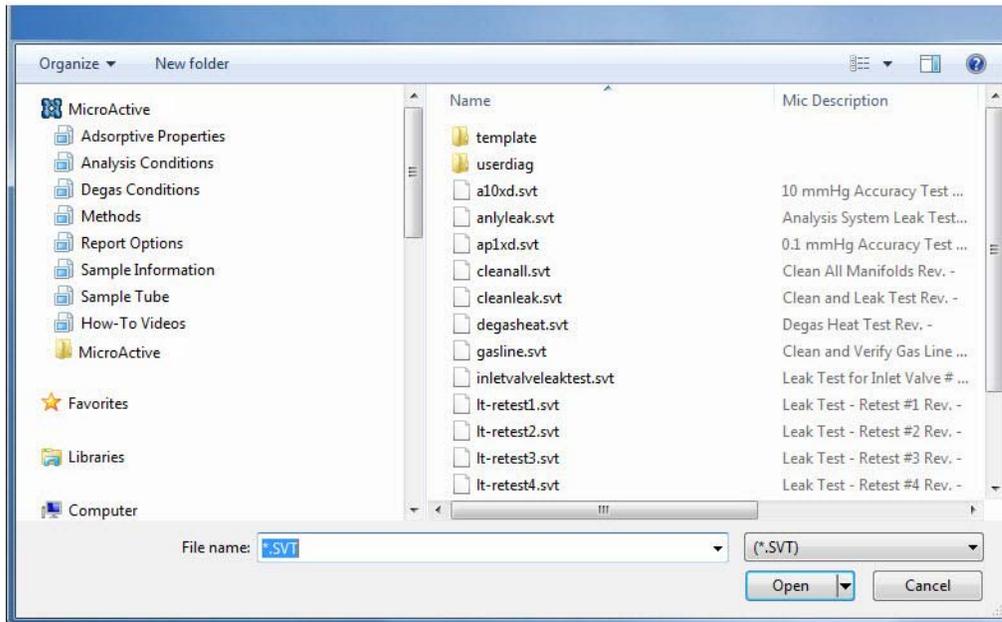
Field or Button	Description
<i>Test Frequency option</i>	Select how often the test is to run unattended.

Field or Button	Description (continued)
<i>Start Test Sequence if Instrument is Idle Any Time Between</i>	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.
<i>Available Tests dropdown list</i>	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.
<i>Test Sequence</i>	<p>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.</p> <p>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.</p> <p>Select a row and click Delete to remove the test from the sequence. Select Clear to remove all entries from the Test Sequence box.</p>
<i>Intervention Required</i>	Check this option if any test requiring operator intervention should be skipped if the operator does not respond within the specified time.
<i>Cancel button</i> <i>OK button</i>	Refer to Common Fields and Buttons - Unit Menu Options , page 4-2.

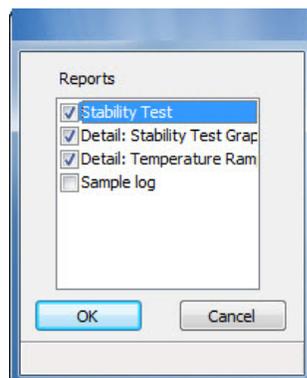
Diagnostic Test Report

Unit [n] > Diagnostics > Diagnostic Test Report

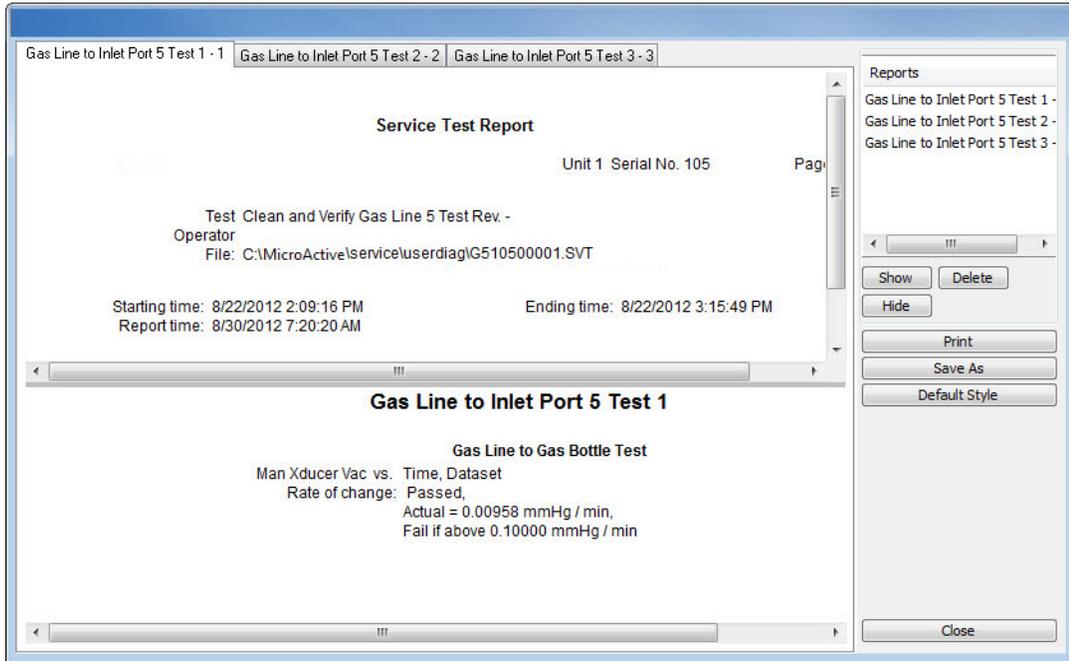
Displays previously run diagnostic service tests. Separate directories store tests run once, daily, weekly, and monthly. Diagnostic Test Report files have a .SVT file extension and are stored in the ...\\Service directory.



1. To open a diagnostic test report, select a service test report and click **Open** or double click the report file name.
2. On the **Selected Reports** window, select which reports to display and click **OK**.



3. The selected reports display on separate tabs across 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:

- 3500.ini
- info[*sn*].dat
- UserInformation.txt
- Any files selected by the user

Follow these instructions to send the problem description and problem files to Micromeritics Customer Support:

1. Complete all fields on this screen. 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 screen 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 screen, the file(s) you added, 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 this computer has no internet connection.
5. Click Save.
6. From a computer with internet connection, go to <http://techsupport.micromeritics.com/portal> to access the Micromeritics Customer Support Center.
7. Either login or register.
8. Click the Requests tab.
9. Click the New Request button and complete all fields on the screen.
10. To attach the zipped file, click the Attach File(s) link and select the Diagnostics-[date].zip file.
11. Click the Add request button to submit the problem request.
12. Return to the portal to track the progress of the problem solution.

User Information

Name:

Address:

Phone number:

Email address:

Micromeritics representative

To the attention of:

Comment:

Include Files:

Follow these instructions to send the problem description and problem files to Micromeritics Customer Support.

1. Complete all fields on this screen. 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 screen 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 screen, the file(s) you added, 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 Center.
7. Either login or register.
8. Click the **Requests** tab.
9. Click the **New Request** button and complete all fields on the screen.
10. To attach the zipped file, click the **Attach File(s)** link and select the *Diagnostics-[date].zip* file.
11. Click the **Add request** button to submit the problem request.
12. Return to the portal to track the progress of the problem solution.

Field or Button	Description
<i>Name</i> <i>Address</i> <i>Phone</i> <i>Email address</i>	Enter information for the person to be contacted by a Micromeritics representative. This information will remain on the screen each time you need to submit files for problem diagnosis but can be modified as necessary.
To the attention of:	Enter the name of your Micromeritics representative. This information will remain on the screen each time you need to submit files for problem diagnosis but can be modified as necessary.
Comment	Type in 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 Customer Support portal.

Field or Button	Description (continued)
<i>Include Files</i>	<ul style="list-style-type: none">• Add - Click to select additional files that you would like sent 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 and click the Delete button to remove the file from the list.• Clear - Click to clear all files from the Include Files box.
<i>Save As</i>	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.
<i>OK</i>	Click to close the window and save all contact information. The Comment and Include Files boxes will be cleared.
<i>Cancel</i>	Click to close the window and cancel changes.

8. TROUBLESHOOTING AND MAINTENANCE

Introduction

The analyzer 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. This chapter includes maintenance and calibration procedures.

Troubleshooting

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 screen and their respective causes and solutions are provided in the following table:

What Happened	Why	What To Do
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. Refer to Replacing the Sample Port Frit , page 8-5.
	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.
Analysis Dewar cannot be raised (or lowered).	Dewar elevator is stuck.	Check for possible obstruction to elevator movement.
Analysis valves cannot be operated.	Cable from computer to the instrument is loose.	Make sure the cable is connected properly

What Happened	Why	What To Do
Elevator is noisy.	The elevator screw may need greasing.	Contact your Micromeritics Service Representative.
Sample is not within specifications.	There may be a leak into or out of the manifold.	Refer to Replacing the Sample Port Frit , page 8-5.
	Gas may be contaminated.	<p>Perform a blank analysis. If results are good, perform a reference material analysis.</p> <p>Replace tank.</p> <p>Check for line leak which could cause contamination.</p> <p>Flush the lines occasionally to help prevent contamination.</p>
	Incorrect type of gas line.	<p>Ensure the gas line is all metal. It is best to use the one shipped with the instrument. Do not use polymer gas lines or flexible gas lines that may be internally coated with a polymer.</p>

Preventive Maintenance

The following table lists the preventive maintenance procedures to keep the analyzer operating at peak performance. Instructions for each procedure follow the table. Micromeritics also recommends that preventive maintenance procedures and calibration be performed by a Micromeritics Service Representative every 12 months.

Maintenance Required	Frequency
Check and clean Dewar	Weekly
Replace port gasket	Every 3 to 6 months depending on the types of analyses you run
Replace sample tube O-ring	As required or every 3 to 6 months
Clean the outside of the analyzer	As required or every 6 months
Test analyzer for leaks	As required or every 12 months
Replace diaphragm in vacuum pump	Every 12 months
Clean or replace power supply air filters	Every 30 days (more often in environments with increased levels of dust)

Recovering 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 started again in order to produce complete results.

Lubricating 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.

Cleaning 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.



Do not allow liquid to penetrate the casing of the analyzer. Doing so could result in damage to the unit.

Cleaning the Analysis Dewar



When handling Dewars, be sure to observe the Dewar precautions outlined in [Filling and Installing the Analysis Dewar](#), page 2-41.

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, in turn, 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 of the sample tube causing 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] > Show Instrument Schematic* to display the instrument schematic, then go to *Unit [n] > Enable Manual Control*.
2. Right-click on the elevator icon and select **Lower** to lower the elevator to its lowest position.
3. Remove the Dewar and pour the liquid nitrogen from the Dewar into an appropriate cryogenic container.



Do not pour liquid nitrogen directly into a sink. Doing so may cause drain pipes to burst.

4. Rinse the Dewar with warm water to melt any ice accumulation which may remain in the Dewar, then dry thoroughly.

Replacing 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.



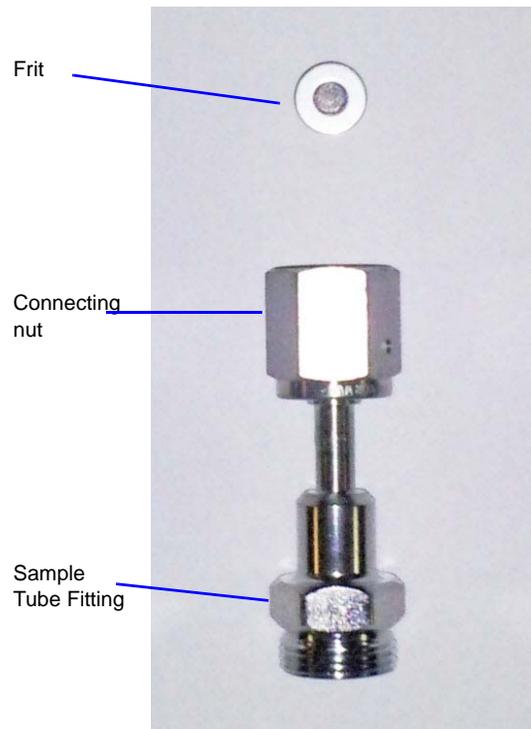
Use a 20 µm frit. The instrument will not operate properly if an incorrect size is used.

1. Go to *Unit [n] > Show Instrument Schematic*, then *Unit [n] > Enable Manual Control*.
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.



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.

Replacing the Psat Fitting Gasket

A gasket is attached to the Psat fitting. Refer to [Installing the Vapor Source Container using a Shelf Support](#), page 2-46.



Each time the Psat tube or Vapor Source container is replaced, a new gasket is required. Do not touch the sealing surfaces of the port fitting or gasket with bare hands.



To avoid degassing problems, the gasket should be clean and should not be touched with bare hands.

Replacing the Sample Tube O-ring

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.

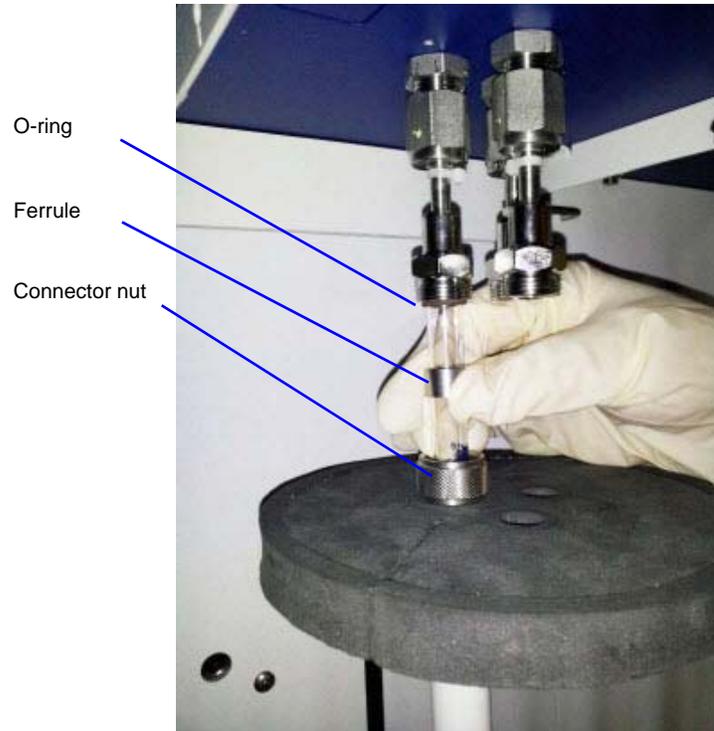


Before removing (or installing) a sample tube, ensure that the port valve is closed. Observe the instrument schematic to verify valve status.

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. Holding the sample tube firmly with one hand, loosen the sample tube connector nut by turning counterclockwise.
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.

Replacing the Psat Tube Ferrules



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 Po 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.

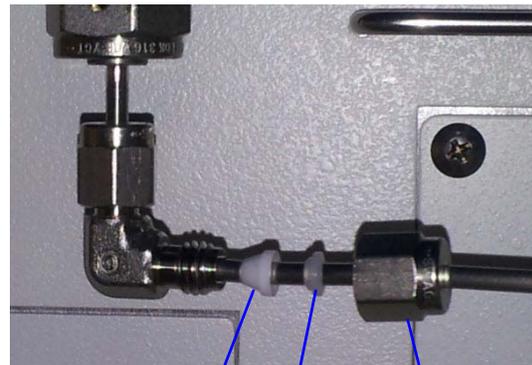


It is recommended that the VCR connector not be removed from the port fitting for this process.



Two ferrule sets are located in the upper and lower Psat nuts. Both sets should be replaced. Additional ferrule sets were included in the instrument's accessory kit.

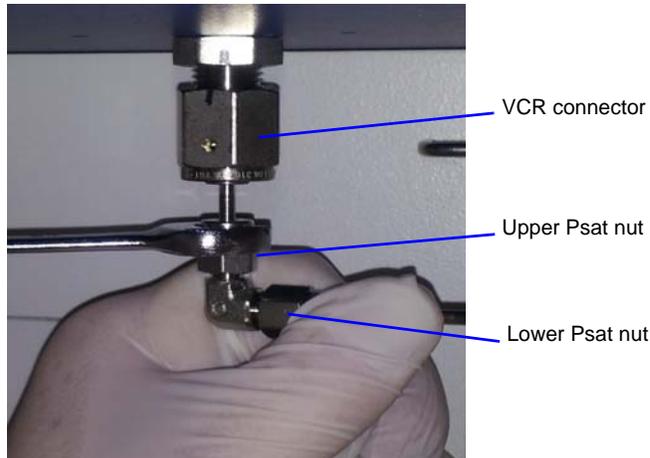
1. Ensure the Psat tube is filled at atmospheric pressure with gas before loosening the Psat nut.
2. To remove the lower Psat nut, use a 7/16 in (11 mm) wrench to loosen the Psat nut by turning the nut counterclockwise.



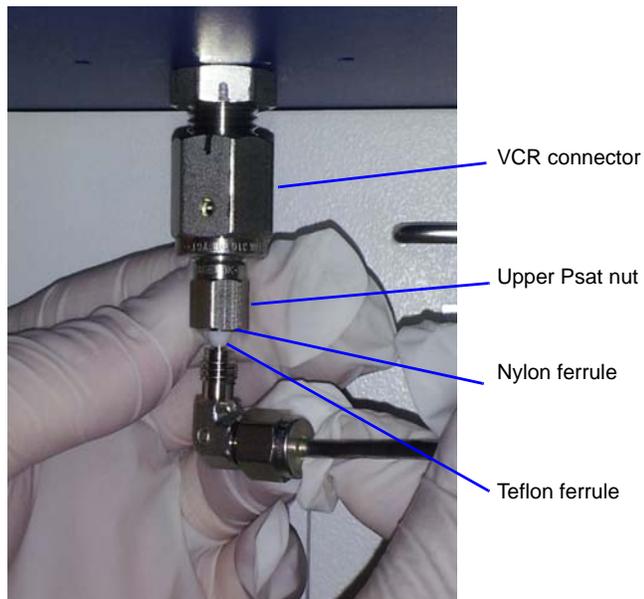
Teflon ferrule
Nylon ferrule
Lower Psat nut

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 counterclockwise.



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.

Cleaning the Power Supply Air Filter

Two power supply air filters are located on the lower rear panel of the instrument and should be cleaned or replaced every 30 days (more often in environments with increased levels of dust).

1. Remove the 4 screws securing the air filter cover.



2. Carefully remove the cover and air filter. Use an air compressor to remove the dust or the rinse with tap water and dry thoroughly. Use caution when removing the cover to avoid breakage.
3. Replace the filter and cover. Secure the cover with 4 screws.

Performing a Reference Analysis

Refer to [Reference Analysis](#), page 4-7.

Performing a Leak 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.

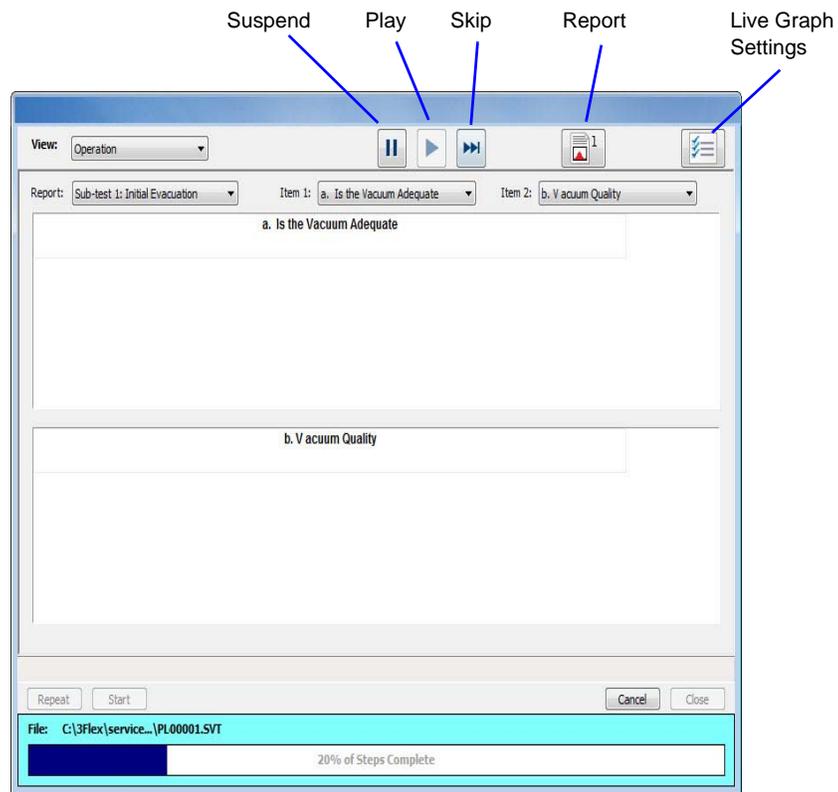
The following information is displayed during the test:

- Prompts on preparing the instrument for the test
 - Approximate time period of the test
 - Prompts in which an operator response is required
1. To start diagnostic testing, go to **Unit [n] > Diagnostics > Start Diagnostic Test**.
 2. Click the dropdown arrow in the **Test** field and select **Ports Leak Test Rev. [n]**.
 3. Resize the window so that the **Report after test** section displays. Select the **Report after test** checkbox and **Preview** and the destination.

The screenshot shows a software window for configuring a diagnostic test. At the top, there is a 'View:' dropdown menu set to 'Operation'. Below this is a 'Test:' dropdown menu with 'Ports Leak Test Rev. P' selected. The 'Operator:' field is empty, and the 'Sequence:' is 'PLdemo00001'. A 'Comments' section contains text: 'This test performs a leak test the system. Requirements: a. The sample ports must have clean sample port plugs or 12 mm sample tubes installed. b. The Po port must have the Po tube installed. c. Nitrogen gas must be connected to inlet port 1. The leak test has Pass / Fail reports. Approximate test time: 2.5 hours.' To the right of the comments is an 'Estimated time:' field set to '168 min.'. Below the comments is a checkbox labeled 'Report after test' which is checked. Underneath is a 'Destination:' dropdown menu set to 'Preview'. Below that is a 'Copies:' field set to '1' with up and down arrows, followed by the text 'copies'. At the bottom of this section is a 'File:' field with a browse button. At the very bottom of the window are three buttons: 'Repeat', 'Start', and 'Close'. A red box highlights the 'File:' field at the bottom of the window.

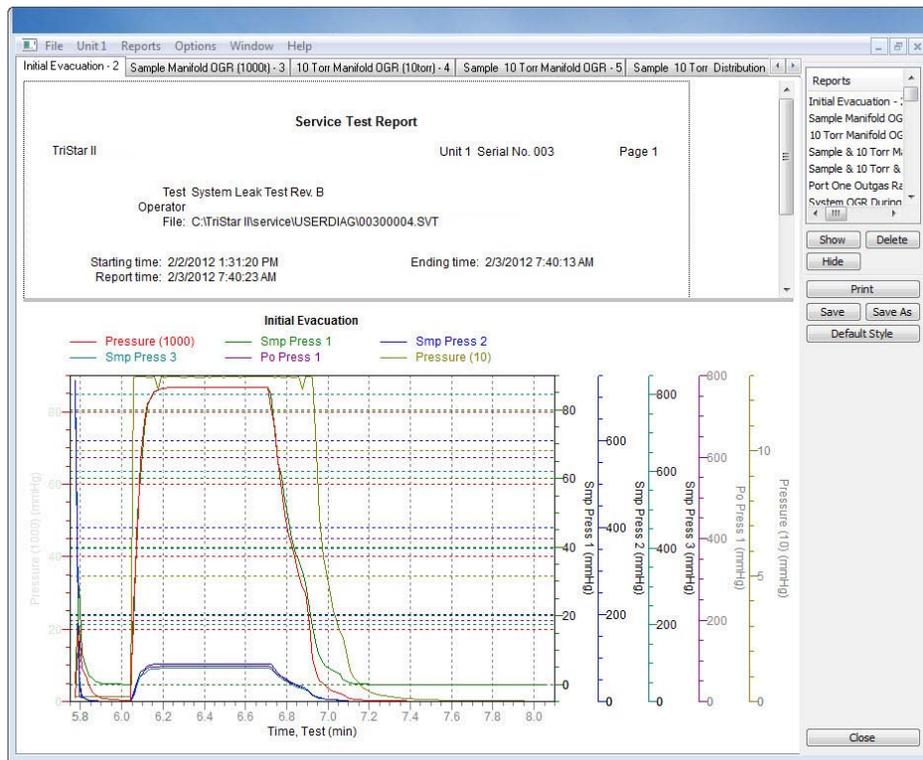
4. Click **Start**.

5. Click **Next**. Data will be inserted into the window as they are collected.

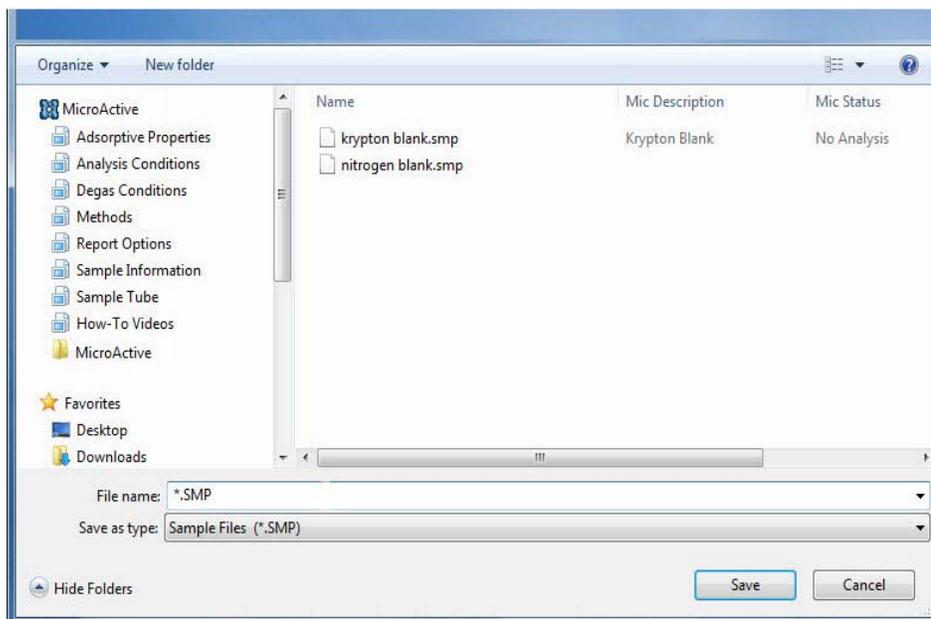


6. Use the **Report** dropdown list to select a report to run.
7. Use the **Item 1** and **Item 2** dropdown lists to change the report details that display in the top and bottom boxes.

- When the test is complete, the report will be displayed.



- To save the report, click **Save** to save the report or click **Save As** to specify a library location and change the default file name.



- E-mail the file to the service representative requesting the report.

Connecting Gases



This procedure is also shown in the Tutorials accessible from the Help menu.

Guidelines for Connecting Gases to the Analyzer

Guidelines when installing regulators and gas lines:

- Place gas bottles close to the analyzer. Using gas line extenders on gas bottles located in remote areas may degrade gas quality and reduce pressure.
- Use a retaining strap (or other appropriate tether) to secure the gas bottle.
- Carefully route the gas lines from the bottle to the analyzer avoiding overlapping or entangling gas lines.
- Label the gas line at the instrument inlet for proper identification and maintenance.
- Ensure the gas bottle is closed before connecting to the analyzer.

The following instructions describe a typical installation. Some configurations require additional components, for example, regulator expansion kits, when one gas source will be used for several operations or when the gas bottle cannot be located close to the analyzer.



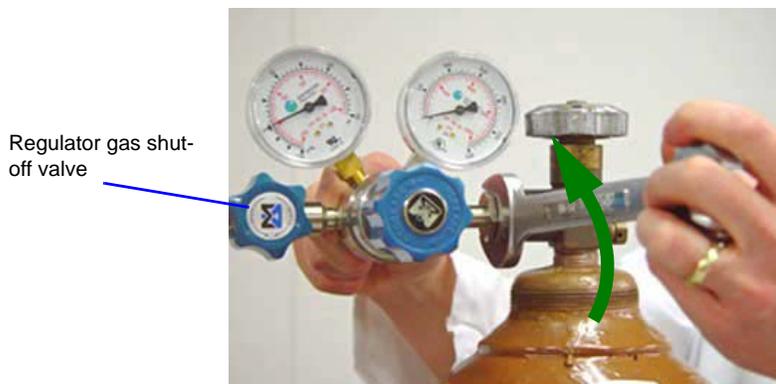
In order to use oxygen, the analyzer must be equipped with an oxygen-compatible vacuum pump that uses Fomblin® (or a suitable equivalent) pump oil or a dry pump. Failure to use the proper vacuum system could result in hazardous conditions including fire and personal injury.

Disconnecting the Depleted Bottle

1. Close the gas bottle shut-off valve then open the regulator shut-off valve.



- Both gauges should read at or near zero. If not, open the regulator gas shut-off valve to release gas. It is not necessary to disconnect the gas line from the regulator or the instrument.
- Use an appropriate wrench to loosen the nut at the regulator/gas bottle connection then remove the regulator from the bottle.



- Replace the protective cap on the depleted bottle. Disconnect the retaining strap and remove the bottle from its current location.

Connecting a Replacement Gas Bottle

Move the replacement bottle close to the instrument and tether it into place. It is not necessary to disconnect the gas line from the regulator or the instrument.



When connecting hazardous gases, ensure there is proper ventilation and always follow the safety procedures established for your lab.



A power failure or loss of cryogen can result in dangerous pressures in the sample tube. The analyzer uses pressure relief valves to vent this pressure into the instrument cabinet and return the instrument to a safe condition. When using toxic or flammable gases, additional venting of the cabinet may be required.

- Use an appropriate cylinder wrench to remove the protective cap from the replacement bottle.



2. Attach the gas regulator to the gas bottle connector. Hand-tighten the nut then use an appropriate wrench to tighten an additional 3/4 turn.



Overtightening the fitting may cause a leak.

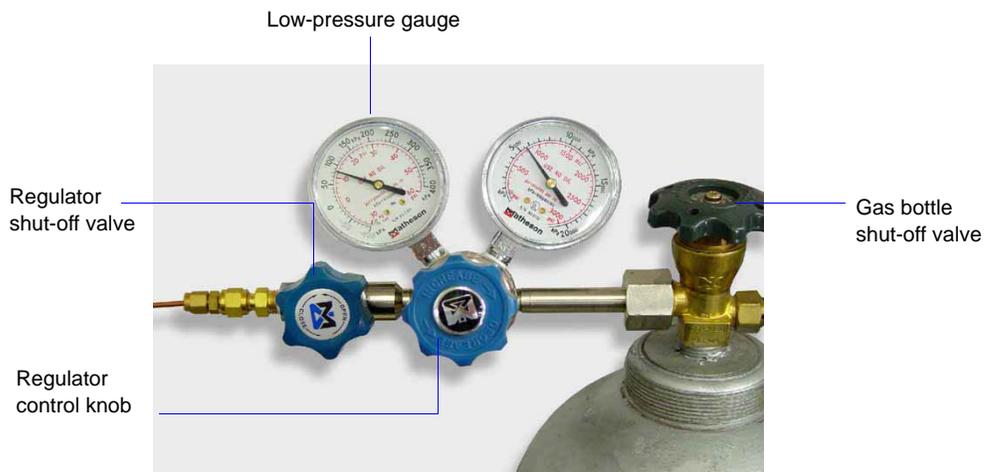
3. Check for leaks at the high-pressure side of the regulator and in the connector.



- a.) Turn the regulator control knob fully counterclockwise.
 - b.) Slowly open the gas bottle shut-off valve then close it.
 - c.) Observe the pressure on the high-pressure gauge.
 - If the pressure is stable, proceed with the next step.
 - If the pressure decreases, tighten the regulator connector nut until it becomes stable.
4. Purge the air from the lines.



- a.) Turn the regulator clockwise until the low pressure gauge shows a few pounds of pressure.
 - b.) Turn the regulator shut-off valve counterclockwise to open.
 - c.) Open the gas bottle shut-off valve to flow gas.
 - d.) Close the regulator shut-off valve to stop flow.
 - e.) Close the gas bottle valve.
5. Set the instrument pressure.



- a.) Turn the regulator control knob clockwise until the low-pressure gauge reads 15 psig (103 kPag).
 - b.) Open the regulator shut-off valve.
 - c.) Open the gas bottle shut-off valve and flow gas for 10 to 30 seconds.
 - d.) Close the gas bottle shut-off valve.
6. If the gas line to the instrument inlet was previously disconnected, reconnect it now.
7. Verify the line for the newly connected gas is clean.
8. If the previously disconnected gas has been reconnected, resume operation.
If a different gas has been connected, the change must be specified in the software. Refer to [Specifying Gas Ports](#), page 8-22.

Cleaning and Verifying the Gas Line

Always clean the gas lines and verify there are no leaks at the connections after a gas bottle is connected. This test examines the gas line from the instrument to the gas bottle, then from the instrument to the regulator shut-off valve. A report is generated at the completion of the test verifying 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. To start diagnostic testing, go to **Unit [n] > Diagnostics > Start Diagnostic Test**.
2. Click the down arrow to the right of the **Test** field and select **Clean and Verify Analysis Gas Line [n] Test Rev [n]**. The length of time a test will run is also indicated on the screen. The **Sequence** field indicates the name of the file created as a result of this test.

View: Operation

Test: [Dropdown]

Operator: [Text Field] Sequence: AL10500001

Comments [Text Area] Estimated time: 60 min.

Report after test

Destination: Preview

Copies: 1 copies

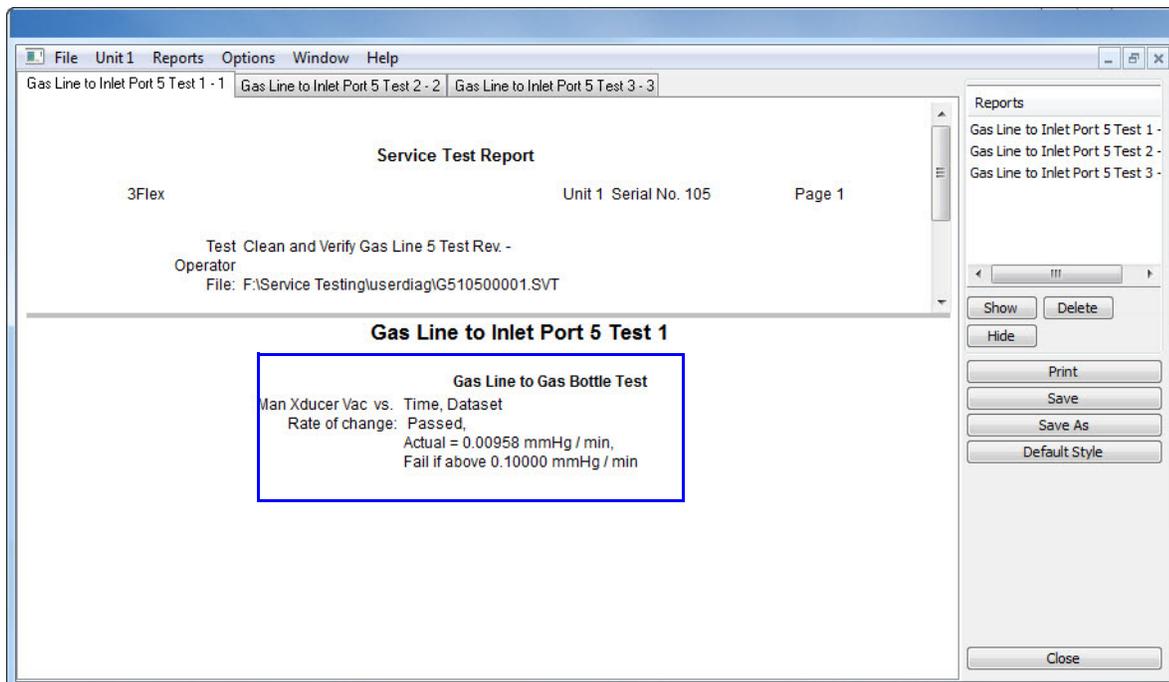
File: C:\3FLEX\DATA\

Repeat Start Close

File: [Text Field] Step: [Text Field]

3. Resize the screen, if necessary to display the **Report after test** and select **Preview** as the destination. Click **Start**.

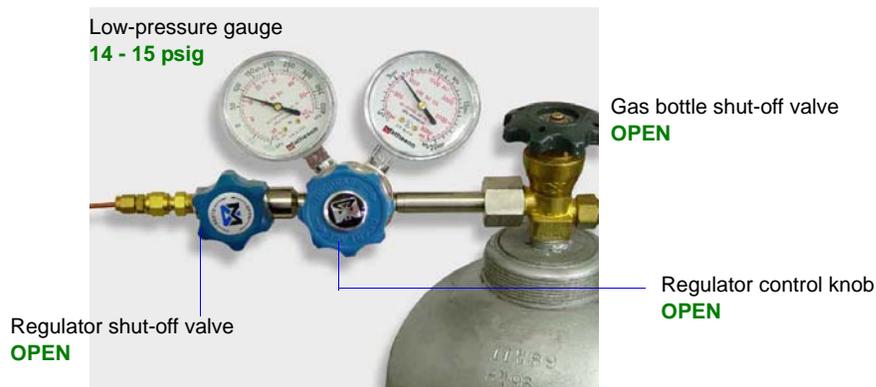
4. From the **View** dropdown list, select either **Operation**, **Instrument Log**, or **Instrument Schematic**. Refer to **Sample Analysis**, page 4-3.
5. A following series of prompts display on the screen requiring operator response.
 - a.) This is the gas line clean and leak check test for inlet port [n]. Inlet ports being tested must be connected to a gas bottle 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 overtightening) 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 bottle 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.
6. A popup window indicates the test is complete. Click **OK**. The reports display on the screen.



7. Click each tab and look for a reading of **Passed**. A Passed reading indicates all valves are in proper state for operation. If any test shows a **Failed** reading, refer to the following table for the location of the gas leak.

Tab	Test	If Failed status, then...
Gas Line to Inlet Port [n] Test 1	Gas Line to Gas Bottle 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 bottle. 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 and ensure the appropriate pressure is displayed on the low-pressure gauge.



If running the test again, close the gas bottle valve before starting the test.

8. Click **Close** to close the test report. Click **Close** again to close the test.

Specifying Gas Ports

The analyzer has gas inlets for up to six analysis gases. The gases connected to the inlets must be specified in the analysis program. If the gases are changed on one of the inlets, make the same change on the **Unit Configuration** window. The analysis program must be kept informed of any changes in gases.

1. Go to *Unit [n] > Unit Configuration*.

The screenshot shows a 'Unit Configuration' dialog box with the following fields and controls:

- Configuration:** IP address: 192.168.77.3, Serial #: 217. Buttons: Change IP..., Board ID...
- Software Versions:** MIC BIOS: Demo Boot Block, Controller: Demo Application, Application: 3Flex
- Options:** Micropore: Port 1, Port 2, Port 3, Chemisorption: No. Button: Calibrations...
- Gas Selections:** Valve 11 gas: Ar, Valve 12 gas: Kr, Valve 13 gas: N2, Valve 14 gas: (empty), Valve 15 gas: (empty), Valve 16 gas: (empty)
- Buttons:** OK, Cancel

2. In the **Gas Selections** group box, enter the mnemonics for the gases attached to the gas inlets.
3. Click **OK**, then click **Close**.

Calibrating the System

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

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

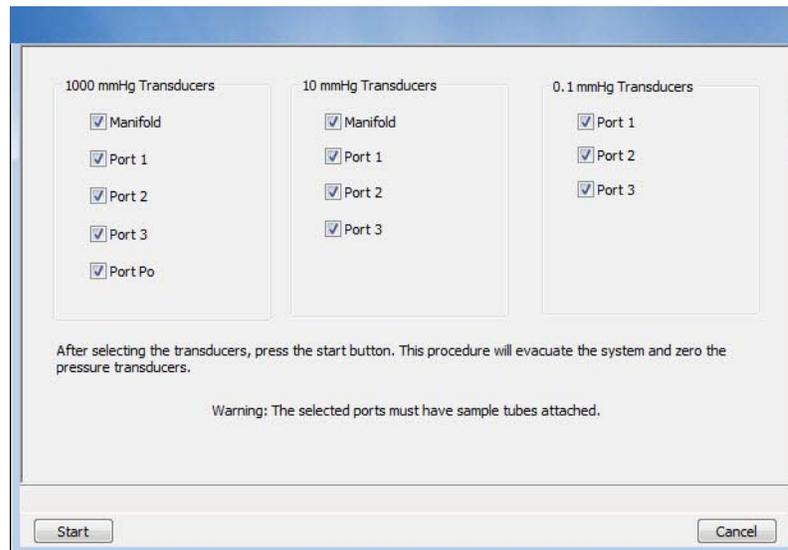
The following calibrations can be performed without the assistance of a service representative:

- Zero pressure
- Match transducers
- Servo valve

Zero Pressure

Use this option to evacuate the system and zero the transducers.

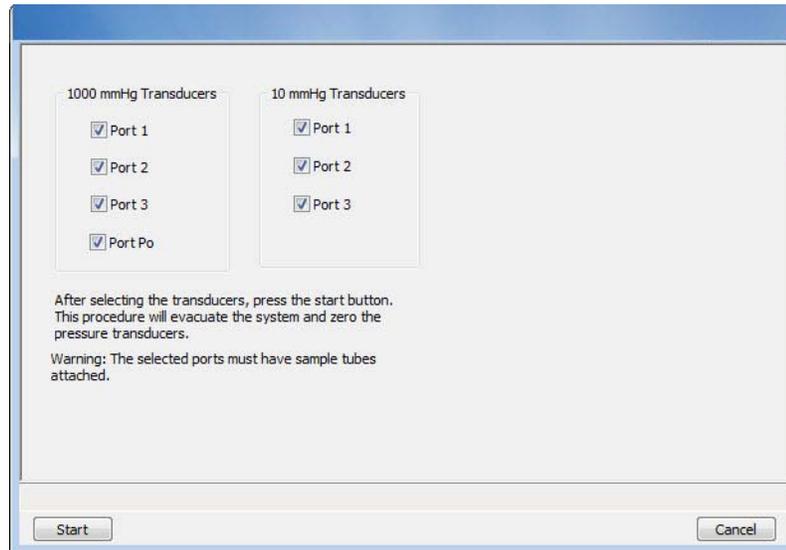
1. Install a small plug on each applicable port.
2. Go to *Unit [n] > Calibration > Pressure Offset*.



3. Ensure that all applicable transducers are selected, then click **Start**. The window closes when the operation is complete.

Match Transducers

Use this option to zero and match the selected transducers to the main manifold transducer.



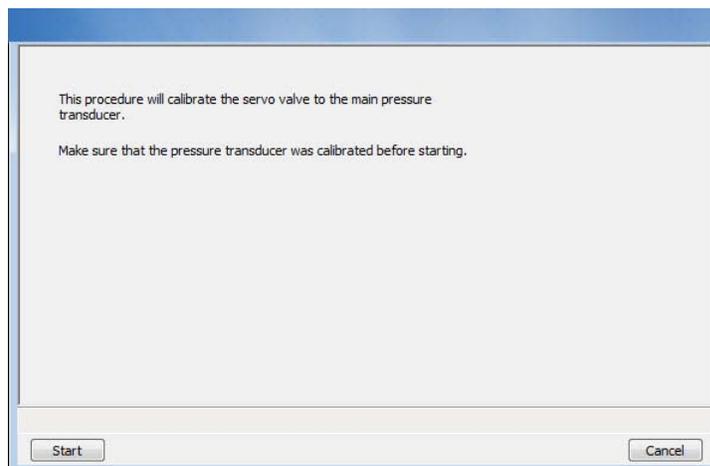
A small plug must be installed on each selected port before clicking **Start**.

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.

1. Go to *Unit [n] > Calibration > Servo Valve*.
2. Click **Start**. The window closes when the calibration is complete.



9. ORDERING INFORMATION

The analysis system components and accessories can be ordered using one of the following methods:

- Call our Customer Service Department at (770) 662-3636
- Email orders to orders@micromeritics.com
- Contact your local sales representative

Please use the following information to place an order.

Part Number	Item and Description
Air Compressors	
011-62701-11	Air Compressor 115 Vac 0.71 CFM for operation of pneumatic valves
011-62701-23	Air Compressor 230 Vac 0.71 CFM for operation of pneumatic valves
350-34040-00	Pneumatic Filter Assembly for air source
Analyzer Optional Equipment	
060-00035-00	FlowPrep 060, degasses up to six samples at up to 400 °C with flowing gas. A gas source and regulator is required.
061-00035-00	VacPrep 061, degasses up to six samples at up to 400 °C; uses flowing gas or vacuum. Evacuation requires a vacuum pump and flowing gas requires a gas source and regulator.
065-00035-00	SmartPrep 065, Windows interface provides programmable ramp and soak rate for degassing up to six samples with flowing gas. A gas source and regulator is required.
350-33015-00	Vapor Adsorption option - includes stainless steel reservoir, isolation valve, and heating mantle. Provides the ability to perform three vapor analyses simultaneously.
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. This option is service installed.
Cables	
003-63801-01	Cable, Ethernet straight-thru for connecting instrument to control module (computer).

Part Number	Item and Description (<i>continued</i>)
Dewar and Accessories	
240-25901-00	Dip stick for checking liquid nitrogen level in Dewar
350-25825-00	Dewar, Glass, 3.2 liters
350-31700-00	Dewar lid, for 9 mm sample tubes
350-31701-00	Dewar lid, for 12 mm sample tubes
Gas Bottle 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)
004-62230-32	Gas Regulator, CGA 320, 30 psig (CO ₂)
004-62230-58	Gas Regulator, CGA 580 fitting, 30 psig (He, N ₂ , Kr, Ar)
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	
350-26001-00	Vapor Heating Mantle
350-53700-00	Degas Mantle top for 350-26000-00 (Glass-Col mantle)
350-53701-00	Degas Mantle with top
350-53701-01	Degas Mantle top for 350-53701-00

Part Number	Item and Description (<i>continued</i>)
Isothermal Jacket	
350-25812-02	Isothermal Jacket, 9 mm ID
350-25812-03	Isothermal Jacket, 12 mm ID
Operating Supplies	
350-33609-00	Extended operating supplies, 9 mm. Includes sample tubes, filler rods, O-rings, reference materials and other accessories.
350-33610-00	Extended operating supplies, 12 mm. Includes sample tubes, filler rods, O-rings, reference materials and other accessories.
Reference Material	
004-16821-00	Reference Material, Silica alumina, ~ 215 m ² /g, 10 g
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
004-27070-00	Frit, 20 µm, 1/4 in.
004-32004-00	Rubber stopper for sample tubes (fits various sample tube sizes)
004-54104-01	16 in. Brush, for cleaning 12 mm sample tubes
004-54104-02	16 in. Brush, for cleaning 9 mm or 12 mm sample tubes
004-54618-00	Tool, for removing the sample port O-ring
240-14855-00	Rack, sample tube holder
240-25853-00	Funnel, sample tube
300-32800-00	Support, sample weighing

Part Number	Item and Description (<i>continued</i>)
350-25843-01	Ferrule, for 9 mm sample tube
350-25843-00	Ferrule, for 12 mm sample tube
350-25864-00	Check Seal assembly for 12 mm 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, 3 Check Seals, and 1 extractor tool
350-61002-02	Sample Tube, 9 mm, flat bottom
350-33608-00	TranSeal Kit, 12 mm
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
Software and Manuals	
350-20800-00	3Flex - current version software
350-33001-00	3Flex - operator's manual and 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
004-62023-01	Service kit for dry diaphragm vacuum forepump

A. FORMS

This appendix contains the following form:

- Sample Data Worksheet

Copy and use this form as needed.



Sample Data Worksheet

Use this worksheet to record the values necessary to calculate the sample mass.

Sample Tube Identification: _____

Sample Mass (g)			
NOTE: 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 (°C):	_____
Time (hours):	_____
Actual time started:	_____
Actual time finished:	_____

Degas notes: _____

B. ERROR MESSAGES

Program error messages are listed numerically. If the **Action** response indicates to contact a Micromeritics service representative, record the error message and make backup copies of any files involved in the operation.



The 1000-series error messages, used primarily for software testing, are not included in this appendix. These errors should not occur during normal operation. If a 1000-series message appears, contact a Micromeritics service representative after making backup copies of any files involved in the operation.

2400 Series

2401- FATAL ERROR: [error message]

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 [file name], 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.
Action B:	Contact your Micromeritics service representative.
Cause C:	A software error occurred when the file was accessed.
Action C:	Contact your Micromeritics service representative.
Cause D:	The file name specified contains one or more invalid characters.
Action D:	Enter a valid file name. Refer to the operating systems manual.

2431- Error writing file [*file name*], error code = [*n*].

Cause:	Insufficient hard disk space to perform the operation.
Action:	Copy files not used regularly from the hard disk to an external media, delete them from the hard disk and try the operation again

2432- Invalid response from MMI 'FILE_READ' request.

Cause:	An internal processing and/or hardware error has occurred.
Action:	Contact your Micromeritics service representative.

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:	Select Yes to update the directory information with data from each new file. This operation may take a minute. Select 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 name of the file is known.

2434- File [*file name*] — Subset # [*n*] wrote wrong amount of data. Expected [*n*] bytes.

Cause:	An internal processing and/or hardware error has occurred.
Action:	Contact your Micromeritics service representative.

2436- Path specification [*path name*] is invalid.

Cause:	An invalid path name and/or extension was entered.
Action:	Type a valid path name (including the proper extension) and press Enter .

2437- File/Overlay file [*file name*] 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.**
2440- Subset not found.
2441- Seek within file failed.
2442- Bad header in subset file.
2443- Subset owner denied access.
2444- Not a valid file format.
2445- Subset wrote the wrong amount of data.
2446- Error reading data.
2447- Error writing data.

Cause:	An unexpected error occurred when trying to access a data file.
Action:	Contact your Micromeritics service representative.

2448- File directory [*path name*] is invalid. Resetting to the installation directory.

Cause:	A working directory specified in the .INI file is invalid or has been moved or 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 and 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 and 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 and retry the operation again.

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 and set the limit for open files to 50 or greater.

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 stopped.
Action:	Wait a short time and attempt to stop 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? [Yes, No]

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 [Unit n - S/N: nnnn]

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 has the power switch 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- Unit [n] - S/N: [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 error messages above.
Action:	Correct the problem as described above then restart the application.

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, for example, consider using a utility, for example, ScanDisk. Contact your Micromeritics service representative.

2479- Unit [n]; Serial [n] 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 allowing the analyzer to reset and 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 [name] 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 [file name].

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. It is already in use.

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 windows, one of which may contain this file).

2483- An analysis cannot be performed on [file name]. 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 and save it.

2484- The edit session for [file name] must be saved before the analysis. Save changes and continue with the analysis? [Yes, No]

Cause:	An attempt was made to start an analysis using a file that contains unsaved changes and is open for editing.
Action:	Select Yes to save the changes and proceed with the analysis. Select No to cancel the analysis and 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 service test file has a status other than <i>No Analysis</i> .
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 [name] report type. Program will terminate.**
2487- Could not start report generator. Error code [number]. 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 [file name] cannot be opened for editing. It is already in use.**

Cause:	The specified file is being used in another edit operation.
Action:	Check the Windows list to locate the other edit session.

- 2489- File [file name] cannot be opened for writing. It is already in use.**

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 file containing initialization information and system options information used during program startup does not exist.
Action:	Run the analyzer setup program, select Change analyzer setup and create the control file used by the analyzer.

- 2491- Highlighted fields contain errors. Please correct the errors before closing dialog box.**

Cause:	The highlighted fields contain invalid entries. The dialog box cannot be closed until all errors are corrected.
Action:	Check the entries, correct the errors and 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.**2495- Value is out of the valid range. Enter a value between [value] and [value].**

Cause:	The entered value in the highlighted field is outside the valid range of values.
Action:	Check the entry and enter or select an appropriate value.

2496- Invalid number.

Cause:	An invalid number entered in the highlighted field.
Action:	Check the entry and 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 and 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 <i>automatically collected</i> to <i>manually entered</i> 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 Series

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:	Select <i>Options > Default Method</i> and 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 on the screen. 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 [*file name*] 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 or 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 <i>No Analysis</i> or <i>Prepared</i> status.
Action:	Select a different file with a status of <i>No Analysis</i> or <i>Prepared</i> .

2513- Unable to read the calibration file [*file name*].

Cause:	An invalid calibration file was selected or one that cannot be read.
Action:	Ensure the media containing the calibration file has no problems.

2514- Unable to write the calibration file [file name].

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:	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, press 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 the controller failed.**2524- CRC check failed on programming controller.****2525- Unknown error programming controller.****2526- Controller download was not successful.****2527- Controller CRC error on boot block.****2528- Controller DRAM error.****2529- Controller Com1: error.****2530- Controller Com2: error.****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, select Yes and 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.

2548- System status [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 [code #].

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- Unable to establish the TCP connection with the instrument.

Cause:	TCP connection could not be established
Action:	Check the cable connection. Ensure the addresses match those selected in the Control Panel for TCP/IP connections (network properties).

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 [file name] can not be created!.

Cause:	A required dialog could not be found by the software.
Action:	Re-install the software.

2556- File [file name] cannot be opened. It is currently being setup for an analysis**2556- Directory database [n] error [n]**

Cause:	The sample file is currently being analyzed 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 is [sn] 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 . Select <i>Unit [n] > Calibration > Load from File</i> and select a file containing calibration data.
Action B:	Click OK . Close the application and use the Setup program to reinstall calibration files.

4000 Series

4002- Thermal Transpiration correction had no effect.

Cause:	The Thermal transpiration correction option was selected on the Report Options window; however, the correction did not change any pressure by more than one percent.
Action:	Deselect this option to disable this message. This correction is only meaningful for very low pressures.

4003- Error Converting Pressures.

4004- Error Computing Volume Adsorbed.

Cause:	An internal processing and/or hardware error occurred during report generation.
Action:	Contact your Micromeritics service representative.

4005- Pressures were not smoothed. Not enough pressures below 0.10 P/P₀

Cause:	The Smooth pressures below 0.10 P/P₀ option was selected on the Report Options window. There must be at least 10 pressures within this range for smoothing to occur.
Action:	Deselect this option to disable this message.

4011- Analysis gas in sample file 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 A:	If the wrong adsorptive was selected in the sample information file, change the adsorptive in the file.
Action B:	If necessary, attach the appropriate gas bottle then enter the gas in the Unit Configuration.

4012- P_{sat} gas in sample file does not match any gas in the unit.

Cause:	If using <i>Measure psat of a gas</i> in P ₀ 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 [name] 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 [file name].

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 [sample file name] has no data for export.

Cause:	The file selected for export has a status of <i>No Analysis</i> . No export file will be created.
Action:	Select a file which contains analysis data.

4017- Damage to the instrument will result if the sample 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, select Yes . If not, select No and then either perform a manual evacuation or go to the Sample Backfill Options window and 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, select Yes . Otherwise, select No and go to the Sample Backfill Options window and select Backfill sample at start of analysis.

4021- The entered Po value (Po and Temperature Options of the Analysis Conditions)

is outside the range of the pressures listed in the Psat vs Temperature Table (Adsorptive Properties).

Cause:	The entered Po value is not within the range of pressures selected for analysis.
Action A:	Enter a new Po value.
Action B:	Add more pressures and corresponding temperatures to the Analysis Conditions pressure table to include the presently selected Po value.

4022- The entered bath temperature value (Po 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 [file name] 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 bottle, then enter the gas in the Unit Configuration.

4026- Cannot calculate Dubinin-Astskahov: 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 and 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 the calculation assignments or 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 assignments or the fitted relative pressure range.

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: Not enough points with a relative pressure in the range $[(pressure), (pressure)]$.

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 number of the error message which preceded this one for an explanation.

4038- Fewer than 2 points available for the Langmuir Qm computation. Cheng/Yang correction will not be applied.

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. Cheng/Yang correction will not be applied.

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 and reproduce the Horvath-Kawazoe reports.

4040- The value of Qm computed from the Langmuir equation is too low. The pore size will not be computed for all data points.

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 V_m . 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 P/P₀.

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 assignments are not being used and 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 assignments are not being used and 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- [file name] could not be opened for reading.

Cause:	A thickness curve file could not be opened.
Action:	If the problem persists, restart the computer and optionally perform a media integrity check (using ScanDisk).

4047- Warning: An error occurred while reading [file name].

Cause:	An error happened during a read operation of a thickness curve file.
Action:	If the problem persists, restart the computer and optionally perform a media integrity check (using ScanDisk).

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 [file name] 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 [*file name*] 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 assignments for 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 assignments 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 assignments 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.

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 assignments for the MP-Method report.

4060- Sample [file name] 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 which no instrument operations has been used. Select a sample file with a status of <i>Prepared</i> , <i>Preparing</i> , <i>Analyzing</i> or <i>Complete</i> 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 [name].

Cause:	For some reason, the list of available models was corrupted, therefore, the model selected could not be loaded for the deconvolution.
Action:	Exit the program and 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 [name] does not match the model gas [name].

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 [nn] does not match the model temperature [nn].

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: [file name]

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 and select a surface area option for Volume Adsorbed .
Action B:	Click Overlays on the Report options window of the primary file and remove the named overlay file from the list.

4078- Slope and Y-Intercept cannot be determined from the selected points.

Cause:	Less than two data points have been selected for the Langmuir report, at least two are required.
Action:	Edit the selection of data points on the Langmuir interactive editor or on the Langmuir pressures window.

4112- Hard-sphere diameter and molecular weight have been updated from the fluid property information.

Cause:	A fluid property information (FPI) file was loaded. Entry fields for hard-sphere diameter and molecular weight were updated with the values in the FPI file
Action:	This message is informational; no action is required.

4400- The computer does not have the communications port specified for the Smart-Prep(s). Cannot initialize.

Cause:	The communications port associated with this unit was not valid.
Action:	Run the setup program and set up the unit on a valid port.

4401- The communications port specified for the SmartPrep(s) is already in use. Cannot initialize.

Cause:	The communications port associated with this instrument is in use by some other program in the system.
Action:	Close the other program to release the port. Restart the analysis application.

4402- The communications port specified for the SmartPrep cannot be accessed. Cannot initialize.**4403- Cannot communicate with SmartPrep Unit [n] - S/N: [nn].**

Cause:	The communications port associated with this unit was not valid.
Action:	Run the setup program and set up the unit on a valid port.

4404- The application version of the SmartPrep Unit [n] - S/N: [nn] is invalid.

Cause:	The controller software running on the designated instrument is invalid.
Action:	Use the SmartPrep setup program to download the proper controller software to the instrument, or if unavailable, contact a Micromeritics service representative.

4405- Fatal communications error with SmartPrep Unit [n] - S/N: [nn].

Cause:	There was a fatal error in the serial communications between the application and the SmartPrep Instrument Controller. All displays for that SmartPrep will be closed.
Action:	Ensure that the SmartPrep is properly chained to the computer on the communications port configured in the Setup program. Stop and re-start the application. Contact your Micromeritics service representative.

6000 Series

6000- An error occurred while loading the application control information. Data entry cannot be performed. [Code Number]

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.

6003- Unable to read the calibration file [number].

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 [number].

Cause:	An attempt to save the calibration to a separate file was unsuccessful.
Action:	Ensure that the disk is not full or write-protected, then try again.

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 and perform the analysis. Then 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.**6013- Cannot read the adsorptive properties parameter file.****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 your 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 selected sample tube file on the QuickStart screen cannot be read.
Action:	Select a different file.

6016- Dosing manifold from valve [number] 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 bottle is empty.
Action B:	Connect a new analysis gas bottle then resume the analysis.

6017- Leak test failed on port [number].

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 1000 cm³ STP. 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 upper/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 the bottom or the neck of the Dewar preventing the elevator from rising completely.
Action B:	Check the Dewar, remove ice and restart the analysis.
Action B:	If results for Actions A and B failed, contact a Micromeritics service representative.

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. Refer to Servo Valve , page 4-32.

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 bottle is empty.
Action B:	Connect a new analysis gas bottle, 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.

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 [PRn] [PR-U] exceeded maximum manifold pressure of [PRn] [PR-U]. 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 and 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 bottle is installed at one of the analysis ports, then Select <i>Unit</i> > <i>Unit configuration</i> and enter N2 for the appropriate valve.

6028- The backfill gas in sample file does not match any gas in the unit.

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 A:	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 or 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 or select a different file for the analysis.

6032- Template file [file name] 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 ([PR1] [PR-U]).

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: <ol style="list-style-type: none"> 1. Evacuate the psat tube. 2. Backfill with krypton gas to 20 mmHg. 3. Raise the Dewar. <p>These steps should condense the krypton gas to a pressure below 3 mmHg.</p>
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: <ol style="list-style-type: none"> 1. Evacuate the psat tube. 2. Backfill with krypton gas to 20 mmHg. 3. Raise the Dewar. <p>These steps should condense the krypton gas to a pressure below 3 mmHg.</p>
Cause: B:	The Dewar does not contain enough cryogen.
Action B:	Refill the Dewar.

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.**6042- Master pressure transducer calibration failed. Offset is out of range.****6043- Port 1 pressure transducer calibration failed. Offset is out of range.****6044- Port 2 pressure transducer calibration failed. Offset is out of range.****6045- Port 3 pressure transducer calibration failed. Offset is out of range.****6046- Po pressure transducer calibration failed. Offset is out of range.****6047- 10 torr pressure transducer calibration failed. Offset is out of range.****6048- Master pressure transducer calibration failed. Scale is out of range.****6049- Port 1 pressure transducer calibration failed. Scale is out of range.****6050- Port 2 pressure transducer calibration failed. Scale is out of range.****6051- Port 3 pressure transducer calibration failed. Scale is out of range.****6052- Po pressure transducer calibration failed. Scale is out of range.****6053- 10 torr pressure transducer 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.

6557- File [file name] 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 [file name] 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 A:	If the wrong adsorptive was selected in the sample information file, change the adsorptive in the file.

10000 Series

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: [PR4] [PR-U], new: [PR4], nominal: [PR4], max: [PR4]).

Cause:	The transducer offset calibration was rejected.
Action:	Contact your Micromeritics service representative.

10190- Transducer scale calibration rejected (current: [PR4] [PR-U], new: [PR4], nominal: [PR4], min: [PR4], max: [PR4]).

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.

10210- Transducer overrange detected.

Cause:	A manifold pressure over 1000 mmHg was detected.
Action:	Observe caution when operating the analyzer manually. If the problem persists contact a Micromeritics service representative.

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: [0] s, max allowed: [0] s, status: [HEX], alarm code: [HEX], inputs: [HEX], position: [0]).

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: [0] s, max allowed: [0] s, status: [HEX], alarm code: [HEX], inputs: [HEX], position: [0]).

Cause A:	There is a problem with the elevator.
Action A:	Check the Dewar and remove ice if necessary. Then restart the analysis. Contact your Micromeritics service representative if necessary.
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: [HEX]).

Cause:	There is a problem with the elevator.
Action:	Contact your Micromeritics service representative.

10390- Homing of the elevator failed (position: [0], home sensor: [0]).

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- [PORT] over-pressure detected (pressure: [PR4] [PR-U], max allowed: [PR4]).

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. If problem repeats, contact your Micromeritics service representative.
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. If problem repeats, contact your Micromeritics service representative.
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. If problem repeats, contact your Micromeritics service representative.

10480- Operation cancelled by operator.

Cause:	The operator canceled the operation.
Action:	None.

10490- Operation cancelled 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. If you are on the analysis window, click the Play button to resume the operation.

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. If you are on the analysis window, click the Play button to resume the operation.

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: [DegC1] [DegC-U], port: [DegC1] [DegC-U], heater: [DegC1] [DegC-U], heater target: [DegC1] [DegC-U], power: [2]).

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 and then turn the power ON. Restart the application program. If the problem repeats or is not resolved, contact your Micromeritics service representative.

10720- Manifold heater breaker open (manifold: [DegC1] [DegC-U], port: [DegC1] [DegC-U], heater: [DegC1] [DegC-U], heater target: [DegC1] [DegC-U], power: [2]).

Cause:	The circuit breaker to the manifold heater is open.
Action:	Contact your Micromeritics service representative.

10730- Mantle temperature error detected (type: [0], actual: [DegC1] [DegC-U], max allowed: [DegC1] [DegC-U], target: [DegC1] [DegC-U], power: [2]).

Cause:	An error was detected with the mantle temperature.
Action:	Contact your Micromeritics service representative.

10740- Mantle breaker open (type: [0], actual: [DegC1] [DegC-U], target: [DegC1] [DegC-U], power: [2]).

Cause:	The circuit breaker to the mantle is open.
Action:	Contact your Micromeritics service representative.

10750- Time limit exceeded during evacuation (target: [PR4] [PR-U], pressure: [PR4] [PR-U], elapsed: [0] 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. Verify that the sample is properly degassed; then restart the analysis.

10760- Time limit exceeded while dosing (gas: [GAS], valve: [0], target: [PR4] [PR-U], pressure: [PR4] [PR-U], elapsed: [0] 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 bottle is empty.
Action B:	Connect a new gas bottle. Then restart the analysis.

10770- Attempts to dose failed on sample port [0]. (qty required: [Q4] [Q-U], qty dosed: [Q4] [Q-U], sample pressure: [PR6] [PR-U], gas: [GAS]).

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: [0], interval: [0] s, leak rate: [PR4] [PR-U]/min, max allowed: [PR4] [PR-U]/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 [0] ([Q2] [Q-U]) has exceeded the maximum of [Q2] [Q-U] (qty dosed this point: [Q2] [Q-U], pressure: [PR4] [PR-U], [P0Sym]: [PR4] [PR-U], gas: [GAS]).

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 and correct the problem before repeating the analysis.

10801- P0-over-sample failed on sample port [0] (pressure: [PR4] [PR-U], last pressure: [PR4] [PR-U], [P0Sym]: [PR4] [PR-U], rel pressure: [4], qty ads: [Q2] [Q-U], doses: [0]).

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 and correct the problem before repeating the analysis.

10830- Warm free-space measurement failed on sample port [0] (qty in free-space: [Q2] [Q-U], qty in port: [Q2] [Q-U], pressure: [PR4] [PR-U], port vol: [V4] [V-U], port temp: [DegC1] [DegC-U]).

Cause:	There is a problem with the warm free-space measurement on the sample port.
Action:	Verify that no problem exists with the sample tube or gas connection.

10840- Cold free-space measurement failed on sample port [0] (qty in free-space: [Q2] [Q-U], qty in port: [Q2] [Q-U], pressure: [PR4] [PR-U], port vol: [V4] [V-U], port temp: [DegC1] [DegC-U], warm free-space: [V4] [V-U]).

Cause:	There is a problem with the cold free-space measurement on the sample port.
Action:	Verify there is no problem with the sample tube or analysis bath.

10850- Maximum target pressure exceeded in sample port [0] (target pressure: [PR4] [PR-U], [P0SYM]: [PR4] [PR-U], max instrument manifold pressure: [PR4] [PR-U], gas: [GAS], max gas manifold pressure: [PR4] [PR-U], max transducer pressure: [PR4] [PR-U]).

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 [GAS] is not condensing. (pressure: [PR4] [PR-U] maximum manifold pressure: [PR4] [PR-U]).

Cause:	The Psat gas is not condensing.
Action:	Review the analysis parameters, gas connections, and analysis bath.

10870- Adsorptive [GAS] is not condensing. (pressure: [PR4] [PR-U] maximum manifold pressure: [PR4] [PR-U]).

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 [GAS] failed at [PR4] [PR-U] (charge pressure: [PR4] [PR-U], minimum allowed: [PR4] [PR-U]).

Cause:	The adsorptive gas failed to purify at the specified pressure.
Action:	Check the gas connection.

10902- Sample pressure on sample port [0] ([PR4] [PR-U]) is below the minimum desorption pressure ([PR4] [PR-U]).

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: [0], voltage: [2] volts, nominal: [1] volts).

Cause:	There is a problem with the power supply voltage.
Action:	Contact your Micromeritics service representative.

11002- Manifold heater temperature error (measurements: [0], mean: [3], std dev: [3], min: [3], max: [3], since: [DATE]).

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.

C. CALCULATIONS

This appendix contains the calculations used in the analysis program.

Saturation Pressure

Saturation pressure (P_o) is selected on the **Po and Temperature Options** window. It may be entered or measured in the Po tube. The uses the following methods to get P_o :

1. P_o is measured in the Po 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_o for all data points.
3. P_o is measured for all points as with #1. After all adsorption points have been taken P_o is measured in the sample tube. The measured P_o points are shifted so that the P_o measured in the Po tube matches the P_o measured in the sample tube. That is, $P_o(i) = P_o(i) + P_{o_s} - P_{o_n}$ where P_{o_n} is the P_o measured in the Po tube when P_o in the sample tube (P_{o_s}) was measured.
4. Determine P_o 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 Po 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_o of the analysis gas is found from the bath temperature as in #6. If dosing is done from the Psat tube, P_o is determined once at the beginning of the analysis and used for all data points. Otherwise, P_o is measured for each data point.
6. P_o 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, $\ln(P) = \frac{a}{T} + b$. 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, $P_o =$ user-entered value.

Relative Pressure Calculations

If P_o is measured in the P_o tube, the current pressure is measured in the P_o tube when each point is taken, and used to calculate relative pressure for that point:

$$P_{\text{rel}} = \frac{P}{P_o}$$

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

$$\text{For } n \text{ in cm}^3, \text{ STP } R = \frac{P_{\text{STD}}}{T_{\text{STD}}}$$

For n in moles, $R = 8.3145 \text{ J mol}^{-1} \text{ K}^{-1}$

T = temperature

V = volume

z = compressibility factor for the gas at the given pressure and temperature

The real gas equation of state

$$n = \frac{PV}{RTz(P, T)}$$

Quantity Adsorbed Calculations

For the i^{th} dose, the quantity dosed is

$$n(i)_{\text{dosed}, i} = n_{\text{dosed}, i-1} + n(P_1, V_m, T_1) - n(P_2, V_m, T_2)$$

The pressure, volume, and temperature are those of the dosing manifold before and after expanding into the sample tube.

$$n_{\text{ads}, i} = n_{\text{dosed}, i} - n_{\text{fs}, i}$$

The quantity of gas in the free space is

$$n_{\text{fs}, i} = \frac{P_{s, i}}{T_{\text{STD}}} \left(\frac{V_{\text{fc}}}{z(P_{s, i}, T_{\text{bath}})} + \frac{V_{\text{fw}}}{z(P_{s, i}, T_{\text{amb}})} \right)$$

with the real gas equation of state. Here, P_s is the sample pressure.

where:

T_{amb}	=	approximate room temperature (298 K)
T_{bath}	=	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

$$Q_{\text{ads}, i} = \frac{n_{\text{ads}, i}}{m}$$

where m is the sample mass.

Free Space

Measured

Measured free-space volumes are calculated using the following equations:

$$V_{\text{fw}} = \frac{V_{\text{man}}}{T_{\text{man}}} \left(\frac{P_1}{P_2} - 1 \right) T_{\text{STD}}$$

$$V_{\text{fc}} = V_{\text{fw}} \left(\frac{P_2}{P_3} \right)$$

$$V_{\text{bath}} = \frac{V_{\text{fc}} - V_{\text{fw}}}{1 - \frac{T_{\text{bath}}}{T_{\text{amb}}}}$$

where:

P_1	=	system manifold pressure before dosing helium onto sample
P_2	=	system manifold pressure after dosing helium onto sample
P_3	=	sample pressure after raising Dewar and equilibrating with helium
T_{amb}	=	approximate room temperature (298 K)
T_{bath}	=	analysis bath temperature (K)
T_{man}	=	system manifold temperature before dosing helium onto sample (K)
T_{STD}	=	standard temperature (273.15 K)
V_{bath}	=	portion of cold free space at analysis bath temperature
V_{fc}	=	volume of free space, cold (cm ³ at standard temperature)
V_{fw}	=	volume of free space, warm (cm ³ at standard temperature)
V_{man}	=	manifold volume (cm ³)

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.

$$V_{fw} = V_{wb} - V_s \left(\frac{T_{STD}}{T_{amb}} \right)$$

$$V_{fc} = V_{cb} - V_s \left(\frac{T_{STD}}{T_{bath}} \right)$$

where

T_{amb}	=	approximate room temperature (298 K)
T_{bath}	=	analysis bath temperature (K)
T_{STD}	=	standard temperature (273.15 K)
V_{cb}	=	cold free space of the empty tube
V_{fc}	=	calculated cold free space
V_{fw}	=	calculated warm free space
V_s	=	sample mass/density
V_{wb}	=	warm free space of the empty tube

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-Golay¹ 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.

$$P_{\text{avg}} = \frac{-36(P_{11} + P_1) + 9(P_{10} + P_2) + 44(P_9 + P_3) + 69(P_8 + P_4) + 84(P_7 + P_5) + 89(P_6)}{429}$$

$$P_{\text{chg}} = \frac{5(P_{11} - P_1) + 4(P_{10} - P_2) + 3(P_9 - P_3) + 2(P_8 - P_4) + (P_7 - P_5)}{110}$$

$$P_{\text{pcp}, i} = 100\% \frac{P_{\text{chg}}}{P_{\text{avg}}} \quad \text{pressure change per equilibration time interval}$$

where the numerical constants are from the Savitzky-Golay convolution arrays, and

P_{avg}	=	average pressure
P_{chg}	=	change in pressure
$P_{\text{pcp}, i}$	=	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.

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.

$$Y = \left(\frac{P \times SD \times MD^2}{2.33 \times T} \right) 10^3$$

$$\mu = \frac{(1 + G)Y}{(1 + H)Y}$$

$$F = \frac{1}{\alpha Y^2 + \beta Y + \mu}$$

$$P = \left(1 - F \left(1 - \sqrt{\frac{T_{\text{bath}}}{T_{\text{amb}}}} \right) \right)$$

where:

α	=	Weber's coefficient, 0.033
β	=	Weber's coefficient, 0.245
F, Y, μ	=	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 $\frac{T_{\text{bath}} + T}{2}$
T_{amb}	=	room temperature (298 K)
T_{bath}	=	analysis bath temperature (K), from the Po and Temperature Options window

BET Surface Area

For each point designated for surface area calculations, the BET² transformation is calculated as:

$$\frac{1}{N_{ads} \left(\frac{P_o}{P} - 1 \right)}$$

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

- a.) Slope (S g/cm³ STP)
- b.) Y-intercept (Y_{int} g/cm³ STP)
- c.) Error of the slope (S_{err} g/cm³ STP)
- d.) Error of the y-intercept (Y_{err} g/cm³ STP)
- e.) Correlation coefficient

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

BET Surface Area (m²/g):

$$SA_{bet} = \frac{CSA \times N_A}{(22414 \text{ cm}^3 \text{ STP}) \left(\frac{10^{18} \text{ nm}^2}{\text{m}^2} \right) (S + Y_{int})}$$

where

CSA = analysis gas molecular cross-sectional area (nm²), user-entered on the **Adsorptive Properties** window

BET C value:

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

Quality of the Monolayer (cm³/g STP):

$$Q_m = \frac{1}{CY_{int}} = \frac{1}{S + Y_{int}}$$

Error of the BET Surface Area (m²/g):

$$BET_{err} = \frac{SA_{bet}(S_{err}^2 + YI_{err}^2)^{0.5}}{Y_{int} + S}$$

Langmuir Surface Area

For each point designated for surface area calculations, the Langmuir³ transformation is calculated as:

$$L = \frac{P_{rel}}{N_{ads}}$$

where L is in units of g/cm³ 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/cm³ STP)
- b.) Y-intercept (Y_{int} g/cm³ STP)
- c.) Error of the slope (S_{err} g/cm³ STP)
- d.) Error of the y-intercept (YI_{err} g/cm³ STP)
- e.) Correlation coefficient

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

Langmuir Surface Area (m²/g):

$$SA_{Lan} = \frac{CSA \times N_A}{(22414 \text{ cm}^3 \text{ STP}) \left(\frac{10^{18} \text{ nm}^2}{\text{m}^2} \right) S}$$

where

CSA = analysis gas molecular cross-sectional area (nm²), user-entered on the **Adsorptive Properties** window

Quantity of the Monolayer (cm³/g STP):

$$Q_m = \frac{1}{S}$$

Langmuir b Value:

$$b = Y_{\text{int}} V_m$$

Error of the Langmuir Surface Area (m²/g):

$$LAN_{\text{err}} = \frac{SA_{\text{Lan}} S_{\text{err}}}{S}$$

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

$$\log Q = \log Q_m C + \frac{1}{m} \log P$$

Temkin Isotherm

The Temkin isotherm has the form

$$\frac{Q}{Q_m} = \frac{RT}{q_0\alpha} \ln(A_0P)$$

where

A	=	$a_0 \exp \left\{ \frac{-q_0}{RT} \right\}$, where α_0 and A_0 are adjustable constants
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 $8.31441 \times 10^{-3} \frac{\text{kJ}}{\text{molK}}$
T	=	bath temperature (K)

In terms of quantity adsorbed

$$Q = \frac{RTQ_m}{q_0\alpha} \left[\ln A_0 + \ln \left(\frac{P}{P_0} \right) \right]$$

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 $\ln A \frac{RTQ_m}{q_0\alpha}$.

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 ($S \text{ cm}^3/\text{g}\cdot\text{\AA} \text{ STP}$)
- b.) Y-intercept ($Y_{int} \text{ cm}^3/\text{g} \text{ STP}$)
- c.) Error of the slope ($S_{err} \text{ cm}^3/\text{g}\cdot\text{\AA} \text{ STP}$)
- d.) Error of the Y-intercept ($YI_{err} \text{ cm}^3/\text{g} \text{ STP}$)
- e.) Correlation coefficient

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

External Surface Area (m^2/g):

$$\frac{SV_{mol}}{F \times 22414 \text{ cm}^3 \text{ STP}} \times 10^4$$

where

- | | | |
|-----------|---|---|
| 10^4 | = | unit conversions |
| F | = | surface area correction factor, user-entered on the t-Plot Report Options window |
| V_{mol} | = | liquid molar volume, from the fluid property information |

Micropore Surface Area (m^2/g):

$$SA_{\mu p} = SA_{total} + SA_{ext}$$

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 ($\text{cm}^3 \text{ liquid/g}$):

$$\frac{Y_{int} V_{mol}}{22414 \text{ cm}^3 \text{ STP}}$$

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.

$$a_i = \frac{Q_i}{Q_{0.4}}$$

where $Q_{0.4}$ is found by linear interpolation.

A least-squares analysis fit is performed on the $(\alpha_i, Q_{\text{ads},i})$ data pairs where α_i is the independent variable and $Q_{\text{ads},i}$ is the dependent variable. The following are calculated:

- a.) Slope ($S \text{ cm}^3/\text{g STP}$)
- b.) Y-intercept ($Q_0 \text{ cm}^3/\text{g STP}$)
- c.) Error of the slope ($\text{cm}^3/\text{g STP}$)
- d.) Error of the Y-intercept ($\text{cm}^3/\text{g STP}$)
- e.) Correlation coefficient

Surface area is calculated as:

$$A = \frac{A_{\text{ref}} S}{Q_{0.4}}$$

Pore size is calculated as:

$$\frac{Q_0 V_{\text{mol}}}{22414 \text{ cm}^3 \text{ STP}}$$

where V_{mol} is liquid molar volume from the fluid property information.

f-Ratio Method

The f-ratio is the quantity adsorbed divided by the quantity adsorbed in a reference isotherm at the same pressure.

$$f_i = \frac{Q_i}{Q_{\text{ref}} P_i}$$

The reference quantity adsorbed is found by spline interpolation of the reference isotherm.

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.

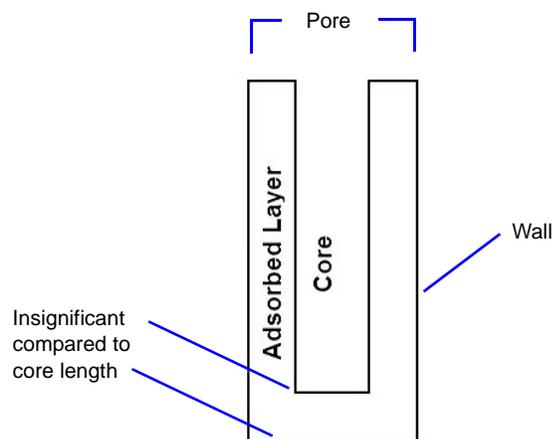
The data used in these calculations must be in order of strictly decreasing numerical value. Points which do not meet this criterion are omitted. The remaining data set is composed of relative pressure (P), quantity adsorbed (Q) pairs from (P_1, Q_1) to (P_n, Q_n) where $(P_n = 0, Q_n = 0)$ is assumed 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 Kelvin⁷ 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 Halenda⁸ 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.



Calculations

The quantities adsorbed (Q_i) are converted to the liquid equivalent volumes (V_i , cm³/g):

$$V_i = \frac{Q_i V_{\text{mol}}}{22414 \text{ cm}^3 \text{ STP}}$$

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 (\AA) 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 Calculations**, page **C-44**.

For the last pressure interval from the lowest Pr_i to zero relative pressure, reference the calculations from the equations in **Thickness Curve Calculations**, page **C-44**.

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:

$$CSA_j = \pi[(Rc_j + \Delta Tw)^2 - Rc_j^2] \left(10^{-16} \frac{\text{cm}^2}{\text{\AA}^2} \right)$$

The total volume of gas desorbed from walls of previously opened pores is calculated:

$$Vd_i = \sum_j (LP_j)(CSA_j) \quad \text{for all previously opened pores}$$

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 ($VI_i - VI_{i+1}$), desorption from walls only is occurring. Total surface of walls exposed thus far (cm^2/g) is calculated as:

$$SA_w = \sum_j \pi(LP_j)(D_{\text{avg},j})\left(\frac{10^{-8} \text{ cm}}{\text{\AA}}\right) \text{ for all previously opened pores}$$

where

$D_{\text{avg},j}$ = 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:

$$\Delta Tw = \frac{(VI_i - VI_{i+1})\left(10^8 \frac{\text{\AA}}{\text{cm}}\right)}{SAw_i}$$

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 ($VI_i - VI_{i+1}$), 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:

$$D_{\text{avg},k} = \frac{2(Rc_k + Rc_{k+1})(Rc_k)(Rc_{k+1})}{Rc_k^2 + Rc_{k+1}^2}$$

$D_{\text{avg},k}$ 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 $D_{\text{avg},k}$ is calculated as:

$$P_{\text{avg},k} = \ln^{-1} \left[\frac{-2A}{(1+F)(D_{\text{avg},k})} \right]$$

The thickness of the adsorbed layer at this pressure is calculated as:

$$Tw_{\text{avg},k} = HP1 \left[\frac{HP2}{\ln(P_{\text{avg},k})} \right]^{HP3}$$

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:

$$\Delta Td = Tw_{\text{avg},k} - Tw_{i+1}$$

The cross-sectional area of the newly opened pores is calculated as:

$$CSAc_k = \left[\frac{D_{\text{avg},k}}{2} + \Delta Td \right]^2 \left(\frac{10^{-16} \text{ cm}^2}{\text{\AA}^2} \right)$$

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:

$$D_{\text{avg},k,\text{new}} = D_{\text{avg},k,\text{old}} + 2(\Delta Td)$$

The layer thickness change during the whole interval is added to diameters of previously opened pores as:

$$D_{\text{avg},k,\text{new}} = D_{\text{avg},k,\text{old}} + 2(\Delta Tdw)$$

(not including) $D_{\text{avg},k}$)

The layer thickness change desorbed during this interval also is added to the radii corresponding to the ends of the pressure intervals as:

$$Rc_{j,\text{new}} = Rc_{j,\text{old}} + \Delta Tw$$

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:

$$Dp_j = 2(Rc_j)$$

for all Rc_j including Rc_{k+1} .

The remaining calculations are based on Dp_i , $D_{\text{avg},i}$, and LP_i . These calculations are only done for $D_{\text{avg},i}$ 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):

$$Vp_i = \pi(LP_i) \left[\frac{D_{\text{avg},i}}{2} \right]^2 \left[\frac{10^{16} \text{cm}^2}{\text{\AA}^2} \right]$$

(2.) Cumulative Pore Volume ($VP_{\text{cum},i}$, cm^3/g):

$$VP_{\text{cum},i} = \sum_j Vp_j \text{ for } (J \leq i)$$

(3.) Incremental Surface Area (SA_i , m²/g):

$$SA_i = \pi(LP_i) \left(\frac{10^{-2} \text{ m}}{\text{cm}} \right) (D_{\text{avg}, i}) \left(\frac{10^{-10} \text{ m}}{\text{Å}} \right)$$

(4.) Cumulative Surface Area ($SA_{\text{cum}, i}$, m²/g):

$$SA_{\text{cum}, 10} = \sum SA_j \text{ for } J \leq 1$$

(5.) dV/dD pore volume (dV/dD_i , cm³/g-Å):

$$\frac{dV}{dD_i} = \frac{VP_i}{Dp_i - Dp_{i+1}}$$

(6.) $dV/d\log(D)$ pore volume ($dV/d\log(D)_i$, cm³/g):

$$\frac{dDv}{d\log D_i} = \frac{VP_i}{\log\left(\frac{Dp_i}{Dp_{i+1}}\right)}$$

(7.) dA/dD pore area (dA/dD_i , m²/g-Å):

$$\frac{dA}{dD_i} = \frac{SA_i}{Dp_i - Dp_{i+1}}$$

(8.) $dA/d\log(D)$ pore area [$dA/d\log(D)_i$, m²/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 ($DF_{\text{avg},j}$, A):

$$DF_{\text{avg},j} = \frac{Dp_{F_j} + Dp_{F_{j+1}}}{2}$$

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 ($VpF_{\text{cum},i}$, cm^3/g):

$$VpF_{\text{cum},i} = \text{INTERP}(Dp_{F_{i+1}})$$

where $\text{INTERP}(x)$ is the value interpolated from the function $X = Dp_{j+i}$ and $Y = VP_{\text{cum},i}$, using an AKIMA semi-spline interpolation.

- (3.) Incremental Pore Volume (VpF_i , cm^3/g):

$$VpF_i = VpF_{\text{cum},i} - VpF_{\text{cum}_{i-1}}$$

where $VpF_{\text{cum},0} = 0$.

- (4.) Cumulative Surface Area ($SAF_{\text{cum},i}$, m^2/g):

$$SAF_{\text{cum},i} = \text{INTERP}(Dp_{F_{i+1}})$$

where $\text{INTERP}(x)$ is the value interpolated from the function $X = Dp_{j+i}$ and $Y = SA_{\text{cum},j}$.

- (5.) Incremental Surface Area (SAF_i , m^2/g):

$$SAF_i = SAF_{\text{cum},i} - SAF_{\text{cum}_{i-1}}$$

where $SAF_{\text{cum},0} = 0$.

(6.) dV/dD pore volume ($dV/dDpF_i$, $\text{cm}^3/\text{g-A}$):

$$\frac{dV}{dDpF_i} = \text{INTERP}(DpF_{i+1})$$

where $\text{INTERP}(x)$ is the value interpolated from the function $X = D_{\text{avg},j}$ and $Y = dV/dD_j$.

(7.) $dV/d\log(D)$ pore volume [$dV/d\log(DpF_i)$, cm^3/g]:

$$\frac{dV}{d\log(DpF_i)} = \text{INTERP}(DpF_{i+1})$$

where $\text{INTERP}(x)$ is the value interpolated from the function $X = D_{\text{avg},j}$ and $Y = dV/d\log(D)_j$.

(8.) dA/dD pore area ($dA/dDpF_i$, $\text{m}^2/\text{g-A}$):

$$\frac{dA}{dDpF_i} = \text{INTERP}(DpF_{i+1})$$

where $\text{INTERP}(x)$ is the value interpolated from the function $X = D_{\text{avg},j}$ and $Y = dA/dD_j$.

(9.) $dA/d\log(D)$ pore area [$dA/d\log(DpF_i)$, m^2/g]:

$$\frac{dA}{d\log(DpF_i)} = \text{INTERP}(DpF_{i+1})$$

where $\text{INTERP}(x)$ is the value interpolated from the function $X = D_{\text{avg},j}$ and $Y = dA/d\log(D)_j$.

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

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:

$$D_i = 2r_k(P_i) + t(P_i)$$

The average pore diameter is the arithmetic mean of the diameters that bound the interval.

$$\bar{D}_i = \left(\frac{D_i + D_{i+1}}{2} \right)$$

Pore Length

$$l_i = \frac{A_{p,i} + 10^8}{\pi \bar{D}_i}$$

$$A_{p,i} = \frac{4 \times (10^8 \Delta V_p)}{\bar{D}_i}$$

$$\Delta V_p = C_v(D(Q_{i-1} - Q_i) - \Delta t \times 10^8 (A_{p,cum} - 2\pi \bar{t} l_{i,cum}))$$

$$C_v = \left(\frac{\bar{D}_i}{2(\bar{r}_k + t(P_i) - t(P_{i+1}))} \right)^2$$

$$\bar{t} = \frac{\bar{D}_i}{2 - \bar{r}_k}$$

$$\bar{r}_k = \frac{(r_{k,i} + r_{k,i+1})}{2}$$

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
C_v	=	Volume correction factor
D	=	Density conversion factor
\bar{r}_k	=	Average Kelvin radius
\bar{t}	=	Average thickness

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 HK)

When you use the original Horvath-Kawazoe⁹ 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 \AA^4
σ	=	gas solid nuclear separation at zero interaction energy (\AA), $\frac{Z_S + Z_A}{2}$
d_0	=	$\frac{D_A + D_s}{2}$

where:

D_A	=	molecular diameter (\AA) from the Horvath-Kawazoe Physical Properties window
D_s	=	diameter of sample atom (\AA) 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_o	=	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 Po 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 you use the Saito-Foley¹⁰ 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.

$$\ln\left(\frac{P}{P_o}\right) = \frac{3\pi K}{4RT} \times \frac{IP \times 10^{32}}{d_0^4} \times \sum_{k=0}^{\infty} \left[\frac{1}{k+1} \left(1 - \frac{d_0}{r_p}\right)^{2k} \times \left\{ \frac{21}{32} \alpha_k \left(\frac{d_0}{r_p}\right)^{10} - \beta_k \left(\frac{d_0}{r_p}\right)^4 \right\} \right]$$

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	=	$\left(\frac{-4.5-k}{k}\right)^2 \alpha_{k-1}, \alpha_0 = 1.0$
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)
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 you use the Cheng/Yang¹¹ 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.

$$\ln \left(\frac{P}{P_o} \right) = \frac{6N_1 \varepsilon_{12}^* + N_2 \varepsilon_{22}^* L^3 \times 10^{32}}{RTL - d_0^3} \left[\left(\frac{d_0}{L} \right)^6 \left(\frac{T_1}{12} + \frac{T_2}{8} \right) + \left(\frac{d_0}{L} \right)^{12} \left(\frac{T_3}{90} + \frac{T_4}{80} \right) \right]$$

where

$$10^{32} = \text{the number of cm}^4 \text{ that are equal to } \text{\AA}^4$$

$$\varepsilon_{12}^* = \frac{\text{\AA}_S}{4d_S^6}, \text{ where } \text{\AA}_S = \frac{6(mc^2)\alpha_S\alpha_A}{\frac{\alpha_S}{\chi_S} + \frac{\alpha_A}{\chi_A}}$$

$$\varepsilon_{22}^* = \frac{A_A}{4D_A^6}, \text{ where } \text{\AA}_A = \frac{3(mc^2)(\alpha_A)(\chi_A)}{2}$$

$$d_0 = \frac{D_A + D_S}{2}$$

where:

$$D_A = \text{molecular diameter (\AA) from the **Horvath-Kawazoe Physical Properties** window}$$

$$D_S = \text{diameter of sample atom (\AA) from the **Horvath-Kawazoe Physical Properties** window}$$

$$L = \text{pore width (nucleus to nucleus) (\AA)}$$

$$N_1 = 4\pi L^2 N_S, \text{ where } N_S = \text{number of sample atoms/cm}^2 \text{ at monolayer}$$

$$N_2 = 4\pi (L - d_0)^2 N_A, \text{ where } N_S = \text{number of gas molecules/cm}^2$$

$$P = \text{equilibrium pressure}$$

$$P_o = \text{saturation pressure}$$

$$R = \text{gas constant } (8.31441 \times 10^7 \text{ erg/mol } K)$$

$$T = \text{analysis bath temperature (K), from an entered or calculated value on the **Po and Temperature Options** window}$$

$$\begin{aligned}
 T_1 &= \frac{1}{(1-S)^3} - \frac{1}{(1+S)^3} \\
 T_2 &= \frac{1}{(1+S)^2} - \frac{1}{(1-S)^2} \\
 T_3 &= \frac{1}{(1-S)^9} - \frac{1}{(1+S)^9} \\
 T_4 &= \frac{1}{(1+S)^8} - \frac{1}{(1-S)^8} \\
 \text{where } S &= \frac{L-d_0}{L}
 \end{aligned}$$

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

$$\begin{aligned}
 G(L) &= \text{one of the Horvath-Kawazoe equations given above} \\
 \theta &= \text{degree of void filling; } \theta \text{ is estimated by first computing the monolayer capacity } (Q_m) \text{ with the Langmuir equation over the range of data points from relative pressure } 0.02 \text{ to } 0.2 \text{ or the maximum relative pressure included in the Horvath-Kawazoe analysis. } \theta \text{ is computed as the quantity adsorbed over } Q_m.
 \end{aligned}$$

Interaction Parameter

The interaction parameter (IP) results from the following calculations:

The Kirkwood-Muller dispersion coefficients

$$A_S = \frac{6mc^2 a_S a_A}{\frac{\alpha_S}{\chi_S} + \frac{\alpha_A}{\chi_A}}$$

$$A_A = \frac{3mc^2 \alpha_A \chi_A}{2}$$

where

- α_A = polarizability of gas molecule (cm³)
- α_S = polarizability of sample atoms (cm³)
- mc^2 = kinetic energy of electron (0.8183 × 10⁻⁶ erg)
- χ_A = diamagnetic susceptibility of gas molecule (cm³)

$$IP = (N_A A_A) + (N_S A_S)$$

where:

- N_A = number of gas molecules/cm² at monolayer from the **Horvath-Kawazoe Physical Properties** window
- N_S = number of sample atoms/cm² from the **Horvath-Kawazoe Physical Properties** window
- χ_S = diamagnetic susceptibility of sample atom (cm³)

Refer to [Interaction Parameter Components](#), page **C-32** for recommended values.

Additional Calculations

Based on the previous calculations, the following can be calculated:

Adjusted Pore Width (Å):
(Shell to Shell)

$$AL_i = L_i - Ds$$

Cumulative Pore Volume (cm³/g):

$$V_{\text{cum},i} = \frac{Q_i V_{\text{mol}}}{22414 \text{cm}^3 \text{STP}}$$

where

$$V_{\text{mol}} = \text{liquid molar volume from the fluid property information}$$

dV/dD Pore Volume (cm³/g-Å):

$$\frac{dV}{dD_i} = \frac{V_{\text{cum},i} - V_{\text{cum},i-1}}{AL_i - AL_{i-1}}$$

Median Pore Width (Å):

$$V_{\text{half}} = \frac{V_{\text{cum},n}}{2}$$

$$D_{\text{med}} = 10 \left[\log(D_1) + [\log(V_{\text{half}}) - \log(V_1)] \times \frac{\log(D_g) - \log(D_1)}{\log(V_g) - \log(V_1)} \right]$$

where

- D_1 = pore width (L_i) that corresponds to V_1
- D_g = pore width (L_i) that corresponds to V_g
- $V_{\text{cum},n}$ = total cumulative pore volume ($V_{\text{cum},i}$) for points designated for Horvath-Kawazoe calculations
- V_g = cumulative pore volume ($V_{\text{cum},i}$) for first point greater than V_{half}
- V_{half} = 50% of total cumulative pore volume
- V_1 = cumulative pore volume ($V_{\text{cum},i}$) for first point less than V_{half}

Interaction Parameter Components

Table C-1. Interaction Parameters

Gas	Bath Temperature (K)	Sample Type	Interaction Parameter Calculated Value*
Argon	87.3	Carbon (Ross/Olivier value)	2.61
		Carbon (Horvath/Kawazoe value)	5.89
		Zeolite	3.19
Carbon Dioxide	298.15	Carbon (Ross/Olivier value)	4.20
		Carbon (Horvath/Kawazoe value)	9.20
		Zeolite	5.08
	273.15	Carbon (Ross/Olivier value)	4.34
		Carbon (Horvath/Kawazoe value)	9.35
		Zeolite	5.22
	194.65	Carbon (Ross/Olivier value)	4.72
		Carbon (Horvath/Kawazoe value)	9.72
		Zeolite	5.60
Nitrogen	77.15	Carbon (Ross/Olivier value)	2.84
		Carbon (Horvath/Kawazoe value)	6.53
		Zeolite	3.49
<p>* The interaction parameter is entered in the Horvath-Kawazoe Report Options window in the following field:</p> <p>Interaction parameter: (calculated value) $\times 10^{-43}$ erg-cm⁴</p>			

The following values were used to calculate the values in Table C-1.

Carbon-Graphite

$$D_S = 3.40$$

$$N_S = 3.845 \times 10^{15}$$

$$\chi_S = 1.05 \times 10^{-29} \text{ (Ross/Olivier)}$$

$$13.5 \times 10^{-29} \text{ (Horvath/Kawazoe, implicit)}$$

$$\alpha_S = 1.02 \times 10^{-24}$$

Zeolite

$$D_S = 3.04$$

$$N_S = 3.75 \times 10^{15}$$

$$\chi_S = 1.94 \times 10^{-29}$$

$$\alpha_S = 0.85 \times 10^{-24}$$

Nitrogen

$$D_A = 3.00$$

$$N_A = 6.71 \times 10^{14}$$

$$\chi_A = 3.6 \times 10^{-29}$$

$$\alpha_A = 1.76 \times 10^{-24}$$

Argon

$$D_A = 2.95$$

$$N_A = 7.608 \times 10^{14}$$

$$\chi_A = 3.22 \times 10^{-29}$$

$$\alpha_A = 1.63 \times 10^{-24}$$

Carbon Dioxide

$$D_A = 3.23$$

$$N_A = 4.567 \times 10^{14} \text{ (25 °C)}$$

$$5.45 \times 10^{14} \text{ (0 °C)}$$

$$7.697 \times 10^{14} \text{ (-78 °C)}$$

$$\chi_A = 5.0 \times 10^{-29}$$

$$\alpha_A = 2.7 \times 10^{-24}$$

D_A values are from van der Waal's constant.

N_A values are from liquid densities.

χ and α values are derived from data found in Ross and Olivier¹².

The physical parameters referenced in Saito/Foley are:

Aluminophosphate

$$D_S = 2.60$$

$$N_S = 1.48 \times 10^{15}$$

$$\chi_S = 1.3 \times 10^{-29}$$

$$\alpha_S = 2.5 \times 10^{-24}$$

Aluminosilicate

$$D_S = 2.76$$

$$N_S = 1.31 \times 10^{15}$$

$$\chi_S = 1.3 \times 10^{-29}$$

$$\alpha_S = 2.5 \times 10^{-24}$$

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 Olivier¹² 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:

$$Q(p) = \int da db dc \dots q(p, a, b, c \dots) f(a, b, c \dots) \quad (1)$$

where

$Q(p)$	=	the total quantity adsorbed per unit weight at pressure p ,
a, b, c, \dots	=	a set of distributed properties,
$f(a, b, c, \dots)$	=	the distribution function of the properties, and
$q(p, a, b, c, \dots)$	=	the kernel function describing the adsorption isotherm on unit surface of material with fixed properties a, b, c, \dots

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:

$$Q(p) = \int d\varepsilon q(p, \varepsilon) f(\varepsilon) \quad (2)$$

where

$$\begin{aligned} Q(p) &= \text{the experimental quantity adsorbed per gram at pressure } p, \\ q(p, \varepsilon) &= \text{the quantity adsorbed per unit area at the same pressure, } p, \text{ on an ideal free} \\ &\quad \text{surface of energy } \varepsilon, \text{ and} \\ f(\varepsilon) &= \text{the total area of surface of energy } \varepsilon \text{ in the sample.} \end{aligned}$$

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

$$Q(p) = \int dH q(p, H) f(H) \quad (3)$$

where

$$\begin{aligned} Q(p) &= \text{the experimental quantity adsorbed at pressure } p, \\ q(p, H) &= \text{the quantity adsorbed per unit area at the same pressure, } p, \text{ in an ideal pore of} \\ &\quad \text{size } H, \text{ and} \\ f(H) &= \text{the total area of pores of size } H \text{ in the sample.} \end{aligned}$$

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:

$$Q(p) = \sum_i q(p, Z_i) f(Z_i) \quad (4)$$

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.

Dubinin-Radushkevich

The Dubinin-Radushkevich¹³ equation is:

$$\log(Q) = \log(Q_0) - \frac{BT^2}{\beta} \times \left[\log \frac{P_0}{P} \right]^2$$

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, $\log(V_0)$ 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, V_0 is the monolayer capacity. Based on this assumption, the following are calculated:

- Slope (S cm³/g STP)
- Y-intercept (YI cm³/g STP)
- Error of the slope (S_{err} cm³/g STP)
- Error of the y-intercept (YI_{err} cm³/g STP)
- Correlation coefficient

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

Monolayer Capacity (cm³/g STP):

$$Q_0 = 10^{YI}$$

Error of Monolayer Capacity (cm³/g STP):

$$Q_{0, \text{err}} = Q_0(10^{YI, \text{err}} - 1.0)$$

Micropore surface area (m²/g):

$$SDP = \frac{\sigma Q_0 N_A}{22414 \text{ cm}^3 \left(\frac{10^{18} \text{ nm}^2}{\text{m}^2} \right)}$$

where

σ = molecular cross sectional area of gas (nm²) from the **Adsorptive Properties** window

Dubinin-Astakhov

The Dubinin-Astakhov equation is:

$$\log(Q) = \log(Q_0) - \left[\frac{RT}{\beta E_0} \right]^N \times \left[\log \frac{P_0}{P} \right]^N$$

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 (cm³/g STP)
 Q_0 = the micropore capacity (cm³/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:

$$LV = \log(Q)$$

$$LP = \left[\log \frac{P_0}{P} \right]^N$$

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 ($S \text{ cm}^3/\text{g STP}$)
- b.) Y-intercept ($YI \text{ cm}^3/\text{g STP}$)
- c.) Error of the slope ($S_{\text{err}} \text{ cm}^3/\text{g STP}$)
- d.) Error of the y-intercept ($YI_{\text{err}} \text{ cm}^3/\text{g STP}$)
- e.) Correlation coefficient
- f.) Optimized Astakhov exponent (N)

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

Monolayer Capacity ($\text{cm}^3/\text{g STP}$):

$$Q_0 = 10^{YI}$$

Micropore Volume (cm^3/g):

$$V_i = \frac{Q_i V_{\text{mol}}}{22414}$$

where

V_{mol} = liquid molar volume conversion factor from the fluid property information

Limiting Micropore Volume (cm³/g):

$$V_0 = \frac{Q_o V_{\text{mol}}}{22414 \text{ cm}^3 \text{ STP}}$$

where

$$V_{\text{mol}} = \text{liquid molar volume from the fluid property information}$$

Error of Limiting Micropore Volume (cm³/g):

$$V_{0, \text{err}} = W_0(10 Y I_{\text{err}} - 1.0)$$

Characteristic Energy (KJ/mol):

$$E = \frac{2.303(RT)}{\beta(2.303 \times S)^{1/N}}$$

Modal Equivalent Pore Diameter (Å):

$$D_{\text{mode}} = 2 \left\{ \left[\frac{3N}{3N+1} \right]^{1/N} \times \left[\frac{10^3 \text{ nm}^3 / \text{Å}^3}{\beta \cdot E_o} \right] \right\}^{1/3}$$

where

$$\beta = \text{affinity coefficient of the analysis gas relative to the Po gas from the **Dubinin Adsorptive Options** window}$$

Maximum Differential Pore Volume (cm³/g-Å):

This value is also known as *frequency of the mode*¹².

$$\frac{dV}{dD_{\text{mode}}} \text{Max} = 0.5(3N+1)W_o \left[\frac{3N+1}{3N} \right]^{1/3N} \left[\frac{\beta \cdot E_o}{((10^3 \text{ nm}^3) / \text{Å}^3)} \right]^{1/3} \exp\left(-\left[\frac{3N+1}{3N} \right]\right)$$

Mean Equivalent Pore Width (Å):

$$D_{\text{mean}} = 2 \times \frac{\left[\frac{(10^3 \text{ nm}^3) / \text{Å}^3}{\beta \cdot E_o} \right]^{1/3}}{\Gamma\left(\frac{3N+1}{3N}\right)}$$

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 $0 \leq x \leq 1$:

$$\Gamma(x+1) = 1 + b_1x + b_2x^2 + b_3x^3 + b_4x^4 + b_5x^5 + b_6x^6 + b_7x^7 + b_8x^8 + \varepsilon x|\varepsilon x| \leq 3(10^{-7})$$

where

$$\begin{aligned} b_1 &= -0.57719\ 1652 \\ b_2 &= 0.98820\ 5891 \\ b_3 &= -0.89705\ 6937 \\ b_4 &= 0.91820\ 6857 \\ b_5 &= -0.75670\ 4078 \\ b_6 &= 0.48219\ 9394 \\ b_7 &= -0.19352\ 7818 \\ b_8 &= 0.03586\ 8343 \end{aligned}$$

and where

$$x+1 = \left(\frac{3N+1}{3N} \right)$$

Equivalent Pore Diameter (Å):

$$D_i = 2 \left[\frac{-\left(\frac{10^3 \text{ nm}^3 / \text{Å}^3}{\beta \cdot E_0} \right)^N}{\ln(W_i) - \ln(W_0)} \right]^{1/3N}$$

dV/dD Pore Volume (cm³/g-Å):

$$\frac{dV}{dD_i} = 0.5 \times W_0 \times 3N \left(\frac{10^3 \text{ nm}^3 / \text{Å}^3}{\beta \cdot E_0} \right)^N \left(\frac{D_i}{2} \right)^{-(3N+1)} \times \exp \left[-\left(\frac{10^3 \text{ nm}^3 / \text{Å}^3}{\beta \cdot E_0} \right)^N \left(\frac{D_i}{2} \right)^{-3N} \right]$$

MP-Method

With the (t_p, Q_i) data pairs, 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 (\AA):

$$R_i = \frac{t_i + t_{i-1}}{2}$$

Remaining pore surface area for the i^{th} point (m^2/g):

$$S_i = \frac{Q_i - Q_{i-1}}{t_i - t_{i-1}} \frac{V_{\text{mol}}}{22414 \text{cm}^3 \text{STP}} \times 10^4$$

where

$$\begin{aligned} 10^4 &= \text{unit conversions} \\ V_{\text{mol}} &= \text{liquid molar volume from the fluid property information} \end{aligned}$$

Incremental pore surface area occluded for the i^{th} point (m^2/g):

$$S_{\text{inc}, i} = S_{i-1} - S_i$$

Cumulative pore surface area occluded for the i^{th} point (m^2/g):

$$S_{\text{cum}_i} = S_{\text{inc}, i} + S_{\text{inc}, i-1} + \dots + S_{\text{inc}, 1}$$

dA/dR pore surface area for the i^{th} point ($\text{m}^2/\text{g}\text{-\AA}$):

$$\frac{dA}{dR_i} = \frac{S_{\text{inc},i}}{t_i - t_{i-1}}$$

Incremental pore volume occluded for the i^{th} point (cm^3/g):

$$V_{\text{inc},i} = S_{\text{inc},i} R_i \times 10^{-4}$$

Cumulative pore volume occluded for the i^{th} point (cm^3/g):

$$V_{\text{cum},i} = V_{\text{inc},i} + V_{\text{inc},i-1} + \dots + V_{\text{inc},1}$$

dV/dR pore volume for the i^{th} point ($\text{cm}^3/\text{g}\text{-\AA}$):

$$\frac{dV}{dR_i} = \frac{V_{\text{inc},i}}{t_i - t_{i-1}}$$

Thickness Curve Calculations

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/p^0	=	relative pressure for the i^{th} point
t_i	=	thickness for i^{th} point

Reference

Interpolated from table.

Kruk-Jaroniec-Sayari

$$t = \left(\frac{C_1}{C_2 - \log(p/p^0)} \right)^{C_3}$$

Halsey

$$t = C_1 \left(\frac{C_2}{\ln(p/p^0)} \right)^{C_3} \quad (\text{Halsey}^5)$$

Harkins and Jura

$$t = \left(\frac{C_1}{C_2 - \log(p/p^0)} \right)^{C_3} \quad (\text{Harkins and Jura}^6)$$

Broekoff-de Boer

$$\log((p/p^0)) = \frac{C_1}{t^2} + C_2 C_3^t$$

Carbon Black STSA

$$t = C_1 + C_2(p/p^0) + C_3(p/p^0)^2$$

SPC Report Variables

Regression Chart Variables

The line of best fit for the Regression Chart is calculated by the usual Least Squares method. (Refer to *BASIC Scientific Subroutines Vol II*, by F.R. Ruckdeschel, Copyright 1981 BYTE Publications/McGraw Hill, p. 16.) 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.

$$\bar{X} = \frac{\sum x_i}{N}$$

$$\bar{Y} = \frac{\sum y_i}{N}$$

$$\text{Slope} = \frac{\sum (x_i - \bar{X})(y_i - \bar{Y})}{\sum (x_i - \bar{X})^2}$$

The coefficient of Correlation for this line is also calculated in the usual way. (Refer to *Mathematical Handbook for Scientists and Engineers*, by Granino A. Korn and Theresa M. Korn, Copyright 1961, 1968 McGraw Hill, Sec. 18.4.)

$$\sigma_x = \sqrt{\frac{\sum (x_i - \bar{X})^2}{N}}$$

$$\sigma_y = \sqrt{\frac{\sum (y_i - \bar{Y})^2}{N}}$$

$$\text{Cov}(x, y) = \frac{\sum (x_i - \bar{X})(y_i - \bar{Y})}{N}$$

$$\text{Correlation Coefficient} = \frac{\text{Cov}(x, y)}{\sigma_x \sigma_y}$$

Control Chart Variables

$$\text{Mean} = \frac{\sum y_i}{N}$$

$$\text{StdDev} = \sqrt{\frac{\sum (y - \text{Mean})^2}{N - 1}}$$

$$\text{CoefVar} = \frac{\text{StdDev}}{\text{Mean}}$$

$$\text{PlusNSig} = \text{Mean} + n \cdot \text{StdDev}$$

$$\text{MinusNSig} = \text{Mean} - n \cdot \text{StdDev}$$

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 (m²/g)

$$S_{1PT} = \frac{[Q(1 - P)] \times CSA(N_A)}{22414 \text{ cm}^3 \text{ STP} \left(\frac{10^{18} \text{ nm}^2}{\text{m}^2} \right)}$$

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. Refer to [BET Surface Area](#), page [C-8](#).
- c.) Langmuir Surface Area. Refer to [Langmuir Surface Area](#), page [C-9](#).
- d.) t-Plot Micropore Surface Area. Refer to [t-Plot](#), page [C-12](#).

- e.) t-Plot External Surface Area. Refer to **t-Plot**, page **C-12**.
- f.) BJH Cumulative Adsorption
- g.) BJH Cumulative Desorption
- h.) Adsorption Total Pore Volume
- i.) Desorption Total Pore Volume
- j.) t-Plot Micropore Pore Volume. Refer to **t-Plot**, page **C-12**.
- k.) Freundlich. Refer to **Freundlich Isotherm**, page **C-10**.
- l.) Temkin. Refer to **Temkin Isotherm**, page **C-11**
- m.) Alpha-S. Refer to **Alpha-S Method**, page **C-13**.
- n.) DFT Pore Size and DFT Surface Energy. Refer to **DFT (Density Functional Theory)**, page **C-34**.
- 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. Refer to **Dubinin-Astakhov**, page **C-38**.

- q.) Dubinin-Astakhov Micropore Volume. Refer to [Dubinin-Astakhov](#), page [C-38](#).
- r.) Dubinin-Radushkevich Micropore Surface Area. Refer to [Dubinin-Radushkevich](#), page [C-37](#).
- s.) Dubinin-Radushkevich Monolayer Capacity. Refer to [Dubinin-Radushkevich](#), page [C-37](#).

- t.) MP-Method Cumulative Surface Area of Pores

$S_{\text{total}} = S_{\text{cum},i}$, (see MP-method Calculations) 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

$V_{\text{total}} = V_{\text{cum},i}$, (see MP-method calculations) 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)

$$\bar{r} = \frac{V_{\text{total}}}{S_{\text{total}}} \times 10^4$$

- w.) Horvath-Kawazoe. Refer to [Horvath-Kawazoe](#), page [C-26](#).

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D. 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 LN₂ 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 system 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 requires the use of helium
- Analysis speed is a major factor; a helium free space measurement of 10 to 15 minutes is required
- Your 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. You must 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 Po and Temperature dialog)
- Enter the sample mass and density (found on the Sample Information dialog)

Enter

This method allows you to enter 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.

E. MAINTAINING HIGH PURITY GASES

The 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 you 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

You should 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 bottle, air is unavoidably trapped on the high- and low-pressure sides of the regulator, as well as in the gas lines. You should remove as much of this air as is possible *before* opening the gas bottle 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 bottle 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

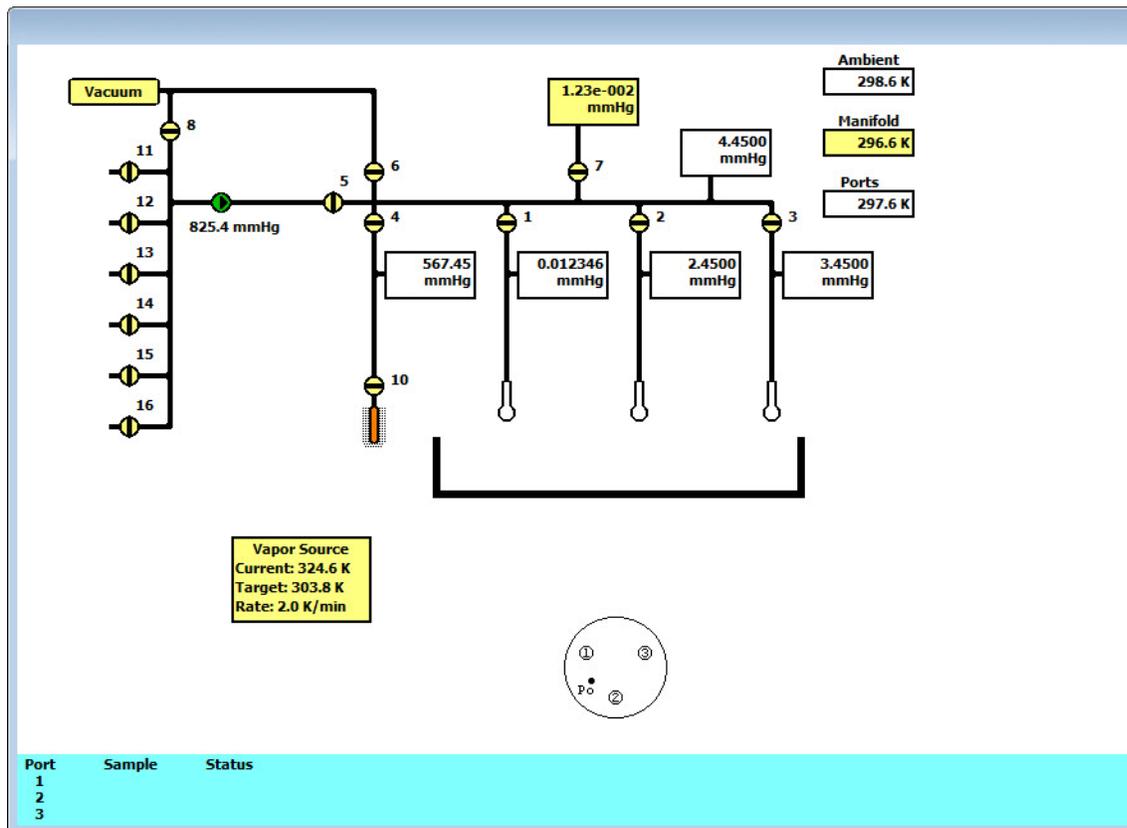
This is the preferred method for removing trapped air.

1. Go to **Unit > Enable Manual Control** (if the instrument schematic is not displayed, select **Show Instrument Schematic**).



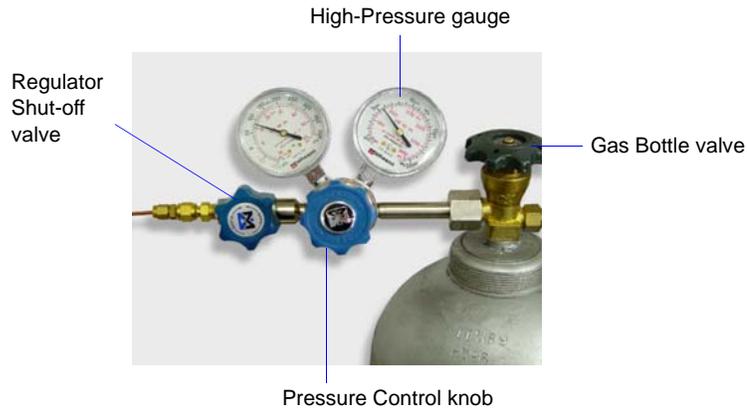
If multiple instruments are installed, make sure to choose the correct Unit menu.

2. Close all valves by right clicking on each valve and selecting **Close**.



3. Open the regulator Shut-off valve.

- Open the gas bottle 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.



- Using the Pressure Control knob, set the output pressure (gas bottle pressure gauge) to 15 psig.
- Loosen the fitting at the helium inlet (on the rear panel of the instrument) until the low pressure side drops to approximately 3 psig (0.02 MPa), then tighten the fitting.
- Repeat steps 4, 5, and 6 three times.
- Briefly open the gas bottle valve; then, using the Pressure Control knob, reset the regulator output pressure to 15 psig.
- After the pressure has stabilized (indicating there are no leaks), open the gas bottle valve.

Evacuation Method



To use this method, the gas tank must be within 10 feet of the instrument.

- 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 bottle valve.
	Open the regulator Shut-off valve.

If...	Then... <i>(continued)</i>
The regulator is filled with gas:	Close the gas bottle valve.
	Open the regulator Shut-off 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).

- Go to **Unit > Enable manual control** (if the instrument schematic is not displayed, select **Show instrument schematic**).



If multiple instruments are installed, make sure to choose the correct Unit menu.

- Close all valves; then open valves 6, 7, and 10.
- Allow evacuation to continue for 20 minutes. This pulls a vacuum on the helium line to the gas bottle. The manifold pressure transducer should fall close to zero.



Be sure to allow evacuation for a full 20 minutes. If evacuation time is too short, trapped air may remain in the lines.

- Close valves 6, 7, and 10.

F. 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:

$$U(s) = 4\varepsilon \left[\left(\frac{\sigma}{s} \right)^{12} - \left(\frac{\sigma}{s} \right)^6 \right]$$

where

- $\varepsilon =$ a characteristic energy of the adsorptive,
- $\sigma =$ 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* 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.

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 **References** at the end of this appendix.

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

Figure D-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.

*.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).

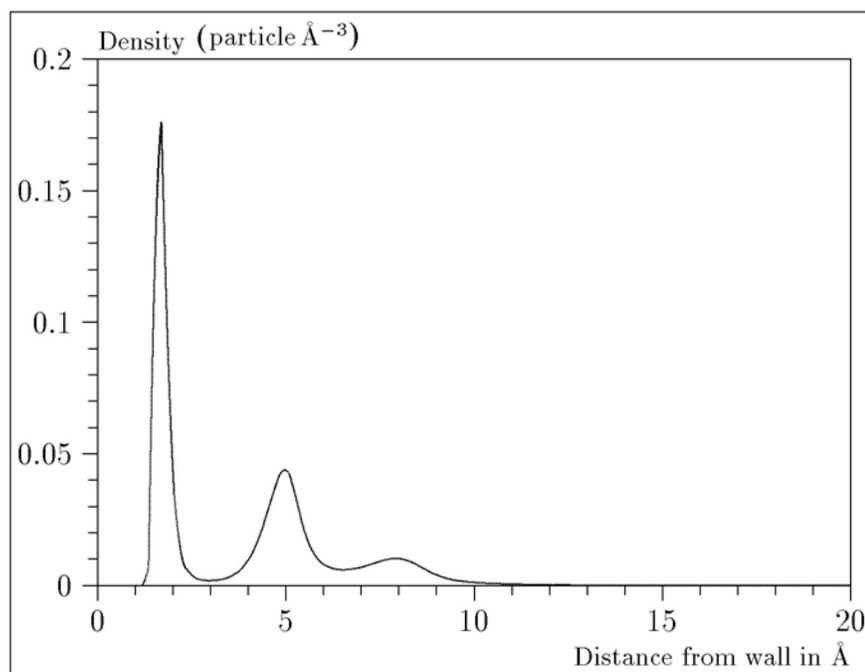


Figure D-1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

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 D-2 shows how the profiles change with pressure for a model pore with $H = 40 \text{ \AA}$. The insets show the density profiles for the corresponding points of the isotherm.

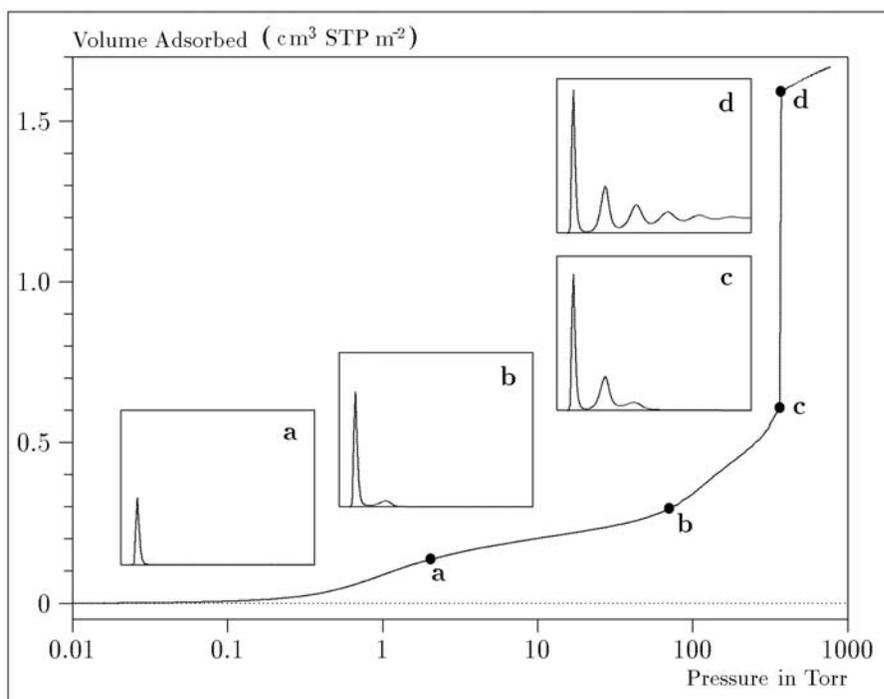


Figure D-2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

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 Figure D-2 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 Figure D-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.

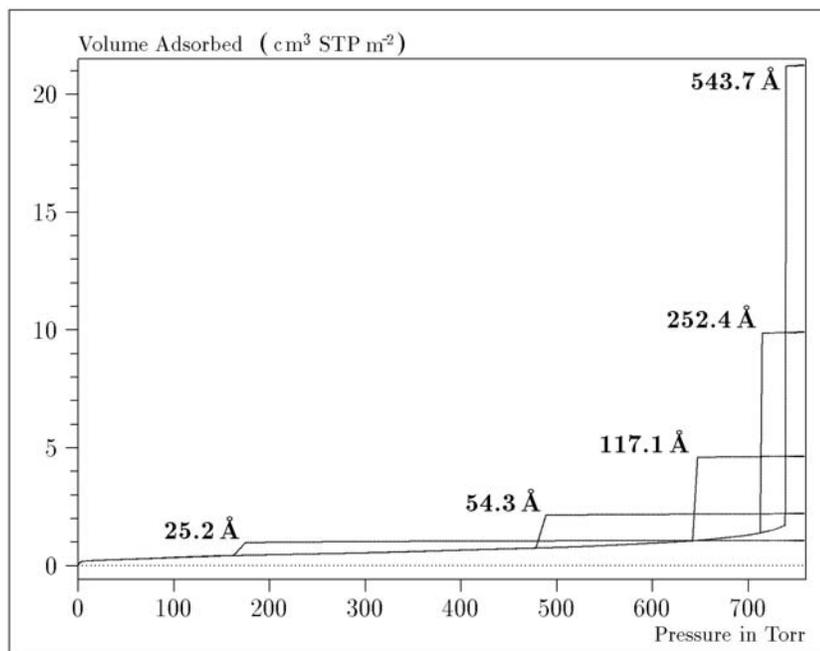


Figure D-3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

Figure D-4 shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.

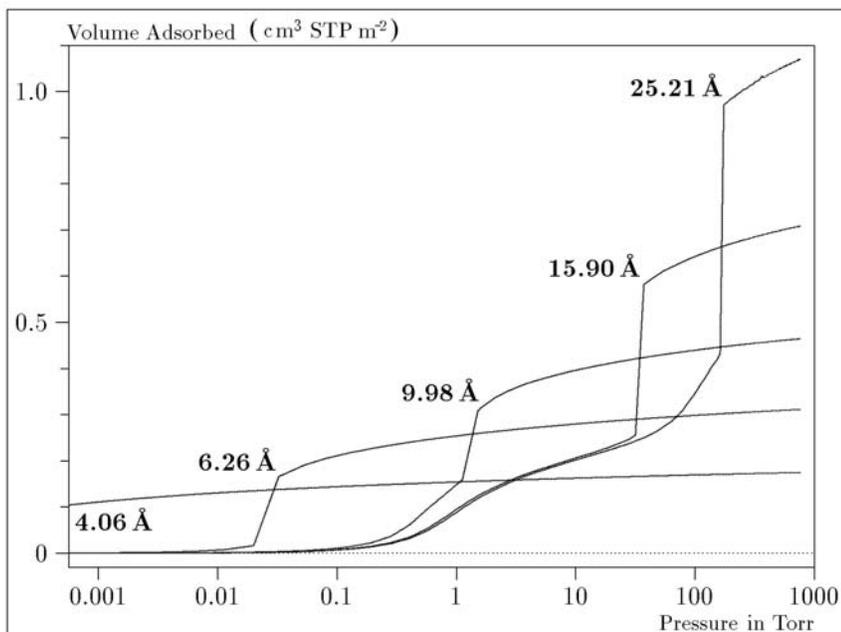


Figure D-4. Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

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 77 K

Using the modified Tarazona prescription described by Olivier (refer to [References](#), references 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 dialog box) 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, i.e., for the adsorptive/adsorbent pair divided by Boltzmann's 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 77 K; Argon at 87 K

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 Å.

Reference: 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 (Montmorillonite)

Geometry	Cylinder
Substrate	Crystalline Silicate
Category:	Porosity
Method:	Nitrogen at 77 K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFT) calculations and experimental isotherms for reference samples of montmorillonite. 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 Å.

- 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 87 K

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 77 K

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 Å.

Reference: 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 77 K; Argon at 87 K

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 Å.

Reference: 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 77 K; Argon at 87 K

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 Å.

Reference: 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, $A_s=12$, 2D-NLDFT

Ar - Carbon Finite Pores, $A_s=12$, 2D-NLDFT

Geometry	Finite Slit
Substrate	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

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.

Reference: See above reference.

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 Å.

Reference: 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 Å.

Reference: See above reference.

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 Å.

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 Å.

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 the calculations appendix. 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 fre-

quently 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 at 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:

$$\text{Surface tension} = 8.88 \text{ dynes cm}^{-1}$$

$$\text{Molar density} = 0.02887 \text{ g cm}^{-3}$$

N₂ - Halsey Thickness Curve

Geometry	Cylinder
Substrate	Average
Category:	Porosity
Method:	Nitrogen at 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 at 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:

$$\text{Surface tension} = 8.88 \text{ dynes cm}^{-1}$$

$$\text{Molar density} = 0.02887 \text{ g cm}^{-3}$$

N2 - Harkins and Jura Thickness Curve

Geometry	Cylinder
Substrate	Average
Category:	Porosity
Method:	Nitrogen at 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

Reference: 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	Slit
Substrate	Average
Category:	Porosity
Method:	Nitrogen at 77 K

The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

$$\log(p/p^0) = \frac{-16.11}{t^2} + 0.1682^{-0.1137t}$$

The nitrogen properties used in the Kelvin equation are:

$$\text{Surface tension} = 8.88 \text{ dynes cm}^{-1}$$

$$\text{Molar density} = 0.02887 \text{ g cm}^{-3}$$

N2 - Broekhoff-de Boer Model

Geometry	Cylinder
Substrate	Average
Category:	Porosity
Method:	Nitrogen at 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).

Reference: 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).

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).

G. USER-DEFINED REPORTS, PYTHON MODULE

The mic Python module allows you to access primary and overlay isotherm data and create graphical, tabular, and summary reports. Graphical reports consist of a single graph with one or more curves on one or two y-axes. Tabular reports consist of one or more tables consisting of one or more labeled columns of data. Summary reports consist of summary sections, each containing a two-column table of label and value pairs.

TABLE 1: Example Python Script for User-defined Reports	
1. import mic	Import the mic (required) and numpy (optional) packages.
2. import numpy as np 3. # Isotherms as list of components	
4. # Isotherms as list of components 5. iso_rel = mic.isotherm('rel')	Get the isotherm data.
6. iso_abs = mic.isotherm('abs') 7. # or as components. 8. prel, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('rel') 9. pabs, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('abs') 10. # Overlays work the same way but are not guaranteed to be valid.	
11. overlays = []	Get the overlay isotherm data.
12. for i in range(8) : 13. ov = mic.overlay(i, 'rel') 14. overlays.append(ov if ov[0].any() else None) 15. # A graphical report with 3 curves.	
16. mic.graph('Graphical Report 1', 'Rel. Press', 'Qads')	Add a graphical report.
17. mic.graph.add('Sample isotherm', prel, qads)	Add some curves to the report.
18. mic.graph.add('Linear transform', prel, qads * 4.0 + 1.0) 19. mic.graph.addyy('Another transform', prel, np.log(qads * qads)) 20. # Another graphical report, this time with 1 curve	
21. mic.graph('Graphical Report 2')	Add another graphical report.
22. mic.graph.add('Sample isotherm', pabs, qads) 23. # A tabular report with 2 tables.	
24. mic.table()	Add a tabular report.
25. mic.table.addtable("Table 1")	Add a table to the report.

TABLE 1: Example Python Script for User-defined Reports (<i>continued</i>)	
26. mic.table.addcolumn("Column 1",	Add columns to the table. Note that the column values are strings; this allows arbitrary formatting.
27. ["1", "2.5", "%8.3f" % np.exp(5.0)]) 28. mic.table.addcolumn("Column 2", 29. ["Val A", "Val B", "Val C" % np.exp(5.0)]) 30. mic.table.addtable("Table Two") 31. mic.table.addcolumn("Column A", ["Small", "Smaller"]) 32. mic.table.addcolumn("Column B", ["Big", "Bigger"]) 33. # Another tabular report	
34. mic.table("Tabular Report Two")	Add another table.
35. mic.table.addtable("One") 36. mic.table.addcolumn("Column A", ["Small", "Smaller"]) 37. mic.table.addcolumn("Column B", ["Big", "Bigger"]) 38. mic.table.addcolumn("Column C", ["Embiggen", "Cromulent"]) 39. # A summary report	
40. mic.summary("A summary report")	Add a summary report.
41. mic.summary.add("Quantity Adsorbed",	Add sections to the summary report. Note that summary sections are just specialized reports with two columns and no column headers.
42. ["First", "Last"], 43. ["%8.3f" % qads[0], "%8.3f" % qads[-1]) 44. mic.summary.add("Rel Pressure", 45. ["First", "Last"], 46. ["%8.3f" % prel[0], "%8.3f" % prel[-1])	

Note that the mic module is automatically imported when the user script is run. For test purposes, a mic.pyc pre-compiled module will be provided.

TABLE 2: Function Reference for mic Python Module (User-defined Reports)

`mic.isotherm(press_type='rel')`

Get the primary isotherm data.

Keyword arguments:

`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.isotherm('rel')`

`p` --- array of pressure (relative or absolute)
`qads` --- array of cumulative quantity adsorbed
`num_ads` --- number of points in the adsorption curve
`warm_fs` --- warm free-space
`cold_fs` --- cold free-space
`mass` --- sample mass
`desc` --- sample description

`mic.overlay(overlay_number=1, press_type='rel')`

Get overlay isotherm data.

Keyword arguments:

`overlay_number` --- the overlay number (1 through 8)
`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.

TABLE 2: Function Reference for mic Python Module (User-defined Reports) (continued)

```
mic.imported_pore_data( import_number = 1 )
```

Get imported pore data.

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 (cm³/g); empty-array if unavailable.

desc --- Name of data set; empty-string if unavailable.

```
mic.adsorptive_data( sample_number = 0 )
```

Get adsorptive data for each sample

Keyword arguments:

sample_number --- Identifier for the adsorptive data to retrieve (default = 0).

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( i )
```

csa --- cross sectional area (nm²)

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')

TABLE 2: Function Reference for mic Python Module (User-defined Reports) (continued)

```
mic.graph(title='User Graph', xlabel='X axis', ylabel='Y axis',
          ylabel='YY axis', xlinear=True, ylinear=True, yylinear=True)
```

Create a new graphical report.

Keyword arguments:

```
title      --- the graphical report title (default = 'User Graph')
xlabel     --- x-axis label (default = 'X axis')
ylabel     --- y-axis label (default = 'Y axis')
ylabel     --- 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)
```

```
mic.graph.add (self, name, x, y, yyaxis=False, color=None, linestyle='-', marker='a',
              graphtype='both')
```

Add a curve to the last created graphical report.

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')
           " or None : no marker
           '+' : plus
           'o' or '0' : circle
           'x' : cross
           '^' : up triangle
           'v' : down triangle
           's' : square
           'd' : diamond
           '8' : hourglass
           '~' : horizontal hourglass
           'a' : automatically selected
```

TABLE 2: Function Reference for mic Python Module (User-defined Reports) (continued)

graphtype --- graph type; (default = 'both')
 'curve' or 'c' : curve
 'points' or 'p' : points
 'both' or 'b' : curve-and-points
 'hist' or 'h' : histogram

mic.table(title='User Table')

Create a new tabular report.

Keyword arguments:
 title --- the tabular report title (default = 'User Table')

mic.table.addtable(name)

Add a table to the last created tabular report.

Keyword arguments:
 name --- the table name

mic.table.addcolumn(header, values)

Add a column to the last created table.

Keyword arguments:
 header --- column header; must be a string (or convertible)
 values --- column values; must be a list of strings (or convertible)

mic.summary(title='User Summary')

Create a new summary report.

Keyword arguments:
 title --- the summary title

mic.summary.add(self, name, labels, values)

Add a summary section to the last created summary report.

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 values

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