

ASAP®

ACCELERATED SURFACE AREA AND POROSIMETRY SYSTEM



ASAP 2060

OPERATOR MANUAL

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ABOUT THIS MANUAL

The following formats may be used in this manual.



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



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



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

Field Labels and Screen Titles

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

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1 ABOUT THE ASAP 2060 ANALYZER



The ASAP (Accelerated Surface Area and Porosimetry) 2060 is a single-port physisorption instrument that includes the ASAP 2020 manifold in a smaller cabinet. The analyzer is equipped with an elevator that raises and lowers the analysis bath fluid dewar automatically. A removable shield is included for safety purposes.

The sample saturation pressure (Psat) tube is located next to the sample analysis port. Gas inlet ports and cable connections are located conveniently on the rear panel of the analyzer.

The instrument includes isothermal jackets for the sample tube in order to maintain a stable thermal profile along the full length of the sample and Psat tubes.

ANALYZER COMPONENTS

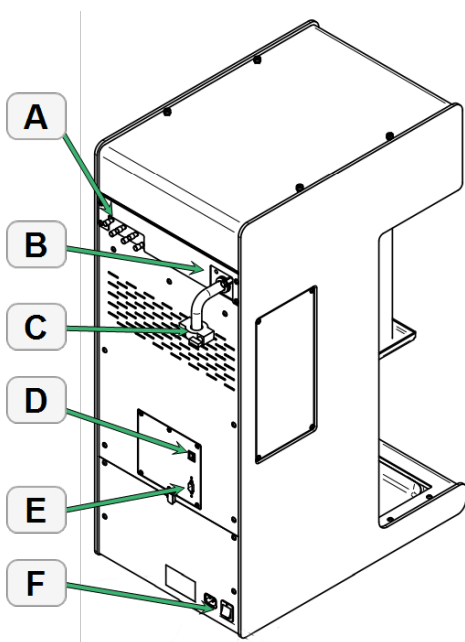
FRONT COMPONENTS



Front Panel Components

Component		Description
A	Elevator	Provides placement of the dewar or furnace.
B	Power indicator light	Illuminates when power is ON.
C	P _{sat} port	Use to measure saturation pressure.
D	Sample port	Use to attach a sample for analysis.

REAR COMPONENTS



Rear Panel Components

Component		Description
A	Gas Inlet ports	For attaching the analysis gases.
B	Helium Inlet port	Provides connection of helium for measuring the free space.
C	Vacuum Port	For attaching vacuum pumps.
D	Ethernet Port	Provides connection to the computer or Ethernet switch.
E	RS-232 Port	Not used.
F	Power	Provides electrical cable connector. Includes On/Off switch.

EQUIPMENT OPTIONS

Equipment Option	Description
MicroPrep	Temperature controller and heating mantle. Used for in-situ degassing.
SmartPrep	The SmartPrep passes flowing-gas over the sample at elevated temperatures. It contains six sample ports in which temperature, ramp rates, and soak times are individually controlled by the analyzer program so that all degas information is integrated into the sample data file for future reference. It contains 2 serial ports, one for connecting to the computer and the other for connection of up to 3 additional SmartPrep instruments permitting 24 preparation ports to be used.

POWER ANALYZER ON AND OFF

Power on the equipment in the following order:

1. Analyzer
2. Computer, monitor, and printer



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

Power off the equipment in the following order:

1. Computer*, monitor, and printer

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

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


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

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

2. Analyzer

SPECIFICATIONS

Specification	Description
Cryogen System	
Analysis Time	Unlimited. Cryogen dewars may be refilled without affecting the accuracy of results.
Capacity	3 liter dewar, which typically provides greater than 72 hours of unattended analysis.
Special Features	Isothermal Jackets maintain cryogen level constant on sample tube and P ₀ tube during analysis while evaporation of cryogen occurs.
Electrical	
Voltage	85 - 264 VAC
Frequency	47 - 63 Hz
Power	150 VA
Environment	
Temperature	10 to 30 °C, operating 0 to 50 °C, storing or shipping
Humidity	20 to 80% relative, noncondensing
Gases	
Argon, carbon dioxide, nitrogen, krypton, and other suitable gases	
Manifold Temperature Transducer	
Type	Platinum resistance device (RTD)
Accuracy	+ 0.02 °C
Physical	
Height	94.5 cm (37.2 in.)
Width	38.1 cm (15.0 in.)
Depth (chassis)	59.0 cm (23.2 in.)
Depth (with vacuum tube))	68.1 cm (27.1 in.)
Weight	68 kg (150 lbs)
Pressure Measurement	
Accuracy — Includes nonlinearity, hysteresis, and nonrepeatability. Transducer manufacturer's specifications. All within 0.15% of reading.	
Range	0 to 950 mmHg
Resolution	
1000 mmHg Transducer	0.001 mmHg
10 mmHg Transducer	0.00001 mmHg

Specification	Description
0.1 mmHg Transducer	0.0000001 mmHg
Sample Size	
Sample tubes are available for various size pellets, cores, and powders. Sample tube stems are normally 1.27 cm (1/2 in.) OD with 9 cc bulbs. Also available are 0.635 cm (1/4 in.) or 0.953 cm (3/8 in.) OD with 9 cc bulbs. A 22-mm (0.87 in.) ID, 25 mm (1.0 in.) OD sample tube kit is also available. Special tubes can be designed to accommodate unusual samples.	
System Capacity	
Analysis	1 sample port and 1 saturation pressure tube
Total Operating Capacity	Up to four complete analysis units can be controlled independently by one computer
Computer Requirements	
Windows 7 Professional or higher operating system is recommended for the best user experience. If the computer is to be connected to a network, a second Ethernet port on the computer must be used for that purpose.	
 All users of the application will need Read / Write permission to all directories and subdirectories where the application is installed.	
<i>In keeping with a policy of ongoing product improvement, specifications are subject to change without notice.</i>	

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2 ABOUT THE SOFTWARE

The *Help* menu provides access to this operator manual and tutorials on using the software.

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

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

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

SOFTWARE SETUP



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

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

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

Insert the setup CD into the CD-ROM drive. The setup program starts automatically. If the program does not start automatically, navigate to the CD drive, locate and double click the *setup.exe* file.



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

SOFTWARE UPDATES

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

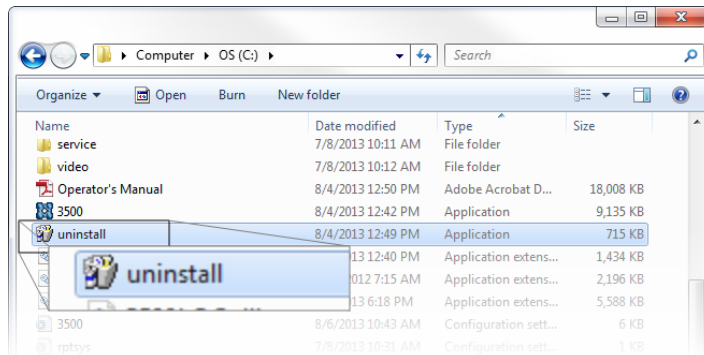
- a later version than the current installation
- the same version as the current installation
- an earlier version than the current installation

Insert the setup CD into the CD-ROM drive. The setup program starts automatically. If the program does not start automatically, navigate to the CD drive, locate and double click the *setup.exe* file.

UNINSTALL THE SOFTWARE

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

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



MENU STRUCTURE

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

Main Menu Bar Options

Option	Description
File	Use to manage files used by the application — such as sample files, analysis conditions files, report options files, etc.
Unit [n]	Use to perform analyses, calibrations, and other analyzer operations. A <i>Unit [n]</i> displays on the menu bar for each analyzer attached to the computer.
Reports	Use to run reports and view the results.
Options	Use to edit the default method, specify system configuration, specify units, and change presentation options.
Window	Use to manage open windows and display a list of open windows. A checkmark appears to the left of the active window.
Help	Provides access to the operator manual, online instructional tutorials, the Micromeritics web page, the analyzer web page, and information about the analyzer.

COMMON FIELDS AND BUTTONS

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

Common Fields and Buttons Table

Field or Button	Description
Add Log Entry	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Autoscale checkbox	When enabled on report parameters windows, allows the x- and y-axes to be scaled automatically. <i>Autoscale</i> means that the x- and y- ranges will be set so that all the data is shown. If <i>Autoscale</i> is not selected, the entered range is used.
Axis Range	On report parameters windows, the <i>From / To</i> fields are enabled when <i>Autoscale</i> options are not selected. Enter the starting and ending values for the x- and / or y-axes.
Bar Code	Enter bar code reader information if a bar code reader is connected to the computer's USB port. If a bar code reader is not used, this alphanumeric field can be used to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Browse	Searches for a file. Select a file from the <i>Name</i> column or from the library, then click Open . Alternatively, double click the file name to open (or import) the file.
Cancel	Discards any changes.
Close	Closes the active window.
Close All	Closes all active windows. If changes were made and not yet saved, a prompt displays for each changed file providing the option to save the file.
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Delete	When working with report parameters, Delete removes the selected report. Deleted reports will have to be regenerated if deleted in error.
Destination group box	<ul style="list-style-type: none"> • Preview. Previews the predefined report on the screen. • Print. Sends the report to the default printer. • Copies. Select the number of copies to print. This field is only enabled when <i>Print</i> is selected. • File. Select the destination directory. Enter a new file name in the <i>File name</i> field, or accept the default. Select to save the file as a report sys-

Common Fields and Buttons Table (continued)

Field or Button	Description
	tem (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
Edit	When working with report parameters, highlight the item in the <i>Selected Reports</i> list box and click Edit to modify the report details.
Exit	If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the application without saving the changes.
Export	Exports isotherm data in a sample information file as a .REP, .TXT or .XLS file. When saved to a file, the data can be imported into other applications.
File name text box	Select a file from either the <i>Name</i> column or from the library. The file name displays in the <i>File name</i> text box. Click Open or double click the file name to open the file. To select more than one file, hold down the Ctrl key on the keyboard while selecting the files, or hold down the Shift key to select a range of files.
From / To text boxes	When working with report parameters windows, enter the <i>From</i> and <i>To</i> range for x- and / or y-axes.
List	Provides the option to create a list of sample or report options file information, for example, file name, date / time the file was created or last edited, file identification and file status.
Name column	A list of files in the selected directory or library.
Next	Click to move to the next window or next step.
OK	Saves and closes the active window.
Open	Opens the selected file. Alternatively, double click the file name in the <i>Name</i> column to open the file.
Prev	Click to move to the previous window.
Preview	Previews predefined reports. Click the tabs at the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled.
Print	Sends the report to the selected destination (screen, printer or file).
Remove	Click to remove an item from the list.
Replace	Click to select another file where the values will replace the current file's values.
Replace All	Click to select another .SMP file where the values will replace all values for the active Sample Information file. The original file will remain unchanged.
Report	Click to display a window to specify report output options. <ul style="list-style-type: none"> • Start Date. Displays a calendar to select the start date for the report.

Common Fields and Buttons Table (continued)

Field or Button	Description
	<ul style="list-style-type: none">• Preview. Previews the predefined report on the screen.• Print. Sends the report to the default printer.• Copies. Select the number of copies to print. This field is only enabled when <i>Print</i> is selected.• File. Select the destination directory. Enter a new file name in the <i>File name</i> field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
Save	Saves changes to the active window.
Save As	Saves a file in the active window under a different file name.
Start	Starts the report, test, analysis, or operation.
Table buttons	Use to modify the table contents. <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.

FILE STATUS, DESCRIPTION, EXTENSION, AND LOCATION

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

File Status and Description Table

File Status	Description
Analyzing	Sample information files that are currently being used for analysis.
Complete	Sample information files used in an analysis that has been completed.
Entered	Sample information files containing manually entered data.
No Analysis	Sample information files which have not been used to perform an analysis.

File Type, Extension, and Location Table

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

APPLICATION SHORTCUTS

MENU SHORTCUTS

Shortcut menus are available for:

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

KEYBOARD SHORTCUTS

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

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



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

Keyboard Shortcut Table

Field or Button	Description
Alt + [Unit n]	Opens the Unit [n] menu.
Alt + F	Opens the <i>File</i> menu.
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Alt + H	Opens the <i>Help</i> menu.
Alt + I	Opens the <i>Options</i> menu.
Alt + R	Opens the <i>Reports</i> menu.
Alt + W	Opens the <i>Window</i> menu.
Shift + F9	Opens the shortcut menu of (1) selected component on analyzer schematic when manual control is enabled or (2) onscreen reports.
Ctrl + N	Opens a new sample file.
Ctrl + O	Opens the <i>File Selector</i> .
Ctrl + P	Opens the <i>File Selector</i> to start a report from a selected .SMP file.
Ctrl + S	Saves the open file.
F1	Opens the online help operator manual.

Keyboard Shortcut Table (continued)

Field or Button	Description
F2	Displays the <i>File Selector</i> window.
F3	When in the <i>File Selector</i> window, opens the file search box.
F4	When in the <i>File Selector</i> window, opens the address bar.
F6	Cascades open windows.
F7	Tiles open windows.
F8	Opens the <i>File Selector</i> to start a report from a selected .SMP file.
F9	Closes all open reports.

OPTION PRESENTATION

Options > Option Presentation

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

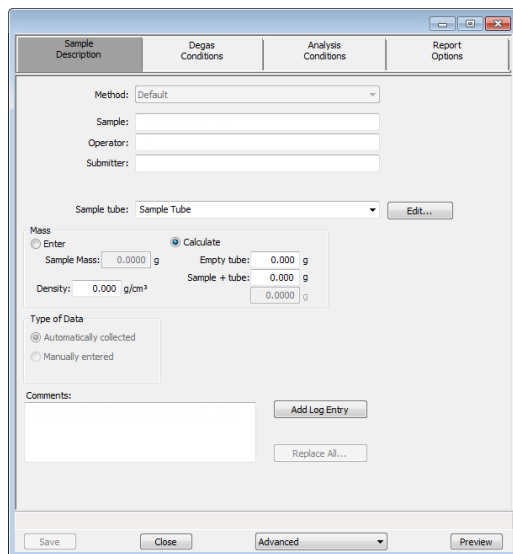
Presentation Display Table

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

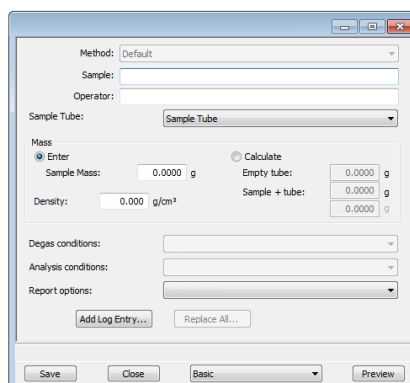


Specify or change the default option presentation by selecting **Options > Option Presentation**, or select *Basic* or *Advanced* from the drop-down list at the bottom of the window.

The following examples show the same sample information file in *Advanced* and *Basic* display. *Basic* and *Restricted* displays will look the same.



**Advanced
presentation option**



**Basic / Restricted
presentation option**

LIBRARIES

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

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

See [Manage Libraries below](#).

MANAGE LIBRARIES

Options > Manage Libraries



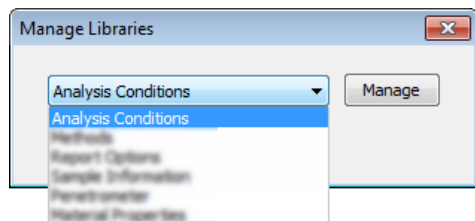
This feature is available only for Windows 7 and higher operating systems.

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

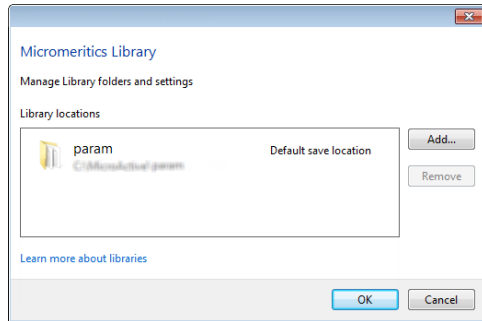


One library can include up to 50 folders. Other items such as saved searches and search connectors cannot be included.

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



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



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



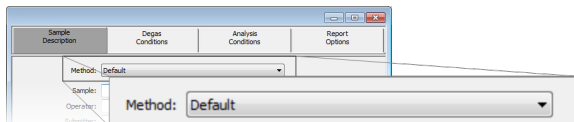
When removing a folder from a library, the folder and its contents are not deleted from the original file storage location. However, when deleting files or folders from within a library, they are deleted from their original file storage location.

2. Click **OK** when done.

METHODS

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

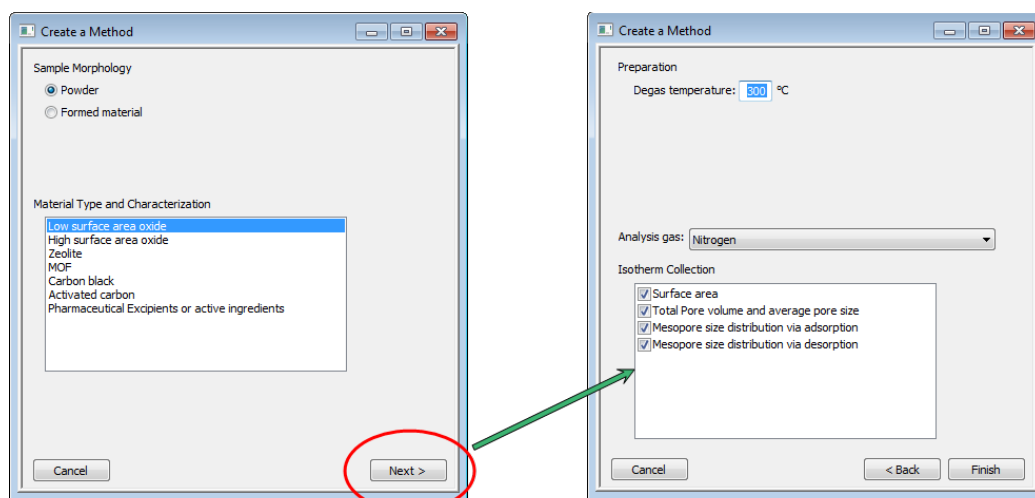
The *Method* drop-down list displays only those methods applicable to the open sample file type.



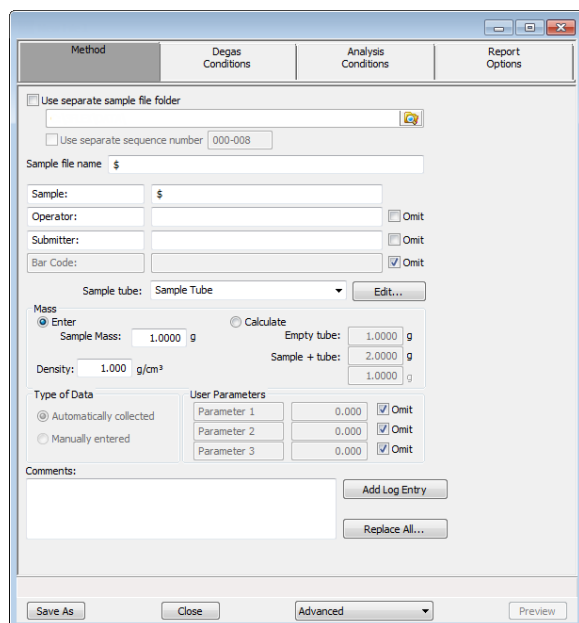
CREATE A NEW METHOD

File > New Method

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



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



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



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

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

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

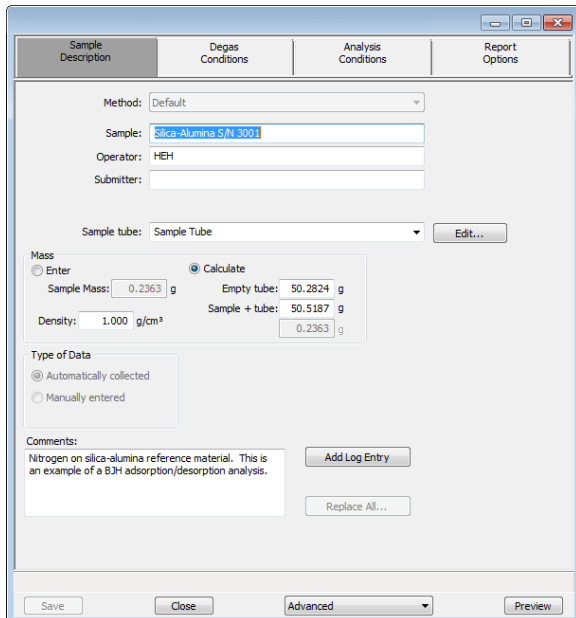
EDIT THE DEFAULT METHOD

Options > Default Method

See [Create a New Method on page 2 - 14](#).

EDIT A METHOD

File > Open > [Method]



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

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

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



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

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

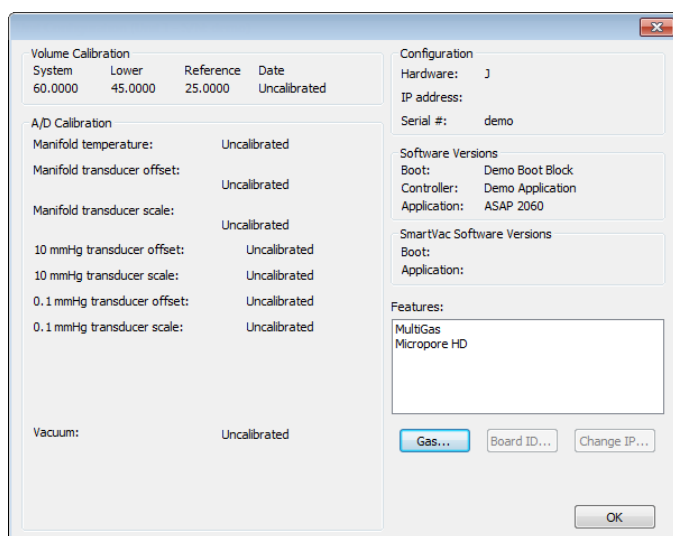
CONFIGURE THE ANALYZER

SPECIFY GAS PORTS

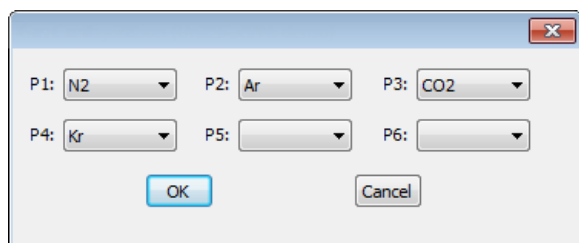
Unit [n] > Unit Configuration

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

The analyzer has gas inlets for up to six analysis gases. The gases connected to the inlets must be specified in the analysis program. If the gas is changed on one of the inlets, the same change must be made on the *Unit Configuration* window. The analysis software must be updated when gases change.




1. Click **Gas**.



2. Use the drop-down arrows to specify the type of gas installed on each port.
3. Click **OK** to close the window and save the changes.

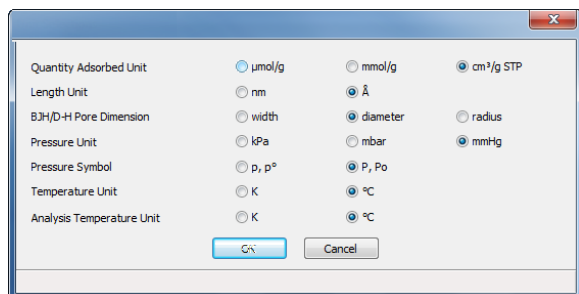
Unit Configuration Fields and Buttons Table

Field or Button	Description
Calibration group boxes	Displays calibration information for analyzer components.
Configuration group box	<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 <i>Unit IP Setup</i> 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 display information from the electronic circuit boards in the instrument.. These parameters cannot be edited.
Features group box	Displays options installed on the analyzer.
Gas button	Displays ports for gas selections.
Software Versions group box	Displays the software versions of the MIC BIOS, controller, and analysis program.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

SPECIFY UNIT SELECTIONS

Options > Units

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

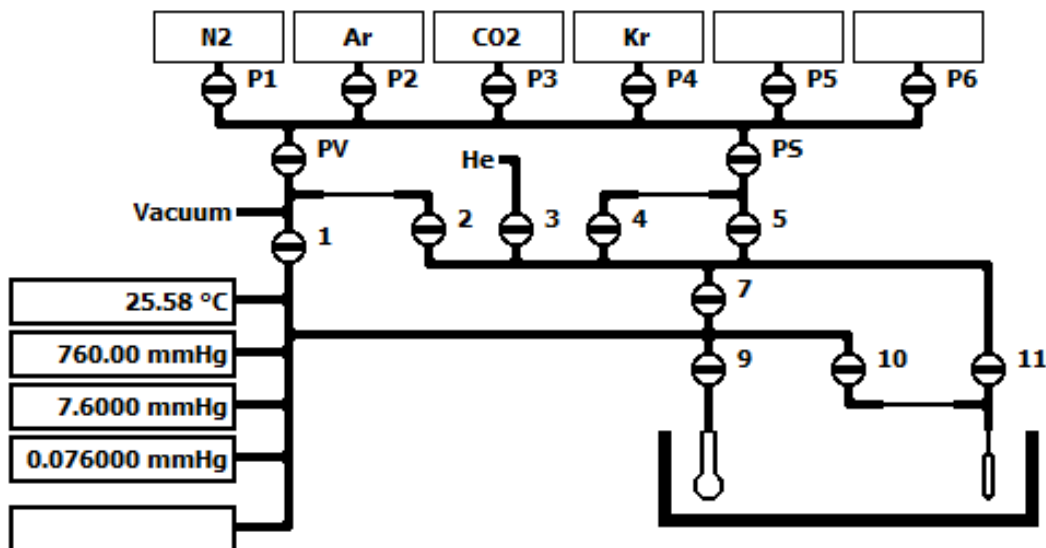


ANALYZER STATUS





SHOW INSTRUMENT SCHEMATIC

Unit [n] > Show Instrument Schematic

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



Analyzer Schematic Icon Table

Icon or Symbol	Description
	Open Valve. Green indicates an open valve.
	Closed Valve. Yellow indicates a closed valve. When manual control is disabled, closed valves appear white.
	Elevator.
	Sample Tube. Cannot be manually controlled.

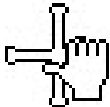

Analysis Valve Descriptions

Valve	Description
1	Unrestricted vacuum
2	Restricted vacuum
3	Helium inlet port valve
4	Restricted analysis gas
5	Unrestricted analysis gas
7	Lower manifold isolation
9	Sample port
10	Restricted Psat tube port
11	Unrestricted Psat tube port
P1 through P6	Gas inlet port valves
PS	Supply valve for physisorption gases
PV	Vacuum valve for physisorption gases

Instrument Schematic Shortcut Menus

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

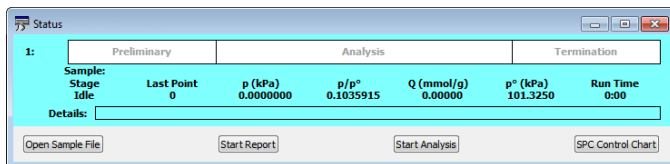
Schematic Shortcuts Table

Schematic Shortcut Icon	Available Options:
Valve options 	<ul style="list-style-type: none"> • Close. Closes the selected valve. • Open. Opens the selected valve. • Pulse. Use to quickly turn the valve on and off allowing the operation to proceed in small increments.
Elevator options 	<ul style="list-style-type: none"> • Raise. Select <i>Raise</i> to raise the elevator. When it is moving, press the keyboard space bar to stop the movement (or right click and select <i>Stop</i> from the menu). • Lower. Select <i>Lower</i> and press the keyboard space bar to lower the elevator. • Stop. Stops the elevator from moving.

SHOW STATUS

Unit [n] > Show Status

Use to show the current status for each port.



The Status window displays the following data for Unit 1:

Sample Stage	Last Point	p (kPa)	p/p ^o	Q (mmol/g)	p ^o (kPa)	Run Time
Idle	0	0.000000	0.1035915	0.00000	101.3250	0:00

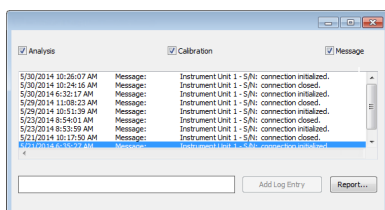
Buttons at the bottom: Open Sample File, Start Report, Start Analysis, SPC Control Chart.

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

SHOW INSTRUMENT LOG

Unit [n] > Show Instrument Log

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




The Instrument Log window shows a list of messages under the 'Message' tab:

- 5/30/2014 10:26:07 AM Message: Instrument Unit 1 - 5%: connection initialized.
- 5/30/2014 10:26:16 AM Message: Instrument Unit 1 - 5%: connection closed.
- 5/30/2014 6:32:17 AM Message: Instrument Unit 1 - 5%: connection initialized.
- 5/29/2014 11:08:23 AM Message: Instrument Unit 1 - 5%: connection closed.
- 5/29/2014 10:51:39 AM Message: Instrument Unit 1 - 5%: connection initialized.
- 5/23/2014 8:54:01 AM Message: Instrument Unit 1 - 5%: connection closed.
- 5/23/2014 8:53:59 AM Message: Instrument Unit 1 - 5%: connection initialized.
- 5/21/2014 10:17:50 AM Message: Instrument Unit 1 - 5%: connection closed.

Buttons at the bottom: Add Log Entry, Report...

Instrument Log Fields and Buttons Table

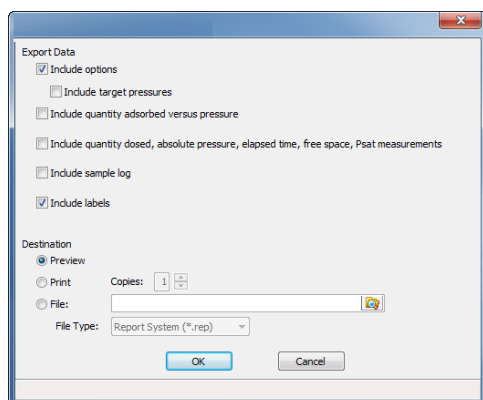
Field	Description
Analysis / Calibration / Message	Select the logs to display.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

EXPORT FILES

File > Export

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

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



Types of data that can be included:

- Options
 - Target pressures
 - Quantity adsorbed versus relative pressure
 - Quantity dosed, absolute pressure, elapsed time, free space, Psat measurements
 - Sample log
 - Labels
4. Specify the export destination in the *Destination* section.
 - **Preview.** Previews the predefined report on the screen.
 - **Print.** Sends the report to the default printer.
 - **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected.

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

LIST FILES

File > List

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



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

File Listing					
No.	File Name	Date	Time	File Identification	
1	Argon.adp	4/18/2015	8:11:29 AM	Argon	
2	Carbon Dioxide.adp	4/18/2015	8:11:29 AM	Carbon Dioxide	
3	Carbon Monoxide.adp	4/18/2015	8:11:29 AM	Carbon Monoxide	
4	Helium.adp	4/18/2015	8:11:29 AM	Helium	
5	Hydrogen.adp	4/18/2015	8:11:29 AM	Hydrogen	
6	Krypton.adp	4/18/2015	8:11:29 AM	Krypton	
7	Nitrogen.adp	4/18/2015	8:11:29 AM	Nitrogen	
8	Oxygen.adp	4/18/2015	8:11:29 AM	Oxygen	
9	Xenon.adp	4/18/2015	8:11:29 AM	Xenon	

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

Blank Page

3 ABOUT SAMPLE FILES

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

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

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

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



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

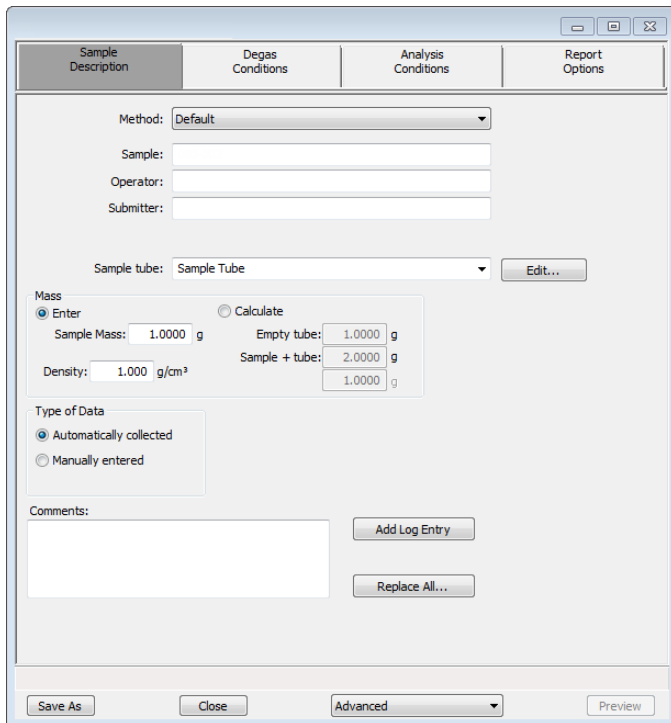
CREATE SAMPLE FILES IN ADVANCED OPTION PRESENTATION

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

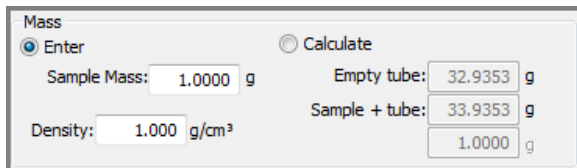
Specify or change the default display option by selecting **Options > Option Presentation** or select *Basic* or *Advanced* from the drop-down list at the bottom of the window.

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

- *Sample Tube* parameters are edited on the *Sample Description* tab
- *Adsorptive Properties* are edited on the *Analysis Conditions* tab
 1. Go to **Options > Option Presentation > Advanced** and ensure *Advanced* has a checkmark.
 2. To create a new sample file, go to **File > New Sample**, or to open a sample file go to **File > Open** and select a sample file.
 3. Select a method from the *Method* drop-down 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 fields may not display (or may have a different field label) if modified in the method from which the sample file was created, either through **Options > Default Method** or **File > Open > Method**.
6. In the *Sample Tube* drop-down list, select a sample tube. If the required sample tube does not appear in the list, click **Edit** and enter the description and other parameters for this tube. Then go to **File > Save As > Sample Tube** to save these values for the next time this sample tube is used.
7. In the *Mass* group box, indicate if mass is to be manually entered by the operator (*Enter*) or calculated by the system (*Calculate*).



Sample File Fields and Buttons Table

Field or Button	Description
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Mass group box	<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. Mass can be changed any time before, during, or after analysis.</p> <ul style="list-style-type: none"> • Enter. Enables the <i>Sample Mass</i> field. Enter a value for the sample mass. • Calculate. Enables the <i>Empty tube</i> and <i>Sample + tube</i> fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass: $Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$ • Density Value is used for the calculated free space method only. Use 0.000 for a blank analysis.
Method	Select a method from the drop-down list. See Methods on page 2 - 14 .
Operator	Enter operator identification information. This field label may have been renamed or may not display if modified in Options > Default Methods .
Sample	Enter a sample description.
Sample Tube	Select a sample tube file from the drop-down list, or click Edit to modify or create a new Sample Tube file. See Sample Tube on page 4 - 1 .
Submitter	Enter submitter identification information. This text box may have been renamed or may not display if modified in Options > Default Methods .

Sample File Fields and Buttons Table (continued)

Field or Button	Description
Type of Data group box	<p>Automatically collected. Select if the type of data will be automatically collected by the system while an analysis is running. Manually entered. Use to enter data manually that was collected from another source. If <i>Manually entered</i> is selected, the Isotherm Report becomes available in the <i>Basic/Advanced</i> drop-down list for pasting or importing data into the file.</p> <p>See Manually Enter Data on page 3 - 8.</p>
User Parameters group box	<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 <i>Summary Report</i>. Some fields may not display (or may have a different field label) if modified in the method from which the sample file was created, either through Options > Default Method or File > Open > Method.</p>
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

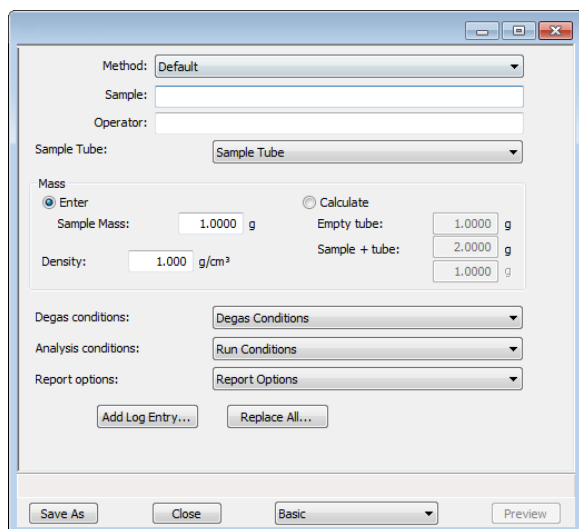
CREATE SAMPLE FILES IN BASIC OPTION PRESENTATION

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

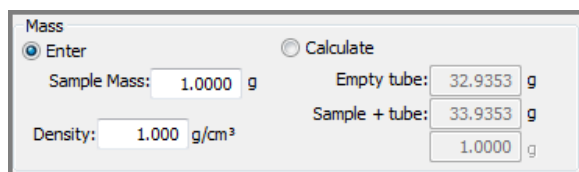


When using the *Basic* option presentation, switch to *Advanced* to edit parameter file values. When using the *Restricted* option presentation, parameter files cannot be edited.

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



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



7. Click the down arrows to select default parameter files for *Degas conditions*, *Analysis conditions*, and *Report options*. The *Degas Conditions* option will not display if it has not been enabled in **Options > Option Presentation > Show Degas Conditions**.

8. To auto-populate fields from another .SMP file, click **Replace All** and select a .SMP file that contains the necessary parameters. Select the file and click **Replace**.
9. Click **Add Log Entry** to enter notes for the analyzer log report. Create entries that cannot be recorded automatically through the software, for example, when the port filter was changed.
10. Click **Save**, then click **Close**. The file can be retrieved later from the *Sample Information* folder in the library.

CREATE SAMPLE FILES IN RESTRICTED OPTION PRESENTATION

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

OPEN A SAMPLE FILE

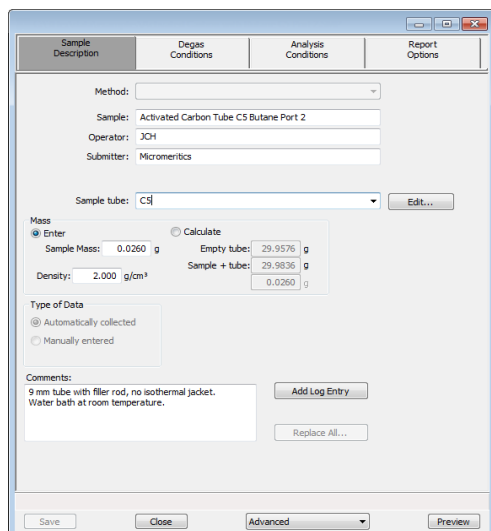
File > Open > [.SMP File]



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

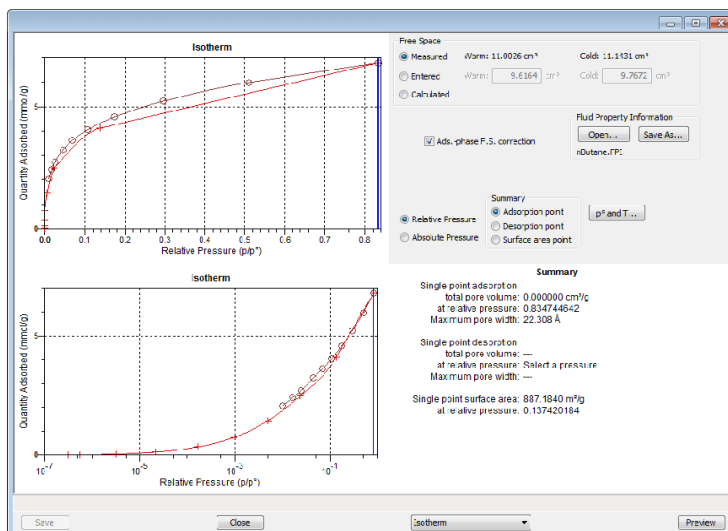
1. Go to **File > Open**.
2. From the *Sample Information* library folder, select a .SMP file:

File Status	Displays
No Analysis	Tabbed file editor
Complete Analyzing Entered	MicroActive report window



The screenshot shows the 'Sample Description' tab of the file editor. It includes fields for Method, Sample, Operator, and Submitter. Below these are mass and density input fields with calculated values. A 'Type of Data' section allows for 'Automatically collected' or 'Manually entered' data. A 'Comments' box at the bottom contains text about the sample tube and isotherm jacket.

Example of tabbed file editor



Example of MicroActive report

MANUALLY ENTER DATA



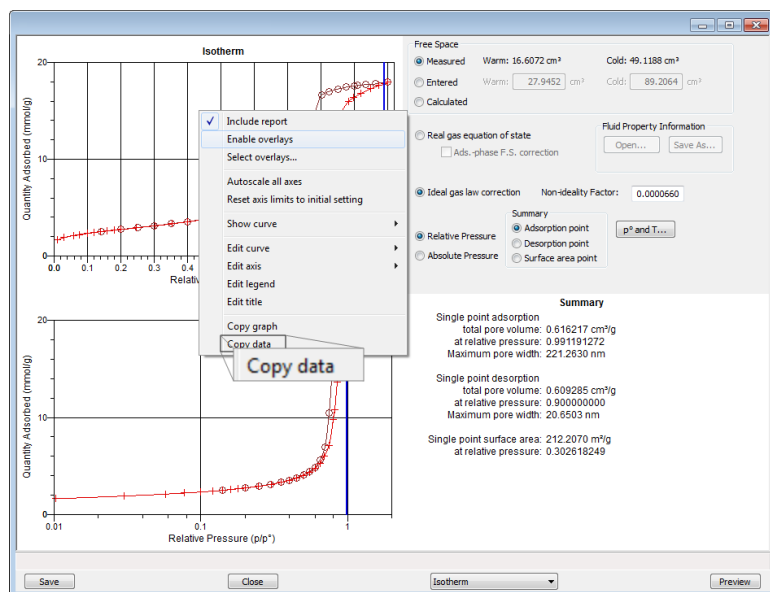
The images shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

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

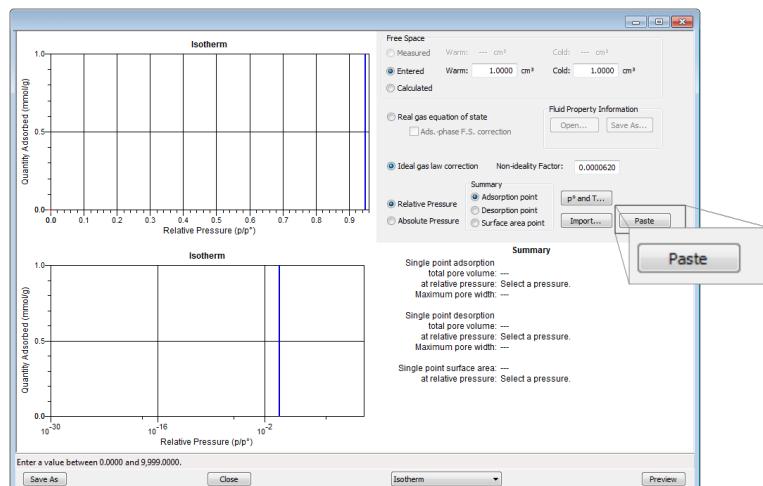
- Copy and paste onto the graph area of the interactive window
- Import data into the interactive window

COPY AND PASTE MANUALLY ENTERED DATA

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



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



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

IMPORT MANUALLY ENTERED DATA

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

ASCII text file format rules

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

Silica Alumina : Adsorption

Relative Pressure	Quantity Adsorbed (cm ³ /g STP)
0.108629	50.6657
0.22288	60.7813
0.339909	71.3095
0.459512	84.4172
0.577447	102.672
0.654583	121.707
0.760074	179.096
0.855713	334.565
0.958511	394.675
0.996251	403.793

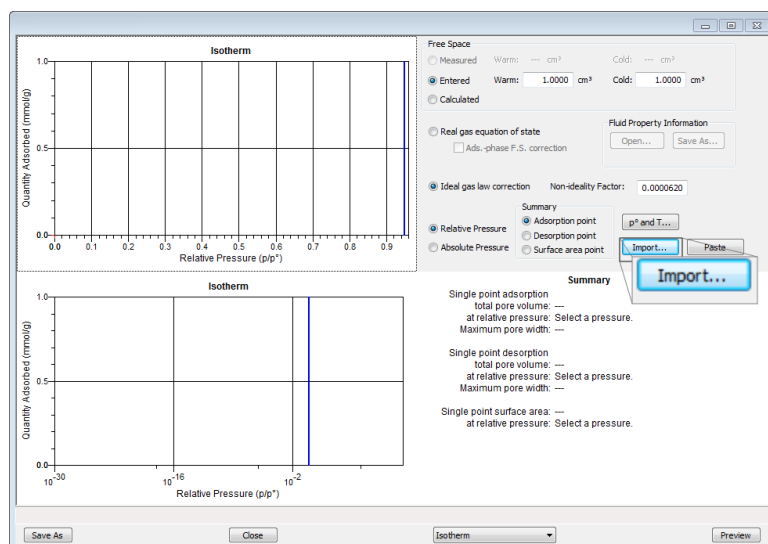
Silica Alumina : Desorption

Relative Pressure	Quantity Adsorbed (cm ³ /g STP)
0.996251	403.793
0.86016	389.626
0.753567	256.264
0.664418	133.099
0.542416	96.7366
0.422295	79.7351
0.346371	71.5994
0.2519	62.8256

0.152718	54.2336
0.103389	49.5803

To import the ASCII text file:

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



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

Blank Page

4 ABOUT PARAMETER FILES

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

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

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

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

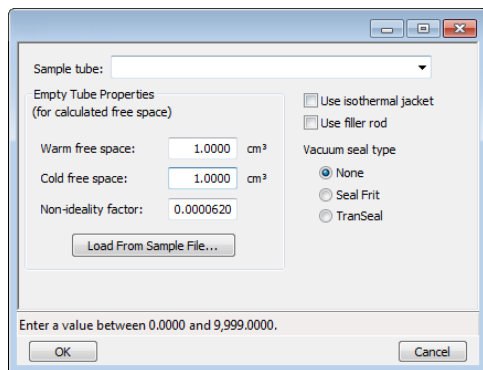
SAMPLE TUBE

File > Open > [.STB File]

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


Sample Tube files specify information about the sample tube.

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



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

Sample Tube Fields and Buttons Table

Field or Button	Description
Cold free space	Empty sample tube gas capacity measured with the dewar raised.
Load from Sample File	Loads parameters from the selected sample file.
Non-ideality factor	Compensates for the forces of attraction between molecules in a real gas. This value is used for a calculated free space.
Sample Tube	It is a good practice to label each sample tube with a unique identification. Enter that information here. This information will also appear in the <i>Sample Tube</i> drop-down list on the <i>Sample Description</i> tab.
Use filler rod	Select if a filler rod is to be used in the sample tube. A filler rod reduces the stem free-space volume resulting in reduction of free-space error.
Use isothermal jacket	Select if an isothermal jacket is to be used. An isothermal jacket maintains a constant temperature profile along the sample tube stem during an extended analysis of more than 1 or 2 hours.
Vacuum seal type	Select the seal type to be used.
Warm free space	Empty sample tube gas capacity measured at room temperature.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

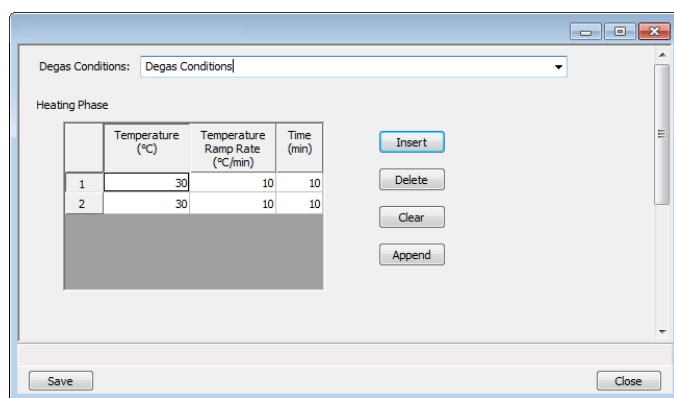
DEGAS CONDITIONS

File > Open > [.DEG File]

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

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

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



	Temperature (°C)	Temperature Ramp Rate (°C/min)	Time (min)
1	30	10	10
2	30	10	10

3. Complete the fields using the following table.
4. Click **Save**, then click **Close**.

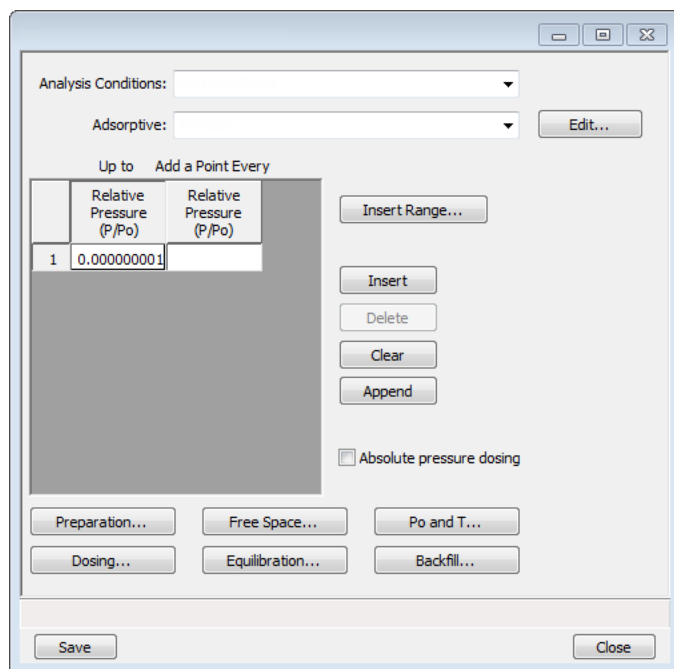
ANALYSIS CONDITIONS

File > Open > [.ANC File]

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

Analysis conditions specify the data used to guide an analysis.

1. Go to **File > Open**.
 - Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
 - Select the appropriate file type from the drop-down list on the lower right portion of the window, then select a file name in the list or enter it in the *File Name* field, then click **Open**.
2. To overwrite analysis conditions with parameters from another *Analysis Conditions* file, on the *Analysis Conditions* tab, click the *Analysis Conditions* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file.
3. To overwrite adsorptive properties with parameters from another *Adsorptive Properties* file, click the *Adsorptive* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file. See [Adsorptive Properties on page 4 - 13](#).



4. Enable *Absolute pressure dosing* to specify pressure targets in mmHg, mbar, or kPa rather than 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.

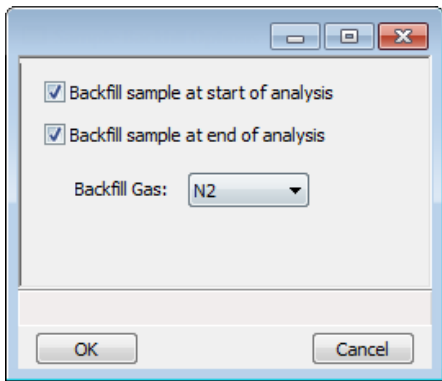
5. Use the following buttons to specify:

Analysis Conditions Buttons Table

Button	Use to Specify...
Backfill	Provides options to backfill sample tube after analysis.
Dosing	Options for dosing tolerance, low pressure dosing, and dosing near saturation pressure.
Equilibration	Provides options to specify the equilibration interval and delay time.
Free Space	How the free space is to be measured.
P₀ and T	How the saturation pressure (P ₀) is to be measured or calculated and the analysis bath temperature.
Preparation	Evacuation rate / time / level, leak test and time values, elevator prompts, and in situ degassing or activation.

6. Click [Save](#), then click [Close](#).

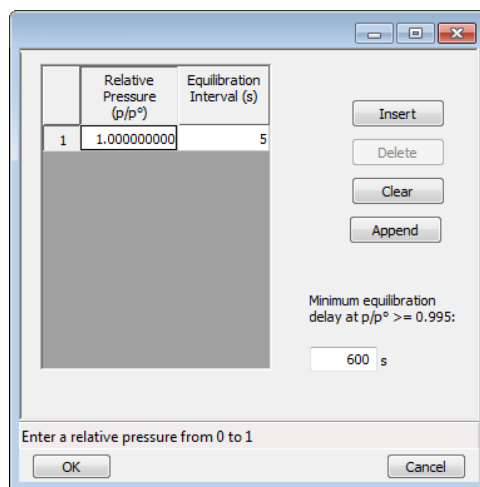
Analysis Conditions Fields and Buttons Table

Field or Button	Description
Absolute pressure dosing	Specifies pressure targets in mmHg, mbar, or kPa instead of relative pressure. If this option is selected, the <i>Relative Pressure</i> labels and entries change to <i>Absolute Pressure</i> in the selected pressure units.
Adsorptive	Select an <i>Adsorptive Properties</i> file from the drop-down list.
Analysis Conditions	Use to browse for a .ANC file that contains analysis condition parameters to be used in the analysis.
Backfill	<p>Select gas for backfilling the sample tube at the start and end of an analysis.</p>  <p>The options are disabled if the file status is <i>Entered</i>, <i>Complete</i>, or <i>Analyzing</i>.</p>

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
Dosing	<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 lower. At higher pressures, the absolute tolerance value is lower. For example: <p>Experiment 1. There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 40 mmHg; 5% of 40 mmHg is 2 mmHg. Since 2 mmHg (relative tolerance) is lower than 5 mmHg (absolute tolerance), 2 mmHg is used. Therefore a minimum pressure of 38 mmHg (40 - 2) must be attained to collect data for a target pressure of 40 mmHg.</p> <p>Experiment 2. There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 200 mmHg; 5% of 200 mmHg is 10 mmHg. Since 5 mmHg (absolute tolerance) is lower than 10 mmHg (relative tolerance), 5 mmHg is used. Therefore a minimum pressure of 195 mmHg (200 - 5) must be attained to collect data for a target pressure of 200 mmHg.</p> <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> First pressure fixed dose. <p>This option is most frequently used when performing a standard nitrogen analysis of mesoporous materials such as catalysts. If the first pressure table point is low and the gas uptake of the sample is expected to be high, this option can shorten the time required to reach the first point on the pressure table.</p> <p>The sample is dosed repeatedly at low pressures with a specified amount of gas until the first pressure point is reached. This initial dosing quickly meets the adsorptive demand of the sample.</p> <p>The first point on the pressure table is the threshold value, triggering the transition from the <i>Fixed Dose Mode</i> to <i>Pressure Table Mode</i>. When the first pressure table value is reached, <i>Fixed Dose Mode</i> is disabled, and points are equilibrated and recorded in accordance with the specified pressure table.</p> <p>In the text box, enter the amount of gas to be added to the sample for each dose cycle.</p>


Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	<ul style="list-style-type: none"> Low Pressure incremental dose mode. Select when performing an analysis of microporous materials. At low pressures on Type 1 isotherms, the pressure points are very closely spaced, making a useful pressure table difficult to define. When enabled, equilibrium points are measured at approximately equal intervals on the quantity adsorbed axis. Each dose is fully equilibrated and recorded as a data point. In this mode, the sample is successively dosed with a specified amount of gas until the first pressure point is reached. The first point is the threshold value, triggering the transition from <i>Incremental Dose Mode</i> to <i>Pressure Table Mode</i>. When the first pressure table value is reached, <i>Incremental Dose Mode</i> is disabled, and points are recorded in accordance with the specified pressure table. Because the data points recorded during <i>Incremental Dose Mode</i> may define most of the analysis, one point on the pressure table can be sufficient and serve as the end point for the analysis. Maximum volume increment. Select to determine when additional data points are collected between target pressures in regions of adsorption. When the maximum increment has been adsorbed since the last collected data point, another point is equilibrated and collected. During desorption, this field is treated as a maximum volume decrement value.
Equilibration	Provides a table for entering relative pressures and equilibration intervals. <div data-bbox="522 1194 998 1677" data-label="Image">  </div> <ul style="list-style-type: none"> Equilibration interval. The number of seconds between successive pressure readings during equilibration. Long equilibration intervals tend to

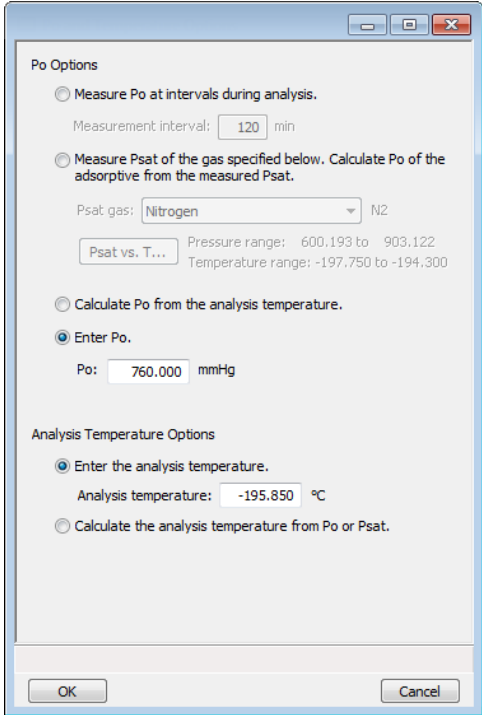
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	<p>lengthen analyses, however, they do improve data integrity. Short equilibration intervals produce a faster analysis but may reduce the accuracy of data.</p> <ul style="list-style-type: none"> • Minimum equilibration delay at $p/p_o \geq 0.995$. 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 <i>Absolute pressure dosing</i> is selected on the <i>Analysis Conditions</i> tab.
Free Space	<p>Use to enter the free space measurement type.</p> <div data-bbox="527 640 1003 1312" data-label="Image"> </div> <ul style="list-style-type: none"> • Measure <p>Lower dewar for evacuation. If the dewar is to be lowered for evacuation, select this options and enter the length of time for evacuation after the free-space measurement in the <i>Evacuation time</i> field.</p> <p>Evacuation time. The length of evacuation time prior to free space measurement.</p> <p>Outgas test. Checks for system leaks or sample outgassing. After free space is measured, the dewar is lowered and the sample evacuated for the specified amount of time. The leak test is performed after evacuation. If a leak is found, the leak test repeats nine times, with 30 minutes evacuation between tests. If the 10th leak check fails, the</p>

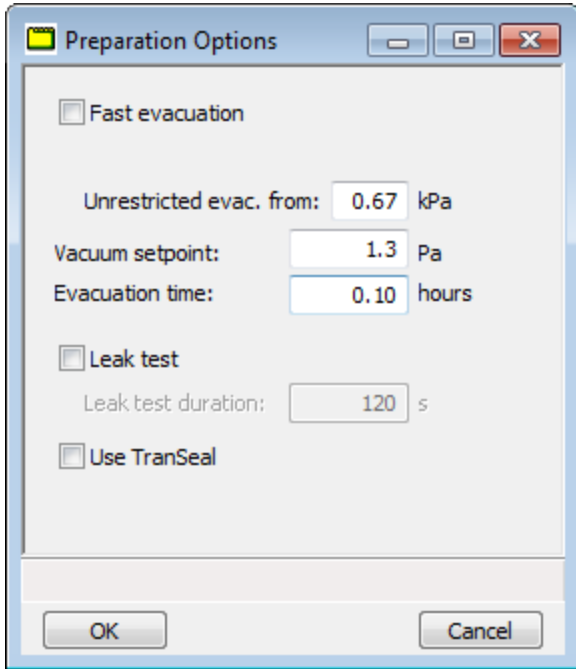
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	<p>analysis stops and the operator is notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak occurs.</p> <ul style="list-style-type: none"> • Enter. Allows manual entry of warm free space and cold free space. • Calculate. Use to have the free space measurement calculated using the sample and tube parameters.
Insert Range	<div>  <p>To insert an absolute pressure range, select <i>Absolute pressure</i> dosing on the Analysis Conditions window.</p> </div> <p>Click to display the <i>Insert Pressure Range</i> window for entering parameters for the system to autofill the <i>Up to</i> 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> <ul style="list-style-type: none"> • Ending relative pressure. The last pressure added to the table. • Geometric from low pressure. Added pressure points will be spaced farther apart as the pressures get higher. • Geometric towards saturation. Added pressure points will be spaced closer together as the pressures get higher (closer to saturation). • Linear. Inserts evenly spaced points into the table. • Number of points. Number of points to be taken between the specified starting and ending relative pressures. • Starting relative pressure. The first pressure added to the table.
P ₀ and Temperature Options	Use to select options for obtaining the saturation pressure (P ₀) and analysis bath temperature.


Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	 <ul style="list-style-type: none"> • Analysis Temperature Options.Select an option to enter analysis temperature manually, or choose to have it automatically calculated from p° or P_0. • p° Options.Select one option indicating how P_0 is to be measured or calculated. • Psat Gas.If choosing to measure the P_0 for each isotherm point using a gas other than the adsorptive, select the P_0 gas from the drop-down list..
Preparation	Use to enter analysis preparation details.

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	 <p>The Preparation Options dialog box contains the following fields and buttons:</p> <ul style="list-style-type: none"> <input type="checkbox"/> Fast evacuation Unrestricted evac. from: 0.67 kPa Vacuum setpoint: 1.3 Pa Evacuation time: 0.10 hours <input type="checkbox"/> Leak test Leak test duration: 120 s <input type="checkbox"/> Use TranSeal OK button Cancel button
	<ul style="list-style-type: none"> • Evacuation time.The length of time for preliminary evacuation. • Fast evacuation. Select for samples (such as pellets) that do not fluidize or shed particles during evacuation. • Unrestricted evac. from. The pressure at which unrestricted evacuation is to begin. • Leak test.Enables the system to check for leaks or sample outgassing before the analysis. The leak test allows sample pressure to rise during the test. If the pressure rises more than 0.15 mmHg, the analysis does not proceed and the operator is notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak exists. • Use TranSeal.Select if using the TranSeal to transfer the sample from the preparation port to the analysis port under vacuum. • Vacuum setpoint.The vacuum level to be achieved before timed evacuation begins.

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

ADSORPTIVE PROPERTIES

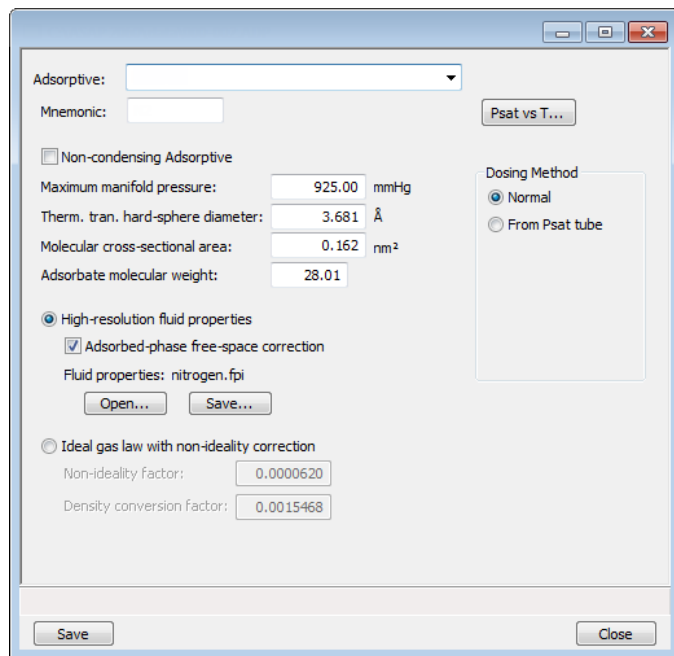
File > Open > [.ADP File]

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

(or click **Edit** next to the *Adsorptive* selection on the *Analysis Conditions* tab when in *Advanced* option presentation)

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

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




3. Enter the mnemonic for the adsorptive gas (for example, N2) in the *Mnemonic* text box. If this gas is connected to a gas inlet port, this mnemonic must be entered in the *Gas Configuration* for the inlet port. See [Specify Gas Ports on page 12 - 5](#).

4. Adsorbed molecules occupy volume in the sample tube, reducing the cold free space. Select the *High-resolution fluid properties* > *Adsorbed-phase free-space correction* option 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.
5. To import parameters from a *Fluid Properties* file, click **Open**, locate and select the .FPI file containing the new parameters, then click **Open**. Click **Save** to save the changes. Changing fluid properties should only be necessary if an adsorptive is to be used for which no adsorptive properties are provided or at an analysis temperature not covered by the standard properties. Contact Micromeritics Scientific Services if new fluid properties are required (<http://tech-support.micromeritics.com/portal>)
6. In the *Dosing Method* group box, select the source to dose the adsorptive.
7. Click **OK**, then click **Close**.

Adsorptive Properties Fields and Buttons Table

Field or Button	Description
Adsorbate molecular weight	The molecular mass is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot.
Adsorbed phase free-space correction	Adsorbed molecules occupy volume in the sample tube, reducing the cold free space. Select the <i>Adsorbed-phase free-space correction</i> checkbox to adjust the reported quantity adsorbed to correct for this effect. This option is appropriate for all sample analyses that use the real gas equation of state.
Adsorptive	Name of the adsorptive gas whose properties are being defined.
Density Conversion Factor	Factor determined by obtaining the ratio of the gas density (STP) to the liquid volume. This field is disabled if <i>Non-condensing Adsorptive</i> is selected.
Dosing Method	<ul style="list-style-type: none"> • Normal. Dose from a pressurized tank of gas attached to a gas inlet port. • From Psat tube. Select if the Psat tube is to be filled with condensed adsorptive and dosed from the Psat tube. Select this option if using Krypton.
High-resolution fluid properties	Use to import parameters from a <i>Fluid Properties</i> file. Click Open to browse and select a .FPI file. Locate and select the file, then click Open on the file selector window. Click Save to save the changes made from the importing selected the .FPI file. Changing fluid properties should only be necessary if an adsorptive is to be used for which no adsorptive properties are provided.
Ideal gas law with non-ideality correction	Use if there is no compressibility table for the gas being used or if you want to match existing data. This factor adjusts the ideal gas law for calculating the quantity of gas in the cold free space. Most gases are nearly ideal near room temperature and at pressures not much above atmospheric pressure, so the cold free space is where the correction is

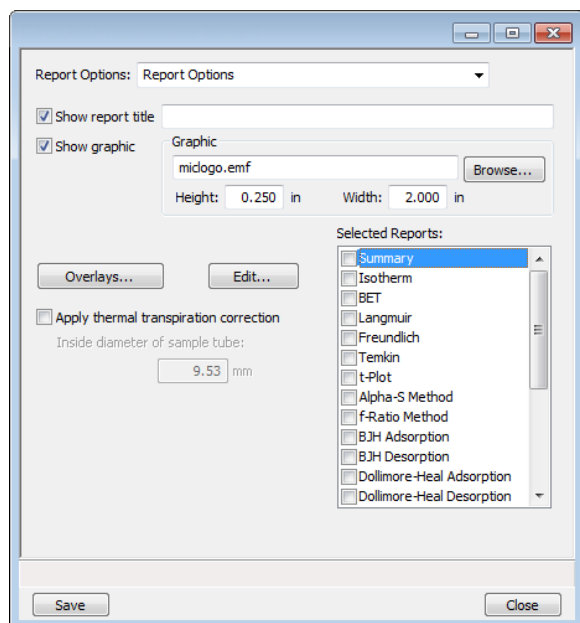
Adsorptive Properties Fields and Buttons Table (continued)

Field or Button	Description
	most important. If selected, enter the <i>non-ideality factor</i> field.
Maximum manifold pressure	The highest pressure to which the manifold will be dosed. To avoid damage to the analyzer, this number is limited to 925 mmHg. Low pressure sources will require lower numbers. For gases to be used for dosing after charging a tube from a gas inlet, enter the maximum pressure for dosing from the inlet, not from the tube of condensate.
Mnemonic	Enter the mnemonic name for the adsorptive. If this gas is connected to a gas inlet port, this mnemonic must be entered in the <i>Unit Configuration Gas Selection</i> for the inlet port. See Specify Gas Ports on page 12 - 5 .
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.
Non-condensing Adsorptive	Select if using a non-condensing analysis gas.
Psat and T	Click to edit the <i>Psat vs Temperature</i> table. The table contains saturation pressures and their corresponding temperatures. To edit, click in a field and enter the value.
Therm. tran. hard-sphere diameter	An estimate of molecular size used in calculating the thermal transpiration correction.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

REPORT OPTIONS

File > Open > [.RPO File]

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



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

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


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

1. Go to **File > Open**.

- Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
- Select the appropriate file type from the drop-down list on the lower right portion of the window, then select a file name in the list or enter it in the *File Name* field, then click **Open**.

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

Report Options Fields and Buttons Table

Field or Button	Description
Apply thermal transpiration correction	<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 approximately 1.0 mmHg.</p> <p>Always use thermal transpiration when performing micropore analyses. See Thermal Transpiration Correction on page B - 44.</p> <ul style="list-style-type: none"> • Inside diameter of sample tube. Enabled when <i>Apply thermal transpiration correction</i> is selected. Enter the inside diameter of the sample tube used in the analysis.
Overlays	See Graph and Sample Overlays on page 7 - 29 .
Report Options drop-down list	Browse for a .RPO file that contains report options parameters to be used in the report.
Selected Reports list box	Select the report names to include in the report.
Show graphic	<p>Use to show a graphic on the report header. Click Browse to locate the graphic.</p> <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.
Show report title	Select and enter a report title to appear on the report header.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

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5 ABOUT DEGASSING

Most solid materials absorb moisture and other contaminants when exposed to the atmosphere. The sample must be clean when an analysis is performed. The degas process heats the sample with an inert gas flowing over it to remove the moisture and contaminants.

DEGAS ON THE SMARTPREP

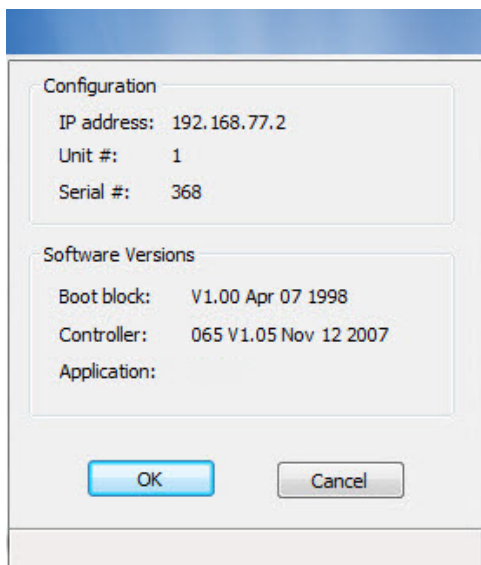
Unit [n] > Degas

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

SMARTPREP CONFIGURATION

Unit [n] > Degas > SmartPrep Configuration

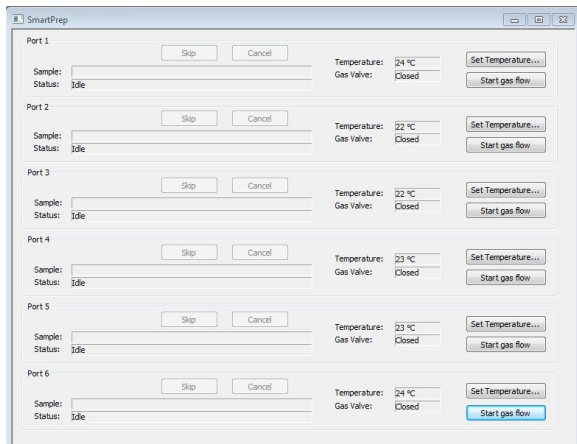
Displays the SmartPrep configuration and software versions.



SHOW SMARTPREP STATUS

Unit [n] > Degas > Start SmartPrep Degas

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



The SmartPrep window displays six ports, each with a 'Skip' and 'Cancel' button. Below these are fields for 'Sample' and 'Status' (both showing 'Idle'). To the right of each port are 'Temperature' and 'Gas Valve' fields, both showing 'Closed'. Further right are 'Set Temperature...' and 'Start gas flow' buttons. The 'Start gas flow' button for Port 6 is highlighted in blue.

Show SmartPrep Status Fields and Buttons Table

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

START SMARTPREP DEGAS

Unit [n] > Degas > Start SmartPrep Degas

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



The Automatic Degas window shows six rows, each with a 'Sample' field and a 'Degas conditions' dropdown menu. To the right of each row are 'Browse...' and 'Clear' buttons. At the bottom of the window are 'Start' and 'Cancel' buttons. A note at the bottom states: '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.'

Start SmartPrep Degas Fields and Buttons Table

Field or Button	Description
Browse	Searches for a file. Select a file from the <i>Name</i> column or from the library, then click Open . Alternatively, double click the file name to open (or import) the file.
Cancel	Discards any changes.
Clear	Clears the sample file selection for a port.
Start	Starts degassing.

DEGAS A SAMPLE ON THE ANALYSIS PORT

The characterization of microporous materials may be significantly improved by degassing the sample on the analysis port before the analysis. To avoid contaminating the analysis system, the sample must be degassed on an external preparation device such as the SmartPrep immediately before attaching it to the sample port.

The sample is heated with a MicroPrep heating mantle controller while under vacuum.



The sample will be open to vacuum for more than 6 hours. If a shared vacuum configuration is used, analyses on the other units may be delayed.

The sample will be open to vacuum for more than 6 hours. If a shared vacuum configuration is used, analyses on the other units may be delayed.

1. Deselect *Backfill sample at start of analysis* in the analysis conditions backfill options.
2. Put an isothermal jacket on the sample tube.
3. Install sample tube on analysis port.
4. Carefully slide the isothermal jacket up while supporting the bottom of the tube.
5. Rotate the P_{sat} tube out of the way.



The isothermal jackets on the sample tube and P_{sat} tube may be damaged if they are close to the heating mantle.

6. Enable manual control.
7. Open valves 2, 7, and 9 to evacuate the sample.
8. Allow sample pressure to reach 10 mmHg then open valve 1.
9. Attach the heating mantle to the sample tube.
10. Set the MicroPrep to 250 °C. Heat for 6 hours.

WHEN DEGASSING IS COMPLETE

1. Turn off the MicroPrep. Do not backfill the sample; leave it under vacuum.
2. Remove the heating mantle.



Be careful, the metal clip and grommets may be hot.

3. When the tube is cool enough to touch, carefully slide the isothermal jacket down while supporting the bottom of the tube. Sliding the jacket down while the tube is hot may damage the jacket.
4. Rotate the P_{sat} tube into position next to the sample tube and install the dewar lid.
5. Ensure that valves 1, 2, 7, and 9 are still open and start the analysis.

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6 PERFORM AN ANALYSIS

DEWAR PRECAUTIONS



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

When handling dewars containing liquefied gases or cryogenic liquids:

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

For glass dewars:

- Use a plastic stirring rod when stirring substances in a dewar containing liquefied gases (or other materials of extremely low temperature). Do not use a glass or metal stirring rod unless it has a protective coating.
- Do not handle heavy objects above the dewar. If unavoidable, place a protective cover over the dewar opening. If an object of sufficient weight is accidentally dropped into the dewar, shattering may occur.
- Do not remove the protective mesh covering. This cover minimizes the risk of flying particles should the dewar be knocked over, dropped, or broken.

PREPARE FOR ANALYSIS

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

Select a Sample Tube below

Step 1 - Clean and Label Sample Tubes on page 6 - 4

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

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

Step 4 - Degas the Sample on page 6 - 7

Step 5 - Install the Sample Tube on page 6 - 8

Step 6 - Fill and Install the Dewar on page 6 - 9

SELECT A SAMPLE TUBE

A sample tube set consists of:

- Sample tube
- Stopper or seal frit
- Filler rod

Standard sample tubes for the ASAP 2060 instrument have a 1.27 cm (1/2 in.) outside diameter (OD). Stepped ferrules, smaller O-rings, isothermal jackets, and filler rods are available for adapting the smaller stems to the degas or analysis ports. The stem diameter selected for use is a matter of accuracy and precision requirements, as well as personal preference and convenience in loading the sample.

A rubber stopper may be used with all size sample tubes; however, seal frits are recommended for 1.27 cm (1/2 in.) OD sample tubes.

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.



Filler rods can interfere with thermal transpiration correction and, therefore, should not be used when performing micropore analyses.

The weight of the empty sample tube should be determined after it has been cleaned, degassed, and filled with backfill gas. The sample tube should be allowed to cool to room temperature before backfilling. After the sample tube has cooled, remove it from the degas port and weigh it.



If a seal frit is not used, insert a stopper immediately after removing the sample from the degas port.

The mass of the isothermal jacket may vary slightly and cannot be considered as constant; therefore, do not weigh it with the sample tube set.

STEP 1 - CLEAN AND LABEL SAMPLE TUBES

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



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

Supplied by Micromeritics	Supplied by User
<ul style="list-style-type: none"> • Filler rod • Funnel • Sample data worksheet • Sample tube • Sample tube brush • Sample tube rack • Sample weighing support • Stopper for sample tube (seal frit, rubber stopper, or TranSeal) 	<ul style="list-style-type: none"> • Acetone or isopropyl alcohol • Analytical balance • Detergent (such as Alconox) • Drying oven • Forceps • Insulated gloves • Pipe cleaners • Rubber gloves or lint-free cloth • Safety glasses • Ultrasonic cleaning unit • Waste container

1. Preheat drying oven to 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). If too much detergent is used, it may be difficult to rinse from the sample tubes. Ensure the detergent is dissolved before placing the sample tubes and filler rods into the water.
4. Fill the sample tubes with warm water and place them in the ultrasonic cleaning unit, then place the filler rods in the unit. Turn on the ultrasonic cleaning unit for approximately fifteen minutes.



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



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



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

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



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

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

STEP 2 - CREATE THE SAMPLE FILE

- [Create Sample Files in Advanced Option Presentation on page 3 - 1](#)
- [Create Sample Files in Basic Option Presentation on page 3 - 5](#)
- [Create Sample Files in Restricted Option Presentation on page 3 - 6](#)

STEP 3 - DETERMINE THE SAMPLE MASS

See [Worksheets on page F - 1](#).

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

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

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



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

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

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

Determine Sample Mass

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



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

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

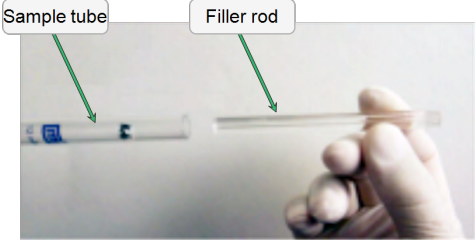


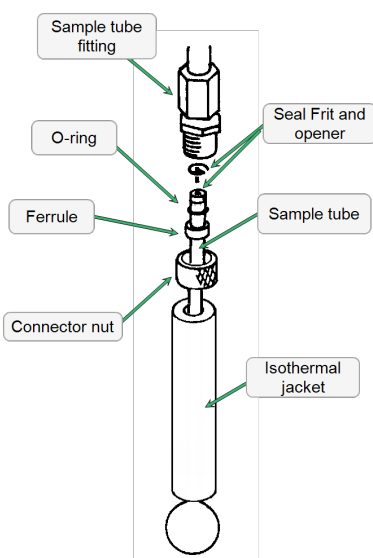
9. Replace the rubber stopper, Check Seal, or TranSeal.
10. Weigh the sample tube set containing the sample and record the value on the *Sample Data Worksheet* as *[B] Sample tube set plus sample mass (Before Degas)*.
11. Subtract the *[A] Mass for empty sample tube set* from the *[B] Mass of sample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

STEP 4 - DEGAS THE SAMPLE

See [Degas on the SmartPrep on page 5 - 1](#).

STEP 5 - INSTALL THE SAMPLE TUBE

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.
A filler rod	Hold the sample tube horizontally and carefully slide the filler rod into the tube. 



1. Loosen the connector nut on the P_0 tube and rotate the P_0 tube out of the way.
2. Install the isothermal jacket.
3. Place the connector nut, ferrule and O-ring onto the sample tube stem.
4. Insert the sample tube into the analysis port and ensure it is completely in the port. Securely hand tighten the connector nut onto the analysis port.
5. Position the dewar lid above the isothermal jacket.

STEP 6 - FILL AND INSTALL THE DEWAR

See [Dewar Precautions on page 6 - 1](#).



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

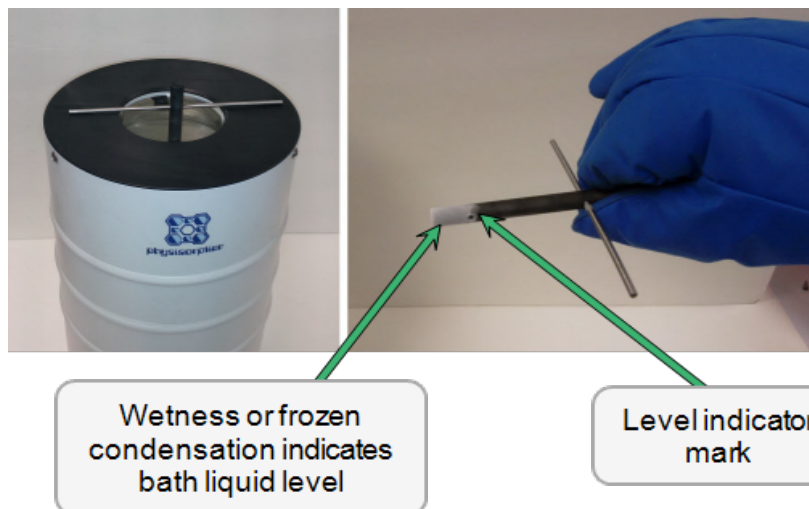


1. Fill the dewar with the analysis bath liquid (such as liquid nitrogen) to no higher than 2 1/4 in. (5.7 cm) from the top. Filling the dewar higher than this will cause an error in the free space measurement.



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

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

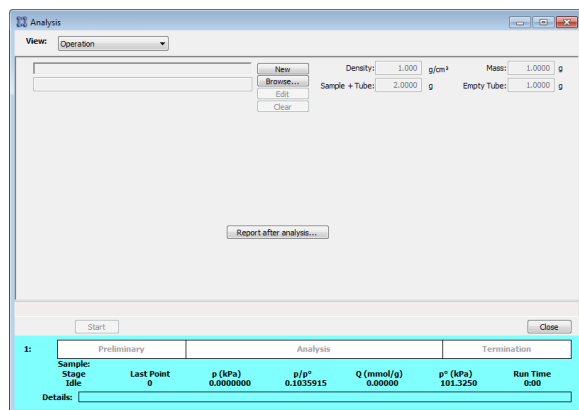


3. For best results, if the dewar has not been used for a while, allow approximately 30 minutes for the temperature of the dewar to stabilize with the bath liquid, then recheck the level of the bath liquid. Add additional liquid if necessary.
4. If using isothermal jackets, slide the jackets down the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

PERFORM A SAMPLE ANALYSIS


Unit [n] > Sample Analysis

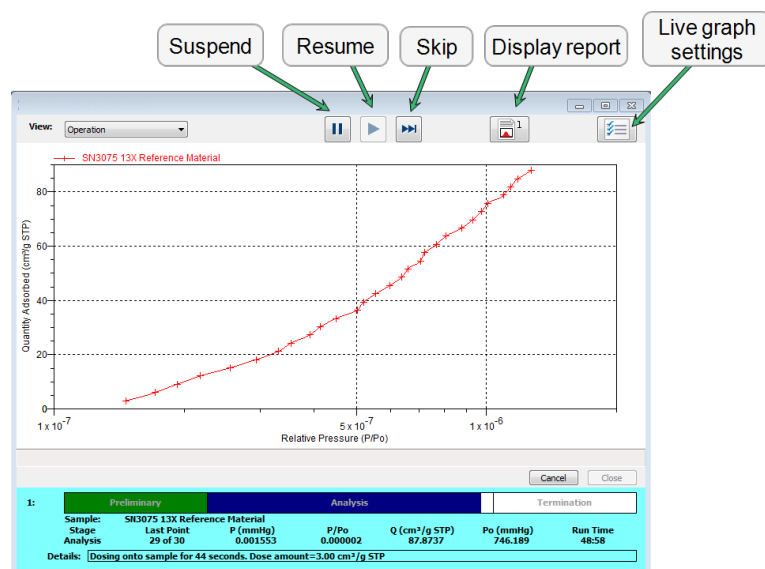
1. Go to *Unit [n] > Sample Analysis*.



2. Click **Browse** and select a sample information file, or click **New** to create a new sample information file.
3. Verify the information populated into the sample identification fields. This information is pulled from the selected or newly created sample file.
4. Click **Report after analysis** to generate reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK** to return to the previous window.
5. 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.
6. When the analysis is complete, remove the sample tube and store (or dispose of) the sample material as applicable.

Analysis Fields and Buttons Table


Field or Button	Description
Density / Mass / Sample + Tube / Empty Tube	Enter values for the sample's mass and density. These values may be edited after analysis. <ul style="list-style-type: none"> • Density • Mass • Sample + Tube • Empty Tube
New	Creates a new sample information file.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	



Sample Analysis Graph Fields and Buttons Table

Field or Button	Description
Live Graph Settings	Select Thermal transpiration, X-axis Quantity (relative or absolute pressure) and the X-Axis Scale (linear or logarithmic).
Report after analysis	Generates reports automatically when the analysis is complete. On the <i>Report Settings</i> window, select the report destination. Click OK to return to the previous window.
Display Port	Generates a report on data being collected. The reports are printed to the screen only.

Sample Analysis Graph Fields and Buttons Table (continued)

Field or Button	Description
Resume	Restarts the suspended analysis.
Skip	Skips to the next step. Select the ports to skip.
Status window	Displays the last point pressure and relative pressure for each port.
Suspend	Suspends an analysis in progress.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

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7 ABOUT REPORTS

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

OPEN AND CLOSE REPORTS

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

Opens saved reports.

Reports > Close Reports

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

START REPORTS

Reports > Start Report

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

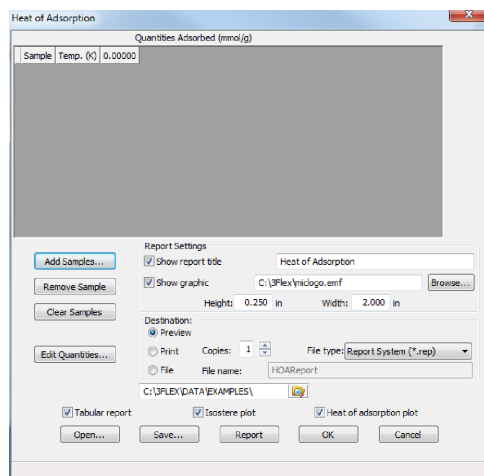


If only one report file was selected in Step 1, the *Selected Reports* window displays allowing the option to select additional reports. Select additional reports as needed, then click **OK**. If multiple files were selected, the reports are displayed in a tiled format.

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

HEAT OF ADSORPTION REPORT

Reports > Heat of Adsorption

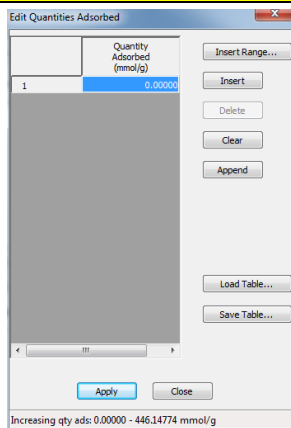



Use to select sample files, define quantities, and generate a *Heat of Adsorption* report. The isosteric heat of adsorption is an important parameter for characterizing the surface heterogeneity and for providing information about the adsorbent and the adsorption capacity. Multiple adsorption isotherms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

Heat of Adsorption Fields and Buttons Table

Field or Button	Description
Add Samples	<p>Adds a sample file to the table.</p> <ol style="list-style-type: none"> 1. Click Add Samples. 2. Double click the file in the <i>Name</i> column. Alternatively, select the file name, then click Open. <p>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.</p>
Clear Samples	Removes all entries from the table.
Edit Quantities	Use to specify the range of surface coverage to include in the report.

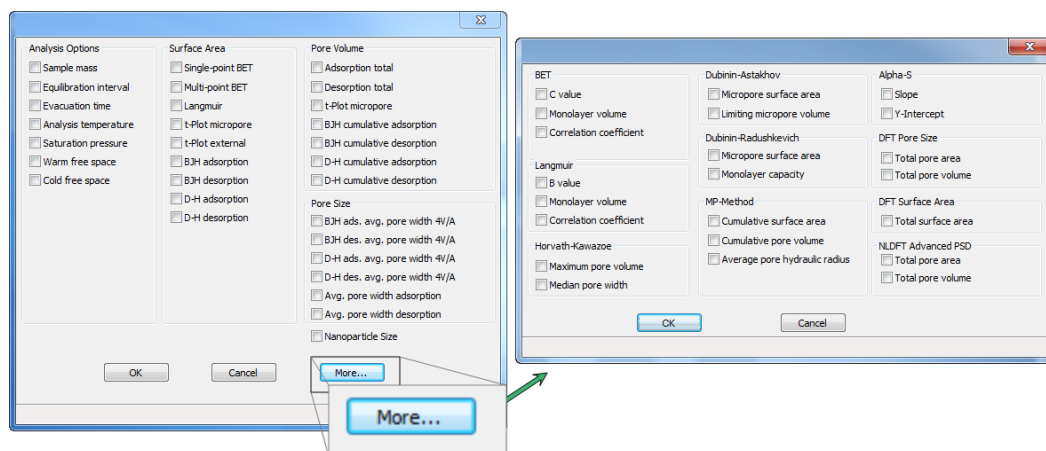
Heat of Adsorption Fields and Buttons Table (continued)

Field or Button	Description
	 <p>Insert Range. Click to specify the starting and ending quantities adsorbed and number of points to insert.</p> <ul style="list-style-type: none"> • Load Table. Imports values from another file. • Save Table. Saves the current table as a .QNT file. • Apply. Applies all table changes.
Heat of adsorption plot	Generates the <i>Heat of Adsorption</i> data in a graphical format.
Isostere plot	Generates a graph showing quantities of gas adsorbed versus the temperature.
Remove Sample	Removes the selected sample from the list.
Show graphic	<p>Use to show a graphic on the report header. Click Browse to locate the graphic.</p> <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.
Show report title	Select and enter a report title to appear on the report header.
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
	<p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>

SPC REPORT

Reports > SPC Report Options

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

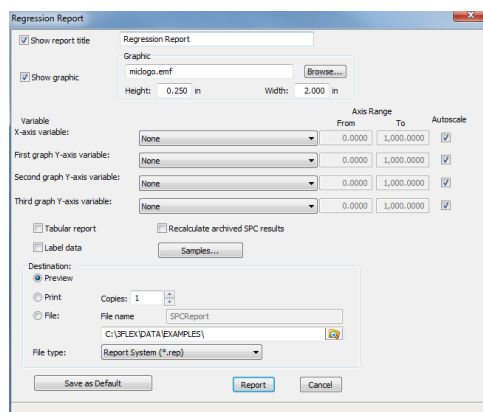


The selected items display as graph variable selections in **Reports > Regression Report** and graph selections in **Reports > Control Chart**. If additional report options are required, click [More](#).


REGRESSION REPORT

Reports > Regression Report


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




Regression Report Fields and Buttons Table

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

Regression Report Fields and Buttons Table (continued)

Field or Button	Description
	<p>speeds up future SPC reports.</p> <p>Do not show me this message again.</p> <hr/> <div>  <p>If <i>Do not show me this message again</i> is selected, the message cannot be redisplayed without Micromeritics assistance.</p> </div> <hr/> <p>The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.</p>
Samples	<p>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.</p> <ul style="list-style-type: none"> • Available Files. Contains files located in the directory specified in the <i>Look In</i> text box. • Selected Files. Files added from the <i>Available Files</i> list box. • Add / Remove. Select a file in the <i>Available Files</i> list box, then click Add to move the file to the <i>Selected Files</i> list box. Or select a file in the <i>Selected Files</i> list box, then click Remove to move the file back to the <i>Available Files</i> list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show graphic	<p>Use to show a graphic on the report header. Click Browse to locate the graphic.</p> <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.
Show report title	Select and enter a report title to appear on the report header.

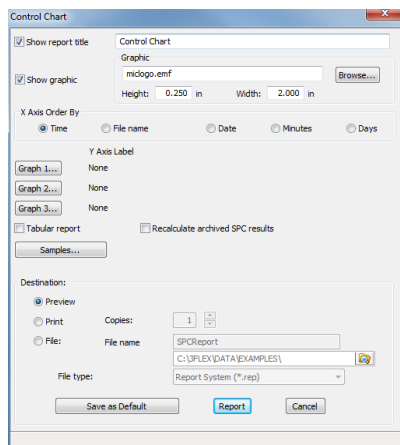
Regression Report Fields and Buttons Table (continued)

Field or Button	Description
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X- and Y-Axis Variable	Use to designate the x- and y-axes variables. The variables in the drop-down lists are those selected in the Reports > SPC Report Options window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

CONTROL CHART REPORT

Reports > Control Chart

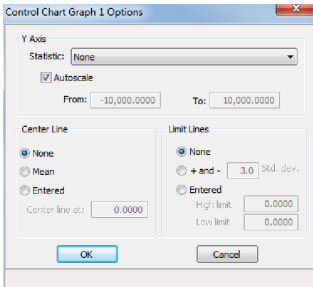

Use to generate a Statistical Process Control (SPC) control chart report which plots the changes in a statistic.




The screenshot shows the 'Control Chart' dialog box with the following settings:

- ☒ Show report title: Control Chart
- ☒ Show graphic: midogo.enf (Browse... button)
- Height: 0.250 in Width: 2.000 in
- X Axis Order By: ☒ Time ☐ File name ☐ Date ☐ Minutes ☐ Days
- Y Axis Label: None None None
- ☐ Tabular report ☐ Recalculate archived SPC results
-
- Destination: ☒ Preview ☐ Print ☐ File
- Copies: 1
- File name: SPCReport
- File type: C:\3PLEX\DATA\EXAMPLES\ Report System (*.rep)
- Buttons: Save as Default, Report, Cancel


Control Chart Fields and Buttons Table

Field or Button	Description
Graph [n]	<p>Click to define the y-axis of each graph.</p>  <ul style="list-style-type: none"> • Statistic. Displays the SPC variables selected on the <i>Reports > SPC Report Options</i> window. The selected variable will be plotted against time. This selection also becomes the y-axis label. • Autoscale. Allows the y-axis to be scaled automatically. To specify a range, deselect this option and enter a range in the <i>From</i> and <i>To</i> fields. • Center Line. Displays placement options for the center line in the graph. Choose <i>Entered</i> 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 (<i>Entered</i>). When <i>Entered</i> is selected, enter the <i>High limit</i> and <i>Low limit</i> fields with appropriate values.
Recalculate archived SPC results	<p>Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report.</p> <hr/> <div>  <p>If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application.</p> </div> <hr/> <p>When this option is selected, the following message displays:</p> <div> <p>Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.</p> <p>Do not show me this message again.</p> </div>

Control Chart Fields and Buttons Table (continued)

Field or Button	Description
	 <p>If <i>Do not show me this message again</i> is selected, the message cannot be redisplayed without Micromeritics assistance.</p> <p>The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.</p>
Report	Generates the report.
Samples	<p>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.</p> <ul style="list-style-type: none"> • Available Files. Contains files located in the directory specified in the <i>Look In</i> text box. • Selected Files. Files added from the <i>Available Files</i> list box. • Add / Remove. Select a file in the <i>Available Files</i> list box, then click Add to move the file to the <i>Selected Files</i> list box. Or select a file in the <i>Selected Files</i> list box, then click Remove to move the file back to the <i>Available Files</i> list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show graphic	<p>Use to show a graphic on the report header. Click Browse to locate the graphic.</p> <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.
Show report title	Select and enter a report title to appear on the report header.

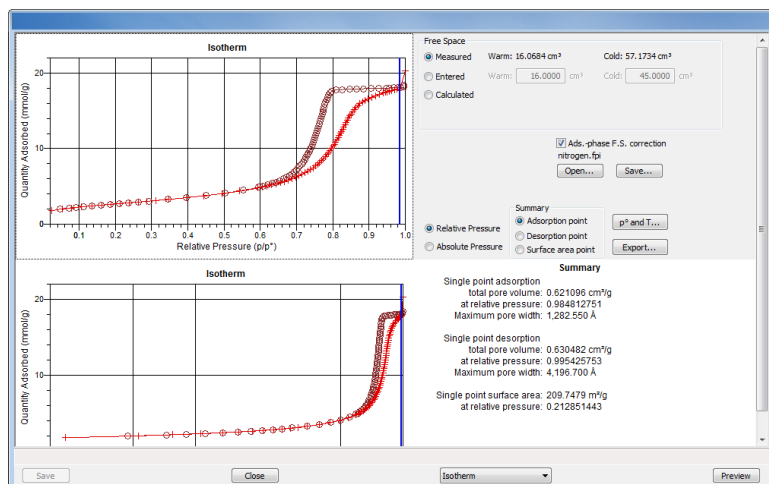
Control Chart Fields and Buttons Table (continued)

Field or Button	Description
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X Axis Order by	<p>Select the order in which x-axis statistics are placed. Sort by:</p> <ul style="list-style-type: none"> • Time. Time the files were analyzed. • File name. Alphanumeric order. • Date. Date the files were analyzed. • Minutes. Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. • Days. Number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

INTERACTIVE REPORTS

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

1. When opening a sample file that contains analysis data, a window with the following information displays:
 - an isotherm linear plot and log plot of the data collected during analysis
 - a summary of the analysis giving a single total pore volume and surface area



2. To view the plots in either relative or absolute pressure, select either the *Relative Pressure* or *Absolute Pressure* option.
3. To view the reports selected for generation during the analysis, click **Preview**.
4. From the drop-down list at the bottom of the window:
 - change the option presentation of the sample information window to either *Basic* or *Advanced* to modify certain file parameters, or
 - select another plot from the list and edit the data contained in the plot.
5. When ranges are edited, the changes are reflected immediately in the plots and the summary data displayed in the window. Some editing options are:
 - Drag the blue bars to increase or decrease the range of data included in the plot.
 - Edit the Isotherm Linear Plot to include or omit the data point from the BET plot.

Right click to display a popup menu to include reports; enable or select overlays; edit curves, axes, legends, titles; and copy and paste the data in a graph or in tabular format.
6. After editing the report, click **Save** to save the changes in the sample information file.

MICROACTIVE REPORTS

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

When a sample information file with a status of , *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. Numerous reports are accessible from a drop-down menu, including:

- Advanced Report
- Alpha-S Method
- BET
- BJH Adsorption
- BJH Desorption
- DFT Pore Size
- DFT Surface Energy
- Dollimore-Heal Adsorption
- Dubinin - Astakhov
- Dubinin - Radushkevich
- f -Ratio Method
- Freundlich
- Horvath-Kawazoe
- Isotherm
- Langmuir
- MP-Method
- NLDFT Advanced PSD
- Options
- Sample Log
- Summary
- Temkin
- t -Plot
- Validation

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

EVALUATE REPORT RESULTS

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

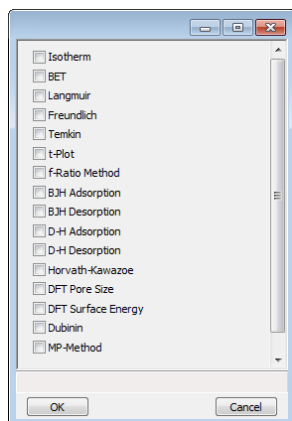
Regardless of the precautions exercised before the analysis, problems still may occur during the analysis, or as a result of using inappropriate parameters or even inappropriate methods. The analysis data should be inspected for evidence of experimental error. The traditional method of confirming the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

Analysis data can be evaluated by:

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

VIEW THE VALIDATION REPORT

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



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

INSPECT THE ISOTHERM PLOT

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

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

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

EVALUATE THE ISOTHERM TABULAR DATA SET

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

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

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

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

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

REVIEW REDUCED DATA

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

PHYSICAL PARAMETERS

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

The area occupied by a single adsorbed molecule is a required parameter in the calculation of surface area by the BET and Langmuir methods. The software provides a default value, but other values are found in the literature. McClellan and Harnsberger²⁾ provide a comprehensive review of such values.

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

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

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

using which, the Kelvin equation³⁾ reduces to

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

²⁾ McClellan, A.L., and Harnsberger, H.F., Journal of Colloid and Interface Science, 23, 577 (1967).

³⁾ Thomson, W., Phil. Mag. S., 42, 448 (1871).

$$\ln \frac{P^*}{P_o} = \frac{A}{r_m}$$

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

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

For terms such as the interaction parameter found in the Horvath-Kawazoe calculation¹⁾, the Dubinin affinity coefficient or Astakhov exponent²⁾, the default values as provided by the software generally are adequate. A search of the technical literature is required if the analysis involves a gas-solid system other than that covered by the default values.

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

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

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

BET C-VALUE

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

¹⁾ Everett, D.H. and Powl, J.C., J. Chem Soc., Faraday Trans. 1, 72, 619 (1976).

²⁾ Dubinin, M. and Radushkevich, L.V., Proc. Acad. Sci. USSR, 55, 331 (1947).

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

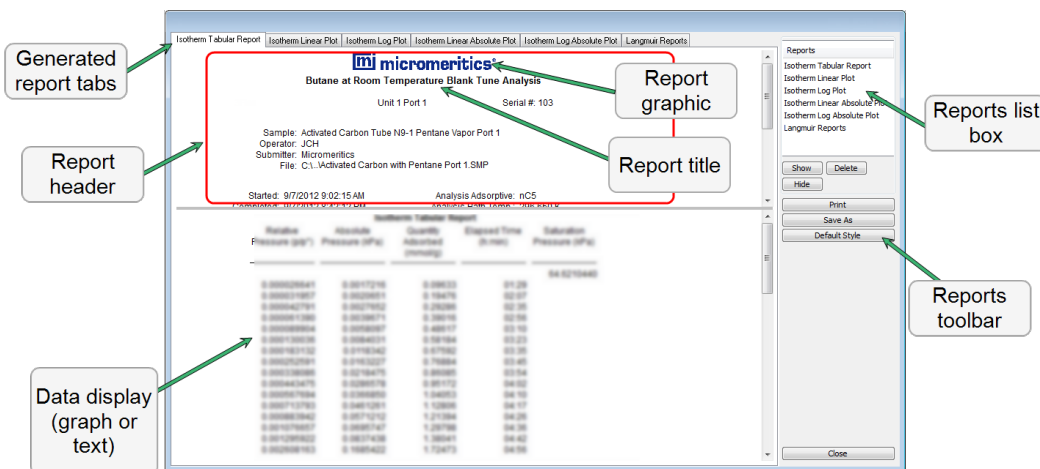
DATA ANALYSES BY THE BJH METHOD

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

REPORT FEATURES AND SHORTCUTS

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

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

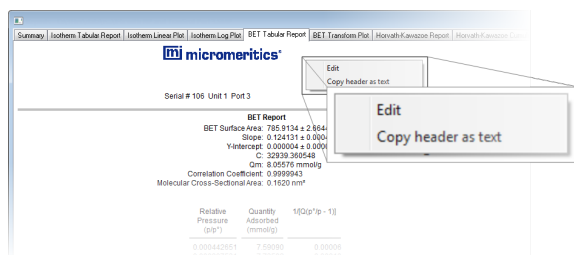


If configured, the report header can also contain a graphic and / or a title.

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

REPORT HEADER SHORTCUTS

Display header shortcuts by right clicking in the report header.

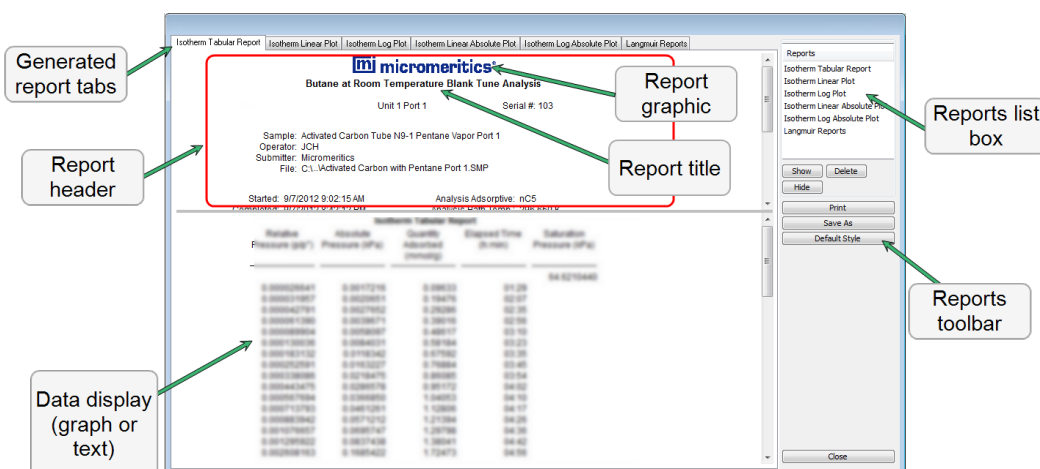


Report header Shortcut Field and Button Table

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

REPORT TOOLBAR


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



Report Toolbar Fields and Buttons Table

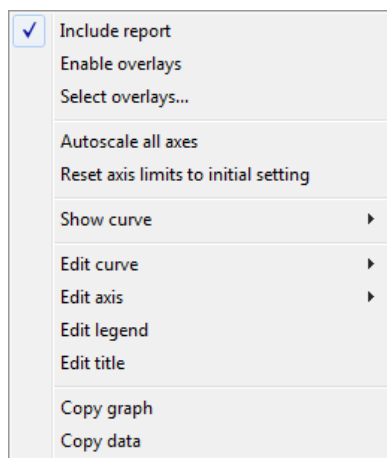
Field or Button	Description
Default Style	Click to specify default report parameters for fonts and curve properties. <ul style="list-style-type: none"> Font Type. Use to edit the font type and attributes for the selected item. Select an item in the list, click Edit, and select from various font options. Thickness. Enter a thickness number for the curve. Histogram Fill Style. Select a histogram fill option. Graph border line thickness. Enter a thickness number for the graph border.
Delete	Deletes the selected report in the <i>Reports</i> list box. Deleted reports will have to be regenerated if deleted in error.
Hide	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the <i>Reports</i> list box.
Print	Displays the <i>Print</i> window for report output. <ul style="list-style-type: none"> Name drop-down list and Properties. Select the printer from the drop-down list and click Properties to change printer setup, etc. Copies. Select the number of copies and collate option. Current. Selects the active report (or selected tab).

Report Toolbar Fields and Buttons Table (continued)

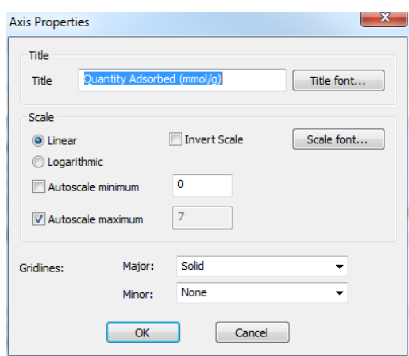
Field or Button	Description
	<ul style="list-style-type: none">• All. Selects all reports in the <i>Reports</i> list box.• Shown. Selects only the reports not hidden.• Clear. Clears all selections.
Reports list box	Contains a list of all generated reports. The same reports display as tabs at the top of the report header unless the report has been hidden using the Hide button.
Show	Displays the selected or hidden report in the <i>Reports</i> list box.
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

GRAPH FEATURES AND SHORTCUTS

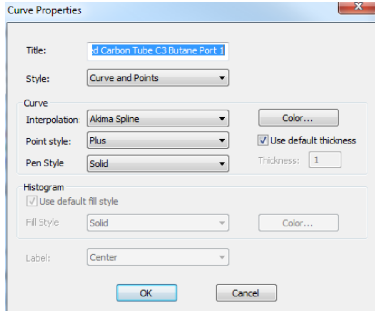
Display graph report shortcuts by right clicking in the body of the graph report.



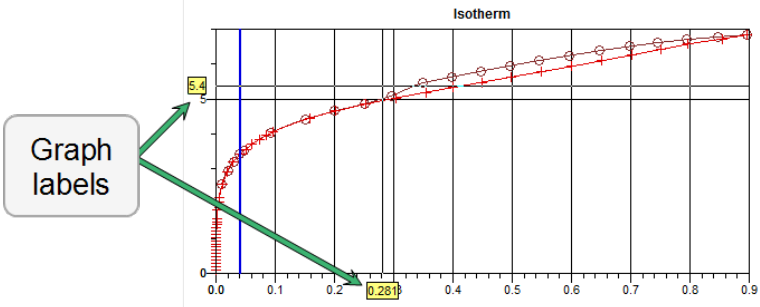
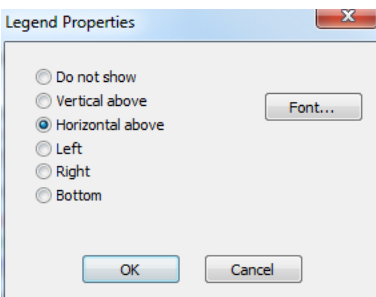
Graph Shortcuts Options and Description Table

Field or Button	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Copy Data	Copies the report data to the clipboard. It can then be pasted into other software programs as tab-delimited columns or copied as an overlay onto another graph.
Copy Graph	Copies the graph to the clipboard. It can then be pasted into other software programs.
Display imported data	Used with pore distribution data reports only. Use to hide or show imported or pasted ASCII text data on the active graph.
Edit axis	Use to edit the selected axis properties. <div data-bbox="620 1312 1026 1665" data-label="Image">  </div> <ul style="list-style-type: none"> • Title. Use to edit the selected axis label.

Graph Shortcuts Options and Description Table (continued)

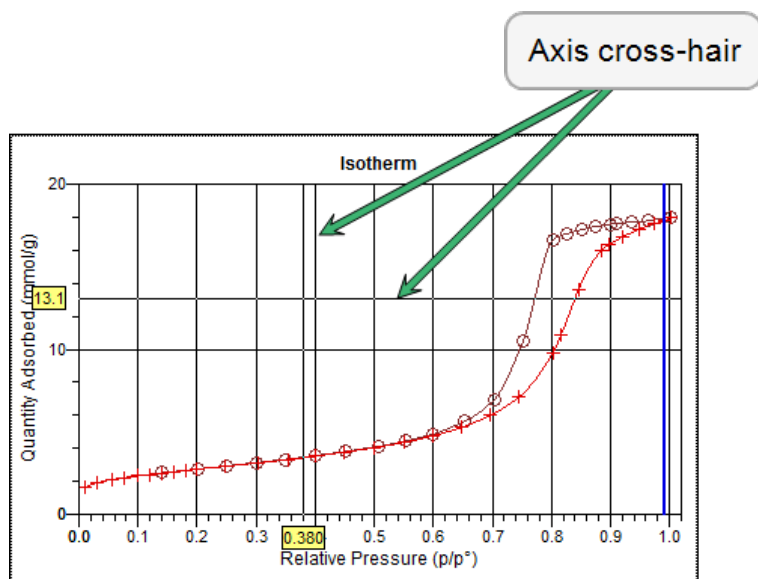
Field or Button	Description
	<ul style="list-style-type: none"> • Title font. Use to modify the font for the selected axis label. Deselect the <i>Use default font</i> to enable font options. Select new font attributes for the report data. To return to the default fonts, enable <i>Use default font</i>. • Linear / Logarithmic. Select the option to scale the graph as linear or logarithmic. • Autoscale minimum / maximum. To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box. • Invert scale. Use to invert the scale. • Scale font. Use to modify the font for the scale label. Deselect <i>Use default font</i> to enable font options. • Grid lines. Use to change how to display major / minor grid lines.
Edit curve	<p>Use to edit selected curve properties.</p>  <ul style="list-style-type: none"> • Title. Use to change the title of the selected curve. • Style. Use to select another style for the collected data curve. • Curve group box. Use to change the interpolation, point style and pen style for the selected curve. These options are disabled if <i>Use default fill style</i> is selected in the <i>Histogram</i> group box. <p>Color. Click to change the curve color.</p> <p>Use default thickness. Uses the default curve thickness. Deselect to enter a new thickness number in the <i>Thickness</i> text box.</p> <ul style="list-style-type: none"> • Histogram group box. Enabled only if <i>Histogram</i> is selected in the <i>Style</i> drop-down list. Use to specify the type of fill, fill color

Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
	<p>and label position for the selected curve.</p> <ul style="list-style-type: none"> Label. Select where the graph point labels will display (left, right, center, etc.) on the SPC report.
	
Edit imported data	Used with pore distribution data reports only. Use to select ASCII text files for import onto the active graph.
Edit legend	<p>Use to change the legend location and font. Click Font to modify font attributes. Deselect the <i>Use default font</i> to enable font options. Select new font attributes for the report data. To return to the default fonts, enable <i>Use default font</i>.</p> 
Edit title	Use to change the graph title and font. Click Font to font attributes. Deselect the <i>Use default font</i> to enable font options. Select new font attributes for the report data. To return to the default fonts, enable <i>Use default font</i> .
Enable Overlays	If overlays have been selected, this option displays (or hides) the overlays.
Include report	When selected, places a checkmark to the left of the report in the <i>Select Reports</i> list box on the <i>Report Options</i> tab.
Paste Data	Used with pore distribution data reports only. Use to paste ASCII text data from the clipboard onto the the active graph.

Graph Shortcuts Options and Description Table (continued)

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

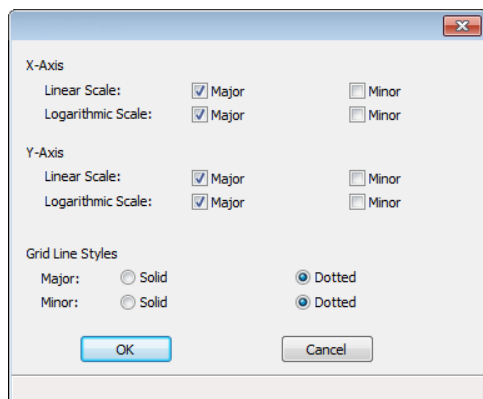
Axis Cross-hair

The cross-hair feature displays axis coordinates.

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

Graph Grid Lines

Options > Graph Grid Lines



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

Graph Grid Lines Fields and Buttons Table

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

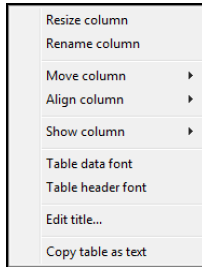
Zoom Feature

Use the zoom feature to examine graph details.

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

TABULAR REPORT FEATURES AND SHORTCUTS

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



Tabular Reports Shortcut Options and Descriptions Table

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

GRAPH AND SAMPLE OVERLAYS

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

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



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

GENERATE PORE-SIZE DISTRIBUTION GRAPH OVERLAYS

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

Multiple graph overlays can only be generated for:

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

ASCII text file format rules:

- 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

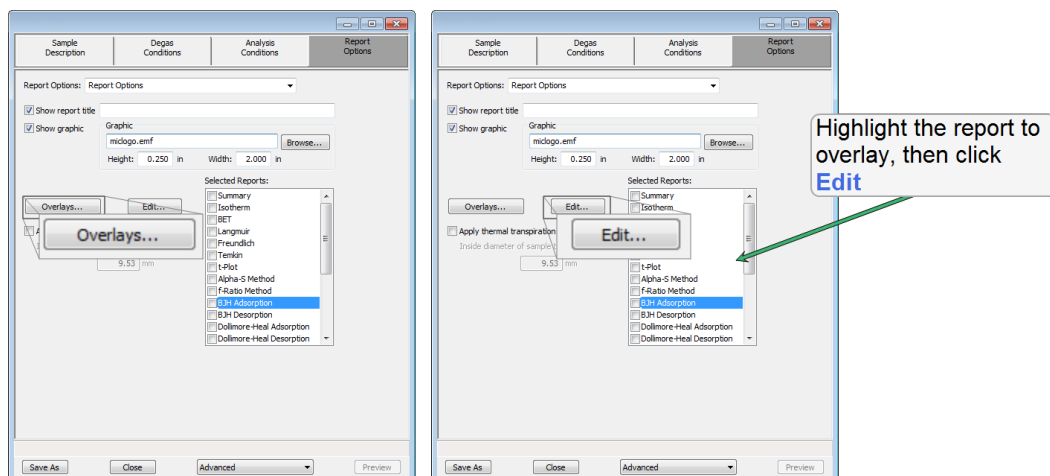
silica alumina bjh	(A, cm ³ /g, cumulative)
456.657	0.0133559
444.847	0.0546427
429.168	0.0869924
425.419	0.119721
419.629	0.132681
360.634	0.156611
340.859	0.197672
326.601	0.233092

To import ASCII text files to generate graph overlays:

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

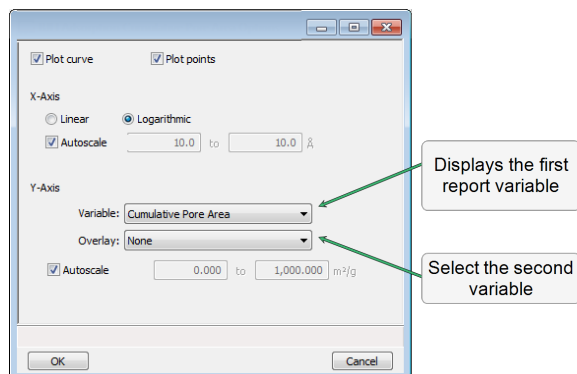
1. Go to **File > Open**. Select a sample file to overlay graphs onto other samples. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. Click **Open**.
2. Select *Advanced* from the drop-down list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

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



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

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

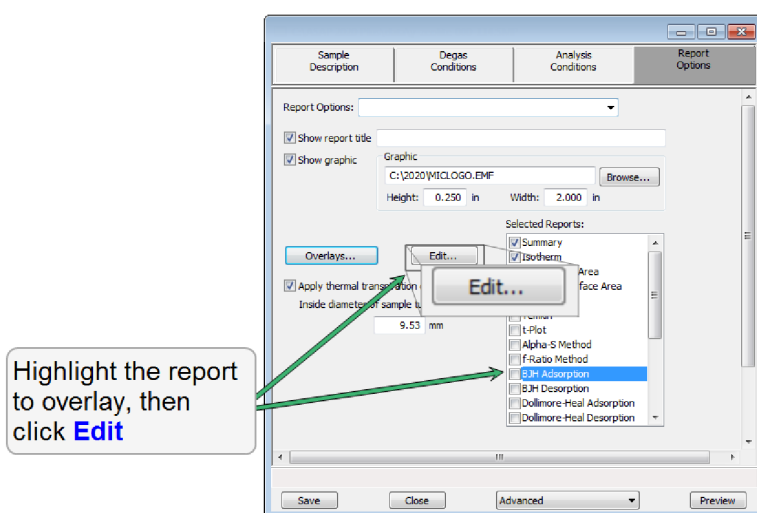


7. Click **OK** again to return to the *Report Options* tab.
8. Click **Save As** to save the selections.
9. To view the report, click **Preview**.

OVERLAY MULTIPLE SAMPLE FILES

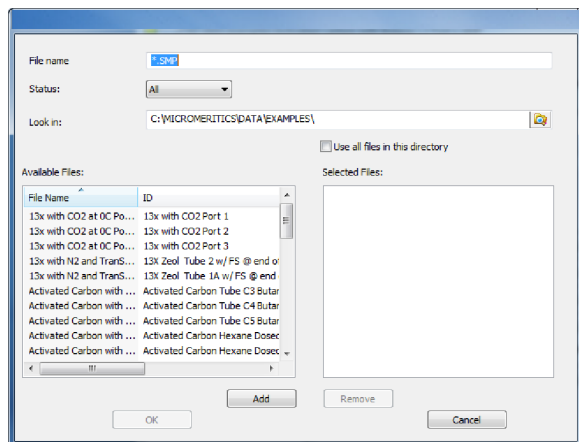
To overlay the same type of graph on multiple samples:

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



If overlaying this type of report...	Then...
<ul style="list-style-type: none"> • Isotherm 	<ol style="list-style-type: none"> On the <i>Isotherm Report Options</i> window, select one or more plots in the <i>Selected Reports</i> group box, then click Options to the right of the selected plot. On the <i>Plot Options</i> window, select <i>Plot curve</i> and / or <i>Plot points</i> if they are to be included in the overlay. If the x- and / or y-axes are to be autoscaled, enable <i>Autoscale</i>; otherwise, enter the <i>From</i> and <i>To</i> points for the axes. Click OK to save and close the window. On the <i>Isotherm Report Options</i> window, in the <i>Plot Options</i> group box, select <i>Plot overlays</i>. Click OK. Continue to Step 5.
<ul style="list-style-type: none"> • Alpha-S Method • BET Surface Area • <i>f</i>-Ratio Method • Freundlich • Langmuir Surface Area • <i>t</i>-plot • Temkin 	<ol style="list-style-type: none"> On the pop-up window, select <i>Overlay samples</i>. Verify other fields. Click OK to return to the <i>Report Options</i> tab. Continue to Step 5.
<ul style="list-style-type: none"> • BJH Adsorption • BJH Desorption • Dollimore-Heal Adsorption • Dollimore-Heal Desorption • MP-Method 	<ol style="list-style-type: none"> Select the report variable from the <i>Selected Reports</i> group box, then click Edit. Click the down arrow on the <i>Overlay</i> field, then select the <i>Samples</i> option. Verify other fields. Click OK to return to the <i>Report Options</i> window. Click OK again to return to the <i>Report Options</i> tab.

- On the *Report Options* tab, click **Overlays**.
- 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:
 - Click **Move Up**.
 - Click **Move Down**.
 - Click **Move All**.



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

IMPORT ASCII PORE DISTRIBUTION DATA

Import an ASCII Text File using Graph Shortcuts

1. Create an ASCII text file. See [Graph and Sample Overlays on page 7 - 29](#).
2. Open a report with a *Complete* status.
3. Select a pore-size distribution report from the drop-down list at the bottom of the window.
4. Right click on the graph and select *Edit imported data* on the shortcut menu.

If the ASCII text file does not display on the *Selected Imported Overlays* window, click **Import**. Locate and select the file, then click **Open**. Header information from the ASCII text file will appear in the *Select Imported Overlays* window. Select the entry, then click **OK**. If an error message appears, verify that the .TXT file format is correct.
5. To hide or show imported data, right click in the graph area and use the *Display imported data* option on the shortcut menu.

Copy / Paste an ASCII Text File using Graph Shortcuts

1. Create an ASCII text file. See [Graph and Sample Overlays on page 7 - 29](#).
2. Copy the ASCII text data to the clipboard.
3. Open a report with a *Complete* status.
4. Select a pore-size distribution report from the drop-down list at the bottom of the window.
5. Right click on the graph and select *Paste data* on the shortcut menu.
6. To hide or show imported data, right click in the graph area and use the *Display imported data* option on the shortcut menu.

Copy / Paste Graph Data from Another Graph

1. Open a source pore distribution data report with a *Complete* status.
2. Right click on the graph and select *Copy Data* on the shortcut menu.
3. Open the target pore distribution data report.
4. Right click on the graph and select *Paste Data* on the shortcut menu.
5. To hide or show imported data, right click in the graph area and use the *Display imported data* option on the shortcut menu.

REPORT EXAMPLES***HORVATH-KAWAZOE DIFFERENTIAL PORE VOLUME PLOT***

ASAP 2060

ASAP 2020 Plus
Serial # 3001 Unit 1

Page 1

Sample: Silica-Alumina S/N 3001
Operator: HEH
Submitter:
File: ...\\Silica Alumina Reference Material with Nitrogen...

Started: 10/31/2014 4:25:41 PM	Analysis adsorptive: N2
Completed: 11/1/2014 9:11:46 AM	Analysis bath temp.: -196.046 °C
Report time: 4/22/2015 1:10:30 PM	Thermal correction: No
Sample mass: 0.2363 g	Warm free space: 16.8084 cm ³ Measured
Cold free space: 49.3399 cm ³	Equilibration interval: 10 s
Low pressure dose: None	Sample density: 1.000 g/cm ³
Automatic degas: Yes	

Comments: Nitrogen on silica-alumina reference material. This is an example of a BJH adsorption/desorption analysis.

ISOTHERM LOG REPORT



Silica Alumina

ASAP 2060

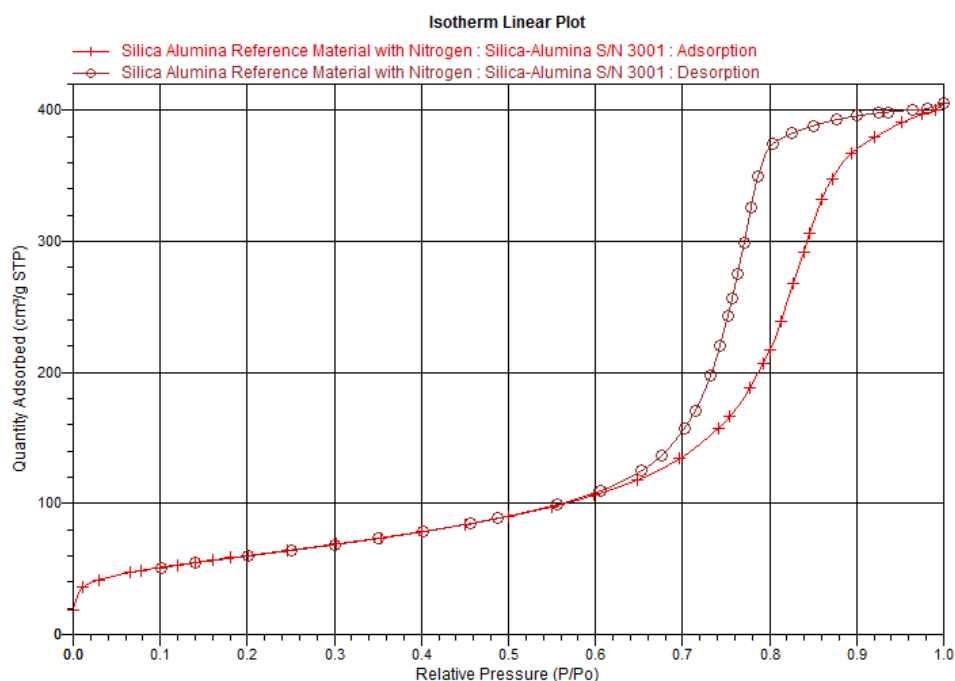
ASAP 2020 Plus
Serial # 3001 Unit 1

Page 1

Sample: Silica-Alumina S/N 3001
Operator: HEH
Submitter:
File: ...Silica Alumina Reference Material with Nitrogen...

Started: 10/31/2014 4:25:41 PM	Analysis adsorptive: N ₂
Completed: 11/1/2014 9:11:46 AM	Analysis bath temp.: -196.046 °C
Report time: 4/22/2015 1:24:46 PM	Thermal correction: No
Sample mass: 0.2363 g	Warm free space: 16.8084 cm ³ Measured
Cold free space: 49.3399 cm ³	Equilibration interval: 10 s
Low pressure dose: None	Sample density: 1.000 g/cm ³
Automatic degas: Yes	

Comments: Nitrogen on silica-alumina reference material. This is an example of a BJH adsorption/desorption analysis.



ISOTHERM TABULAR REPORT



ASAP 2060

ASAP 2020 Plus
Serial # 3001 Unit 1

Page 1

Sample: Silica-Alumina S/N 3001
 Operator: HEH
 Submitter:
 File: ...Silica Alumina Reference Material with Nitrogen...

Started: 10/31/2014 4:25:41 PM Analysis adsorptive: N2
 Completed: 11/1/2014 9:11:46 AM Analysis bath temp.: -196.046 °C
 Report time: 4/22/2015 1:20:50 PM Thermal correction: No
 Sample mass: 0.2363 g Warm free space: 16.8084 cm³ Measured
 Cold free space: 49.3399 cm³ Equilibration interval: 10 s
 Low pressure dose: None Sample density: 1.000 g/cm³
 Automatic degas: Yes

Comments: Nitrogen on silica-alumina reference material. This is an example of a BJH adsorption/desorption analysis.

Isotherm Tabular Report				
Relative Pressure (P/Po)	Absolute Pressure (mmHg)	Quantity Adsorbed (cm ³ /g STP)	Elapsed Time (h:min)	Saturation Pressure (mmHg)
0.000145515	0.107397	19.0457	01:11	737.678772
0.010251690	7.566013	35.9704	02:00	738.051819
0.029498596	21.773464	41.7878	02:39	738.025879
0.065256389	48.164612	47.3856	02:48	738.118652
0.077847531	57.458382	48.8575	02:58	738.082703
0.099974516	73.785477	51.2015	03:02	738.088684
0.119623994	88.283333	53.1109	03:07	738.042847
0.139612656	103.038452	55.9512	03:12	738.006897
0.160051709	118.121658	56.7634	03:16	738.030884
0.180230232	133.022308	58.5092	03:21	738.021851
0.200381535	147.907745	60.2211	03:25	738.068787
0.246373293	181.856903	64.1403	03:30	738.130615
0.302200927	223.054718	69.0442	03:36	738.135620
0.353435506	260.815643	73.8073	03:42	738.100708
0.400065249	295.267273	78.4571	03:48	737.944092
0.449772108	331.928192	83.9297	03:54	738.047791
0.499766434	368.809662	90.1663	04:01	737.991943
0.549428156	405.528381	97.4021	04:09	737.964050
0.598835392	441.993042	106.2761	04:18	738.091736
0.648185004	478.429688	117.8782	04:28	738.087708
0.696828966	514.388306	134.0569	04:39	738.106689
0.742047444	548.028442	157.5955	04:53	738.184448
0.754149258	557.041321	166.1242	05:17	738.535583
0.777757789	574.718384	187.8578	05:30	738.635376
0.792595361	585.631897	206.7200	05:52	738.942627
0.800523802	591.579468	216.8606	06:11	738.878784
0.813262182	600.999512	238.9143	06:25	738.990479
0.827529693	611.618286	268.2593	06:50	738.998474
0.839238954	620.231445	292.0869	07:11	739.089233
0.846139970	625.242126	306.0871	07:30	739.040344
0.859492409	634.935486	331.6346	07:46	738.934631
0.871496999	643.728088	348.0035	08:06	738.733093
0.894621336	660.483032	367.4664	08:23	738.646362
			08:40	738.282227

OPTIONS REPORT

Silica Alumina

ASAP 2060

ASAP 2020 Plus
Serial # 3001 Unit 1

Page 1

Sample: Silica-Alumina S/N 3001
Operator: HEH
Submitter:
File: ...Silica Alumina Reference Material with Nitrogen...

Started: 10/31/2014 4:25:41 PM	Analysis adsorptive: N2
Completed: 11/1/2014 9:11:46 AM	Analysis bath temp.: -196.046 °C
Report time: 4/22/2015 1:30:04 PM	Thermal correction: No
Sample mass: 0.2363 g	Warm free space: 16.8084 cm ³ Measured
Cold free space: 49.3399 cm ³	Equilibration interval: 10 s
Low pressure dose: None	Sample density: 1.000 g/cm ³
Automatic degas: Yes	

Comments: Nitrogen on silica-alumina reference material. This is an example of a BJH adsorption/desorption analysis.

Sample Information

Method: Default
Sample: Silica-Alumina S/N 3001
Operator: HEH
Submitter:
Mass type: Calculated
Empty tube: 50.2824 g
Sample + tube: 50.5187 g
Sample mass: 0.2363 g
Density: 1.000 g/cm³
Type of data: Automatically collected
Instrument type: 2060
Original instrument type: 2020
Comments: Nitrogen on silica-alumina reference material. This is an example of a BJH adsorption/desorption analysis.

Sample Tube

Sample tube: Sample Tube
Warm free space: 1.0000 cm³
Cold free space: 1.0000 cm³
Non-ideality factor: 0.0000620
Use isothermal jacket: No
Use filler rod: No
Vacuum seal type: None

Degas Conditions

Degas conditions: Degas Conditions

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8 REPORT OPTIONS

File > Open > [.RPO File]

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



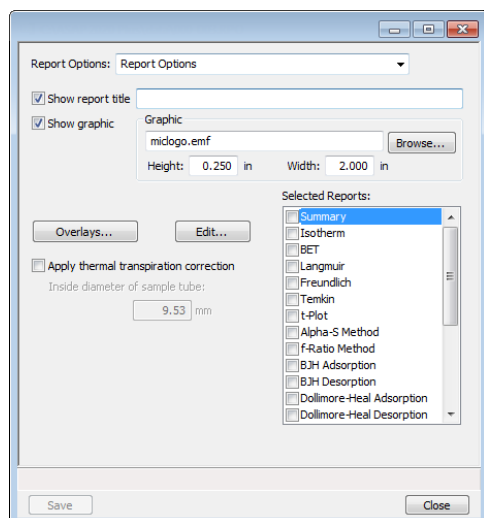
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

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


Reports can be generated for data:

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

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



Report Options Fields and Buttons Table

Field or Button	Description
Apply thermal transpiration correction	<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 approximately 1.0 mmHg.</p> <p>Always use thermal transpiration when performing micropore analyses. See Thermal Transpiration Correction on page B - 44.</p> <ul style="list-style-type: none"> • Inside diameter of sample tube. Enabled when <i>Apply thermal transpiration correction</i> is selected. Enter the inside diameter of the sample tube used in the analysis.
Name column	Displays a list of files in the selected directory.
Overlays	See Graph and Sample Overlays on page 7 - 29 .
Report Options drop-down list	Browse for a .RPO file that contains report options parameters to be used in the report.
Selected Reports list box	Select the report names to include in the report.
Show graphic	<p>Use to show a graphic on the report header. Click Browse to locate the graphic.</p> <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.
Show report title	Select and enter a report title to appear on the report header.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

ADVANCED REPORT OPTIONS

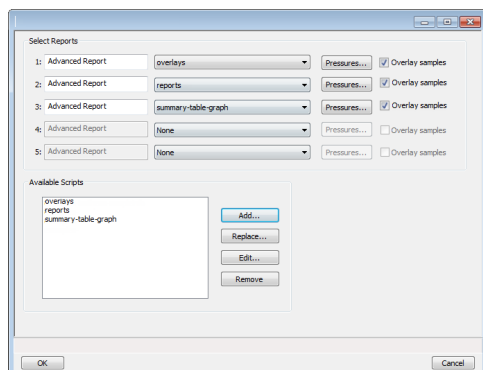
See [Python Module - Advanced Reports on page E - 1](#)



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


Up to five Advanced reports, each with up to 10 summary reports, 10 tabular reports, and 10 graphical reports can be created. To use this feature, a file containing a Python script that imports a "mic" Python module must be created. See [Mic Module Python Calls on page E - 12](#) for an example of Python script and functions for the "mic" Python Module.

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



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

Advanced Report Options Fields and Buttons Table

Field or Button	Description
Add	Click to add additional Python reports.
Available Scripts	Lists the available reports and provides the option to add, replace, edit or remove reports.
Overlay samples	Use to overlay samples as defined by the function.
Advanced Report 1 through 5	Use the drop-down lists to select currently-defined functions used to define the report calculations and output.
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

ALPHA-S METHOD REPORT OPTIONS

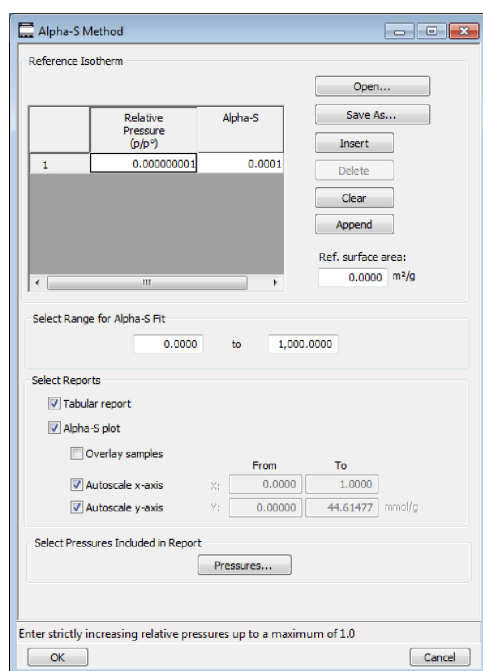


A tutorial is available for this topic. To view the tutorial, click the *Tutorial* tab in *Online Help*.



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

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

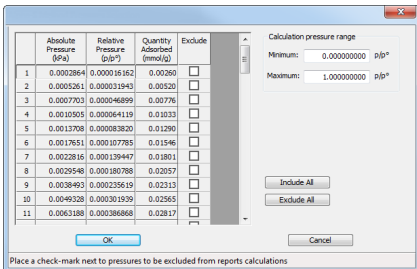


The dialog box titled "Alpha-S Method" contains the following sections:


- Reference Isotherm:** A table with columns "Relative Pressure (p/p⁰)" and "Alpha-S". The first row shows "1" and "0.00000001" respectively. Below the table is a "Ref. surface area:" field with the value "0.0000 m²/g". To the right of the table are buttons: "Open...", "Save As...", "Insert", "Delete", "Clear", and "Append".
- Select Range for Alpha-S Fit:** A range from "0.0000" to "1,000.0000".
- Select Reports:**
 - ☒ Tabular report
 - ☒ Alpha-S plot
 - ☐ Overlay samples
 - ☒ Autoscale x-axis: X: 0.0000 To 1.0000
 - ☒ Autoscale y-axis: Y: 0.00000 To 44.61477 mmol/g
- Select Pressures Included in Report:** A "Pressures..." button.
- Footer:** "Enter strictly increasing relative pressures up to a maximum of 1.0" with "OK" and "Cancel" buttons.

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

Alpha-S Method Report Options Fields and Buttons Table

Field or Button	Description
Alpha-S plot	<p>Use to plot data in graph format.</p> <ul style="list-style-type: none"> • Overlay samples. Use to overlay sample files on the plot. • Autoscale x-axis. The x-axis field shows the relative pressure. • Autoscale y-axis. The y-axis field shows the quantity of gas adsorbed.
Open	<p>To import values from an existing thickness curve (.ALS file), click Open and select the file containing the values.</p> <p>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.</p>
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. <p>To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <ul style="list-style-type: none"> • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Ref. surface area	<p>Enter the surface area from the reference curve. This value is used to calculate the sample surface area.</p>

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

Field or Button	Description
Select Range for Alpha-S Fit	Enter minimum and maximum relative pressures to determine the fit.
Selected Reports	<ul style="list-style-type: none"> • Tabular Report. Use to have a tabular report of data generated. • Alpha-S Plot. Use to plot data in graph format. <ul style="list-style-type: none"> ◦ Overlay samples. Use to overlay sample files on the plot. ◦ Autoscale x-axis. The x-axis field shows the relative pressure. ◦ Autoscale y-axis. The y-axis field shows the quantity of gas adsorbed.
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

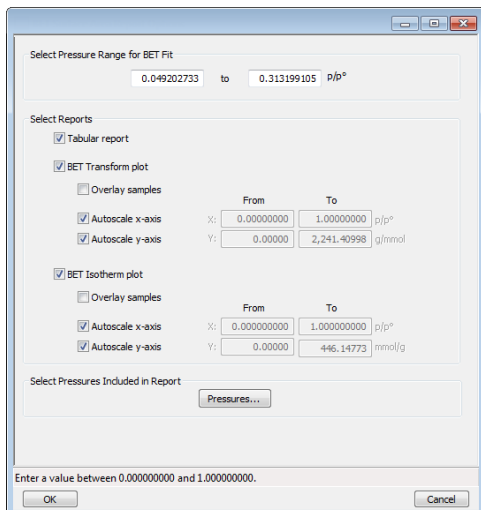
BET SURFACE AREA REPORT OPTIONS



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



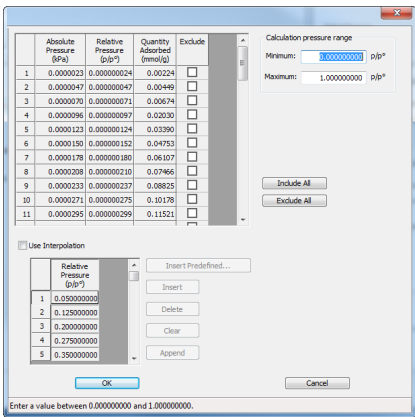
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.




The screenshot shows a dialog box titled "Select Pressure Range for BET Fit". At the top, it displays a pressure range from 0.049202733 to 0.313199105 p/p°. Below this, there are two sections for selecting reports. The first section, "Select Reports", includes checkboxes for "Tabular report" (checked), "BET Transform plot" (checked), "Overlay samples" (unchecked), "Autoscale x-axis" (checked), and "Autoscale y-axis" (checked). It also shows axis ranges for X (0.00000000 to 1.00000000 p/p°) and Y (0.00000 to 2,241.40998 g/mmol). The second section, "BET Isotherm plot", includes checkboxes for "Overlay samples" (unchecked), "Autoscale x-axis" (checked), and "Autoscale y-axis" (checked). It shows axis ranges for X (0.00000000 to 1.00000000 p/p°) and Y (0.00000 to 446.14773 mmol/g). At the bottom, there is a "Select Pressures Included in Report" section with a "Pressures..." button. The dialog box has "OK" and "Cancel" buttons at the bottom.

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

BET Report Options Fields and Buttons Table

Field or Button	Description
Pressures	<p>This option is available when the sample file has a status of <i>Analyzing</i> or <i>Complete</i>. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table if not using the <i>Use Interpolation</i> option. • Use Interpolation. Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only. • Insert Predefined. Click to insert a predefined (default) set of points into the report. <i>Use Interpolation</i> must be selected to enable this button. This button displays for BET reports only. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Select Pressure Range for BET fit	<p>Enter values to indicate the fitted pressure range.</p>
Selected Reports	<ul style="list-style-type: none"> • Tabular report. Use to have a table of measured and calculated values generated. • BET Transform plot. Use to generate a traditional BET surface area plot used to determine monolayer volume and BET C constant. <ul style="list-style-type: none"> ◦ Overlay samples. Use to overlay sample files on the BET transform plot. ◦ Autoscale x-axis. Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET. ◦ Autoscale y-axis. The y-axis field shows BET transformation.

BET Report Options Fields and Buttons Table (continued)

Field or Button	Description
	<ul style="list-style-type: none">• BET Isotherm plot. Uses BET monolayer volume and constant to produce an isotherm.<ul style="list-style-type: none">◦ Overlay samples. Use to overlay sample files on the BET isotherm plot.◦ Autoscale x-axis. Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET.◦ Autoscale y-axis. The y-axis field shows the quantity of gas adsorbed.
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

BJH ADSORPTION / DESORPTION REPORT OPTIONS



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

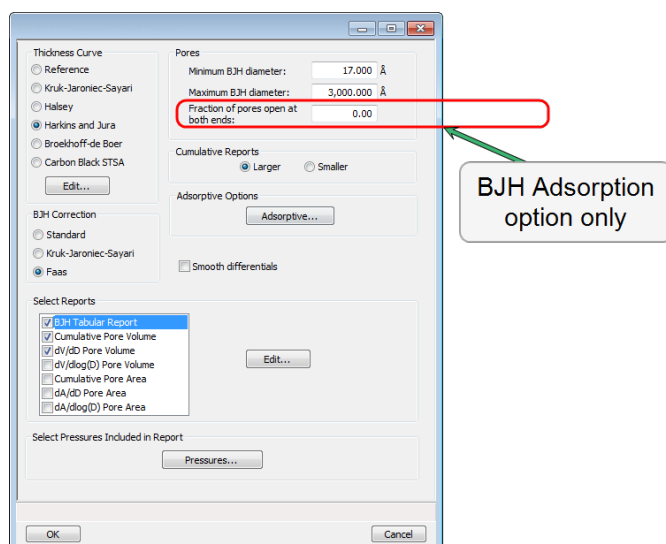


To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

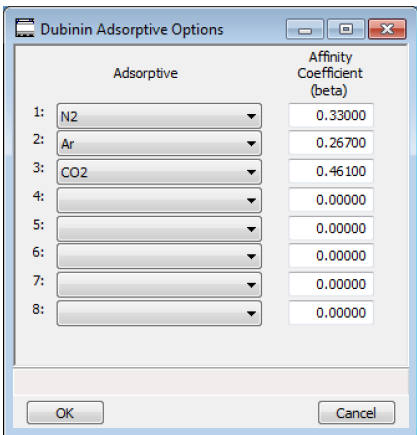
The BJH calculation determines the mesopore volume / area distribution, which accounts for both the change in adsorbate layer thickness and the liquid condensed in pore cores. Reports can be generated from both adsorption and desorption data. The fields for both *BJH Adsorption Report Options* and *BJH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



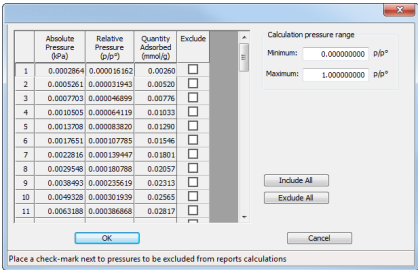
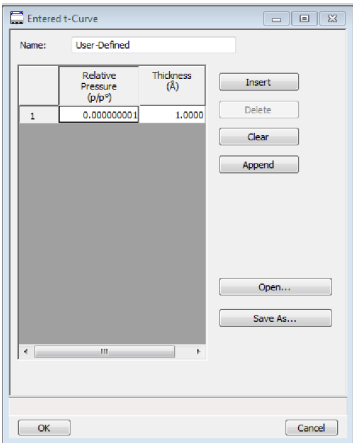
An incomplete pore distribution may be generated if a thickness curve selection is not a good match for the sample being analyzed.




BJH Adsorption / Desorption Report Options Fields and Buttons Table

Field or Button	Description
Adsorptive	<p>Displays the <i>Adsorptive Options</i> window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.</p> 
BJH Correction	<p>Select the type of correction to apply to calculations. The selected type displays in the report header.</p> <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.
Cumulative Reports	<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.
Pores	<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 <i>BJH Desorption Report Options</i> 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>
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>

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

Field or Button	Description
	 <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Select Reports	Select the report names to include in the report. Highlight the report name, then click Edit to modify report parameters.
Smooth differentials	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.
Thickness Curve	<p>Select the thickness curve, then click Edit to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option.</p> <p>Reference. Select <i>Reference</i>, then click Edit to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the <i>Reference</i> directory.</p> 

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

Field or Button	Description
	<p>To import values from an existing thickness curve (.THK file), click Open, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space or a tab.</p> <p>Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA. Select the thickness curve option, then click Edit. Modify the equation for the selected curve as needed.</p>
	<p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>

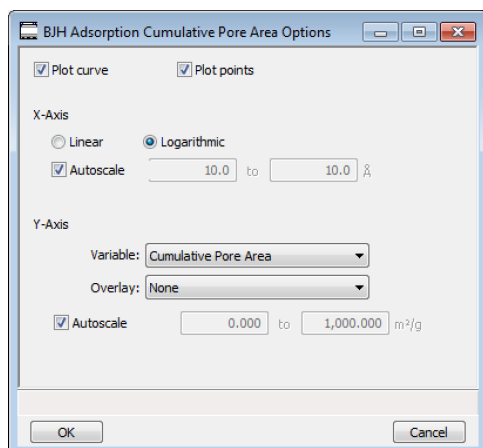
BJH PLOT OPTIONS



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




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



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

BJH Plot Options Fields and Buttons Table

Field or Button	Description
Autoscale	When enabled on the report parameters windows, allows the x- and y-axes to be scaled automatically. <i>Autoscale</i> means that the x- and y-ranges will be set so that all the data is shown. If <i>Autoscale</i> is not selected, the entered range is used.
Plot curve / Plot points	Select to plot points on the graph.
X-Axis	Use to have the x-axis on a logarithmic or linear scale.
Y-Axis	<ul style="list-style-type: none"> Variable. Select a variable. Overlay. Select an option to overlay onto the current report.
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

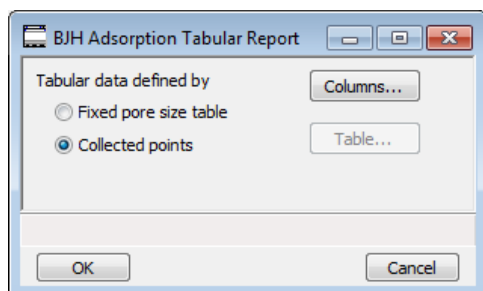
BJH TABULAR REPORT OPTIONS



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

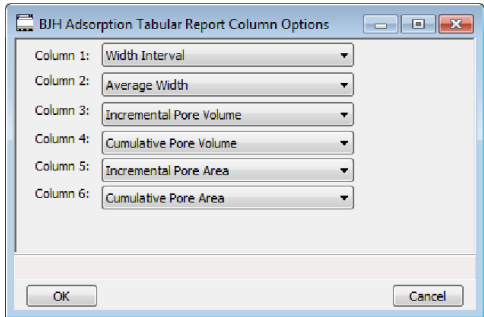


To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.




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

BJH Tabular Report Options Fields and Buttons Table

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

BJH Tabular Report Options Fields and Buttons Table (continued)

Field or Button	Description
	points must be strictly decreasing. Enabled only when <i>Fixed pore size table</i> is selected.
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

DFT PORE SIZE REPORT OPTIONS

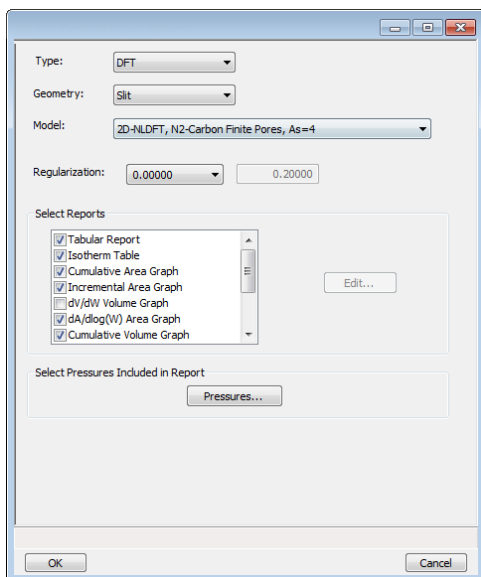


*A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.*



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

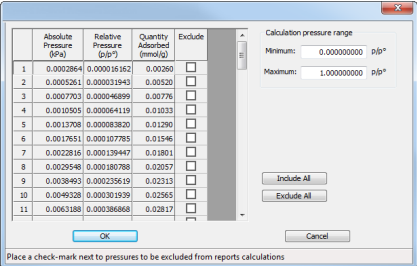
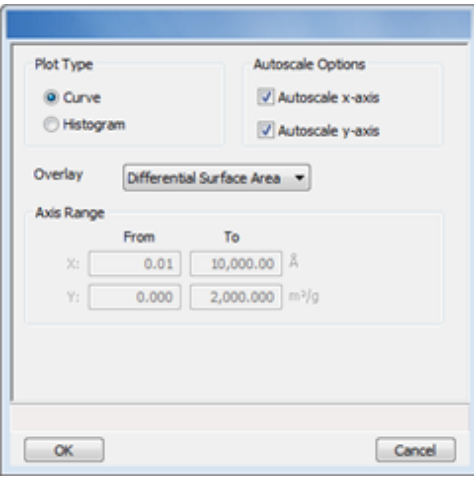
The *DFT Pore Size* report contains the results of pore size distribution analyses using a non-local DFT range of micro and mesopore ranges.




DFT Pore Size Report Options Fields and Buttons Table

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

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

Field or Button	Description
	 <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Regularization	Select the extent of smoothing to apply to the data. If <i>0.20000 (user)</i> is selected, enter a number in the text box giving a relative weight for the smoothing during deconvolution. Larger values produce more smoothing.
Select Reports	<p>Select the reports to generate. To edit graph details, highlight the graph option and click Edit. The <i>Log Goodness of Fit</i> and <i>Goodness of Fit</i> graphs cannot be edited.</p>  <ul style="list-style-type: none"> • Plot Type. Select the method for data display. • Autoscale Options. Use to autoscale the x-axis and / or y-axes. • Overlay. Select an overlay for the report.

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

Field or Button	Description
	<ul style="list-style-type: none">• Axis Range. <i>From / To</i> fields are enabled when <i>Autoscale</i> 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.
Type	<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. See DFT Models on page C - 1.
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

DFT SURFACE ENERGY REPORT OPTIONS

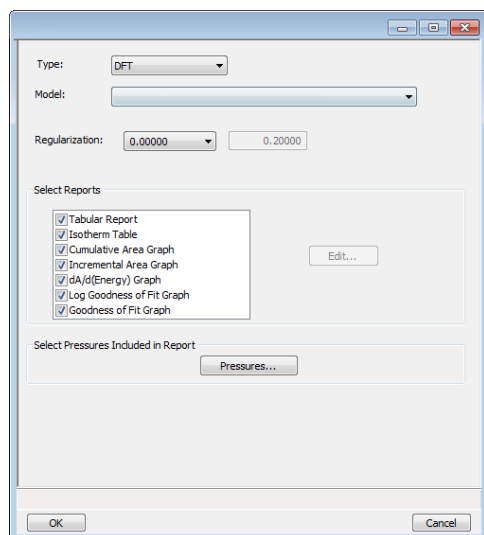


A tutorial is available for this topic. To view the tutorial, click the *Tutorial* tab in *Online Help*.



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

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



The screenshot shows the 'DFT Surface Energy Report Options' dialog box. It has a 'Type' dropdown set to 'DFT' and an empty 'Model' dropdown. The 'Regularization' section has two input fields: '0.00000' and '0.20000'. Below this is a 'Select Reports' section with a list box containing the following items, all of which are checked: 'Tabular Report', 'Isotherm Table', 'Cumulative Area Graph', 'Incremental Area Graph', 'dA/d(Energy) Graph', 'Log Goodness of Fit Graph', and 'Goodness of Fit Graph'. To the right of the list box is an 'Edit...' button. Below the list box is a 'Select Pressures Included in Report' section with a 'Pressures...' button. At the bottom of the dialog are 'OK' and 'Cancel' buttons.

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

DOLLIMORE-HEAL ADSORPTION / DESORPTION REPORT OPTIONS

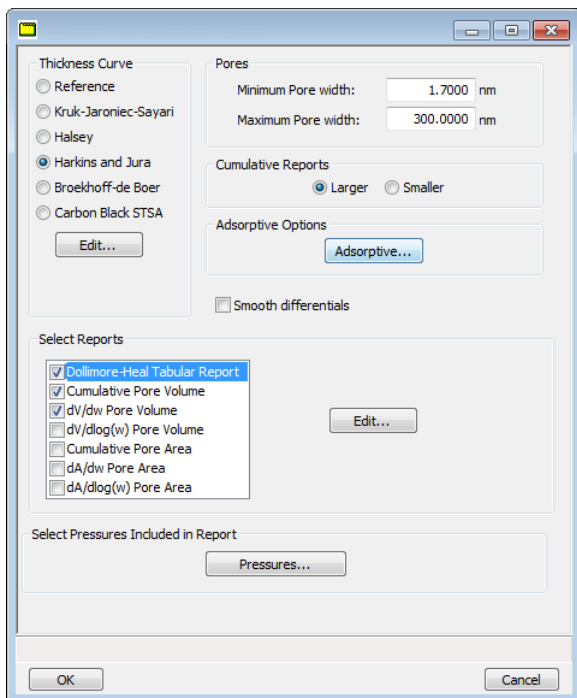


A tutorial is available for this topic. To view the tutorial, click the Tutorial tab in Online Help.



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

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



The screenshot shows the 'Dollimore-Heal Report Options' dialog box. It contains 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 (1.7000 nm) and Maximum Pore width (300.0000 nm).
- 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 Area
 An 'Edit...' button is to the right of the list.
- Select Pressures Included in Report:** A 'Pressures...' button.

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

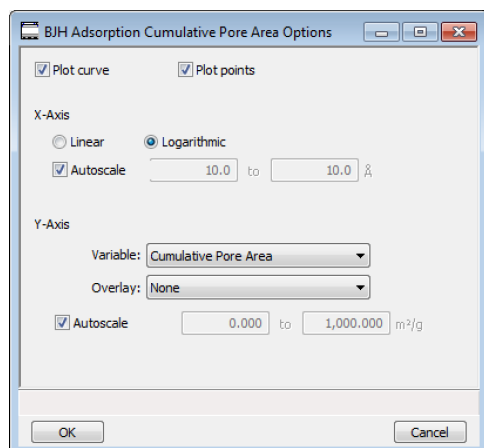
DOLLIMORE-HEAL PLOT OPTIONS



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



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

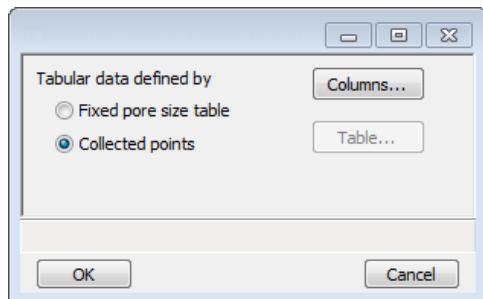


The fields for all plot options are identical for specifying plotting methods and customizing plots. Highlight any plot option in the *Selected Reports* list box in the *BJH Report Options* window, then click **Edit**. The fields and buttons for these reports are identical to the *BJH Plot Report Options*.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

DOLLIMORE-HEAL TABULAR REPORT OPTIONS



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



Dollimore-Heal Tabular Report Options are identical to the *BJH Tabular Report Options*. See [BJH Tabular Report Options on page 8 - 16](#).

DUBININ REPORT OPTIONS

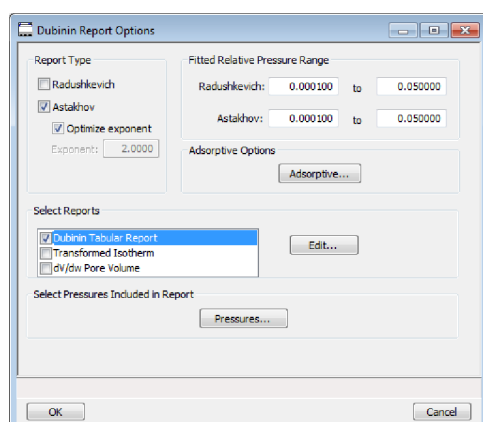


*A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.*



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

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

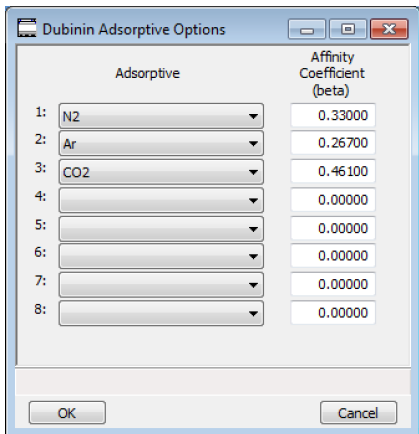
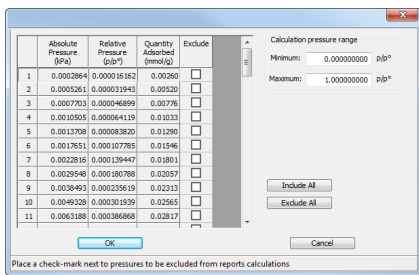


The screenshot shows the 'Dubinin Report Options' dialog box. It has several sections:


- Report Type:** Contains checkboxes for 'Radushkevich' (unchecked), 'Astakhov' (checked), and 'Optimize exponent' (checked). Below 'Optimize exponent' is a text box for 'Exponent' with the value '2.0000'.
- Fitted Relative Pressure Range:** Contains two rows of input fields. The first row is for 'Radushkevich' with values '0.000100' and '0.050000'. The second row is for 'Astakhov' with values '0.000100' and '0.050000'.
- Adsorptive Options:** Contains a button labeled 'Adsorptive...'.
- Select Reports:** Contains a list box with three items: 'Dubinin Tabular Report' (selected), 'Transformed Isotherm', and 'dV/dw Pore Volume'. To the right of the list box is an 'Edit...' button.
- Select Pressures Included in Report:** Contains a button labeled 'Pressures...'.

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

Dubinin Report Options Fields and Buttons Table

Field or Button	Description
Adsorptive	<p>Displays the <i>Adsorptive Options</i> window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.</p> 
Fitted Relative Pressure Range	<p>Enter the minimum and maximum limits for Radushkevich or Astakhov relative pressures included in the line fit.</p>
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.

Dubinin Report Options Fields and Buttons Table (continued)

Field or Button	Description
Report Type	Select report types. If <i>Astakhov</i> is selected, either select <i>Optimize exponent</i> or enter an appropriate exponent value in the text box.
Select Reports	Select the reports to generate. Highlight the report, then click Edit to modify report options.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

DUBININ PORE VOLUME REPORT OPTIONS



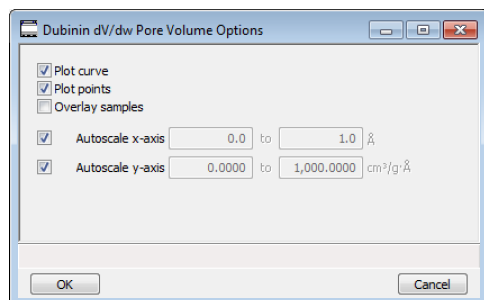
A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.




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

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

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



Dubinin Pore Volume Report Fields and Buttons Table

Field or Button	Description
Autoscale x-axis / Autoscale y-axis	Select an option to have the x- and / or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges. Enable to enter beginning and ending values manually.
Overlay samples	Use to overlay sample files on the plot.
Plot curve / Plot points	Select to plot points on the graph.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

DUBININ TABULAR REPORT OPTIONS

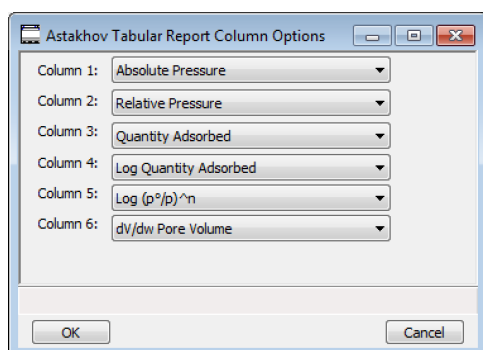


A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.



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

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



Log (p⁰/p)ⁿ. The value for *[n]* is the optimized exponent if *Optimize exponent* is selected on the *Dubinin Report Options* window. If not, then the value for *[n]* is the entered exponent value.

DUBININ TRANSFORMED ISOTHERM PLOT OPTIONS



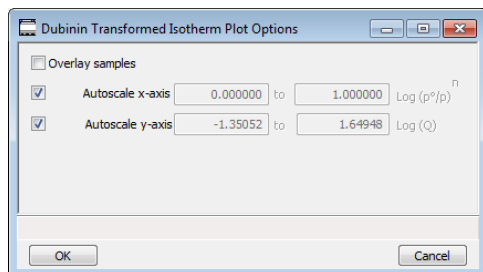
A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.




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

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

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



Dubinin Transformed Isotherm Plot Options Fields and Buttons Table

Field or Button	Description
Autoscale x-axis / Autoscale y-axis	<p>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.</p> <p>Deselect to enter beginning and ending values manually.</p> <ul style="list-style-type: none"> Autoscale x-axis. Shows the quantity of gas adsorbed at standard temperature and pressure. Autoscale y-axis. Shows the log of relative pressure.
Overlay Samples	Use to overlay sample files on the plot.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

F-RATIO METHOD REPORT OPTIONS

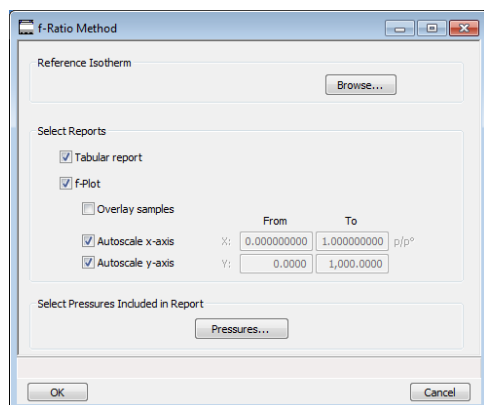


*A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.*



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

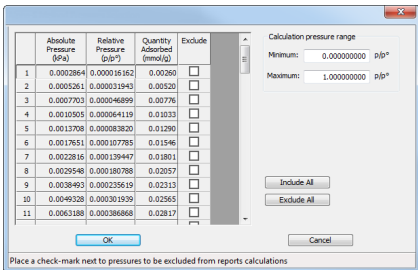

The *f*-Ratio report uses the measured isotherm and normalizes it using a reference isotherm.



The screenshot shows the 'f-Ratio Method' dialog box with the following settings:

- Reference Isotherm:** A 'Browse...' button is present.
- Select Reports:**
 - ☒ Tabular report
 - ☒ F-Plot
 - ☐ Overlay samples
 - ☒ Autoscale x-axis
 - ☒ Autoscale y-axis
- Axis Scaling:**
 - X-axis: From 0.000000000 to 1.000000000 p/p⁰
 - Y-axis: From 0.0000 to 1,000.0000
- Select Pressures Included in Report:** A 'Pressures...' button is present.
- Buttons:** 'OK' and 'Cancel' buttons are at the bottom.

f-Ratio Method Report Options Fields and Buttons Table

Field or Button	Description
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Reference isotherm	<p>Click Browse to select a sample file to use as a reference for the isotherm. Select a file containing an isotherm measured from a non-porous sample of the same material as the current sample. When the referenced file is selected, the file name appears to the left of Browse.</p>
Selected Reports	<ul style="list-style-type: none"> • Tabular Report. Use to have a tabular report of data generated. • f-Plot. Use to generate a normalized isotherm. <ul style="list-style-type: none"> ◦ Overlay samples. Use to overlay sample files on the f-plot. ◦ Autoscale x-axis. The X-axis field is dimensionless in units of f-ratio. ◦ Autoscale y-axis. The Y-axis field shows the quantity of gas adsorbed.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

FREUNDLICH REPORT OPTIONS

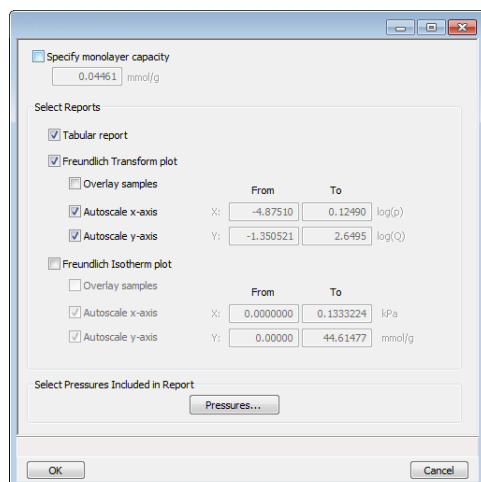


A tutorial is available for this topic. To view the tutorial, click the *Tutorial* tab in *Online Help*.



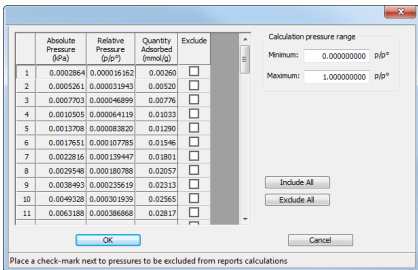
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

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




The screenshot shows the 'Freundlich Report Options' dialog box. It has a title bar with standard window controls. Inside, there's a section for 'Specify monolayer capacity' with a value of 0.04461 mmol/g. Below that is a 'Select Reports' section with two main options: 'Freundlich Transform plot' (checked) and 'Freundlich Isotherm plot' (unchecked). Each has sub-options for 'Overlay samples' (unchecked) and 'Autoscale' (checked for both x and y axes). The 'Freundlich Transform plot' section shows 'From' and 'To' values for X (log(p)) and Y (log(Q)). The 'Freundlich Isotherm plot' section shows 'From' and 'To' values for X (kPa) and Y (mmol/g). At the bottom, there's a 'Select Pressures Included in Report' section with a 'Pressures...' button. The dialog has 'OK' and 'Cancel' buttons at the bottom.

Freundlich Report Options Fields and Buttons Table

Field or Button	Description
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Select Reports	<ul style="list-style-type: none"> • Tabular report. Select to include pressure points included in the report. • Freundlich Isotherm plot. Plots the absolute pressure vs quantity adsorbed. Shows best fit line. <ul style="list-style-type: none"> ◦ Overlay samples. Use to overlay sample files on the Freundlich isotherm plot. ◦ Autoscale x-axis. Linear x-axes begin at zero. The x-axis field shows the absolute pressure. ◦ Autoscale y-axis. Y-axes begin at zero. The y-axis field shows the quantity of gas adsorbed. • Freundlich Transform plot. Plots the log(P) vs log(Q) and the best fit. <ul style="list-style-type: none"> ◦ Overlay samples. Use to overlay sample files on the Freundlich transform plot. ◦ Autoscale x-axis. The x-axis field shows the absolute pressure. ◦ Autoscale y-axis. The y-axis field shows the quantity of gas adsorbed.
Specify monolayer capacity	Select and enter the monolayer capacity of the sample.

Freundlich Report Options Fields and Buttons Table (continued)

Field or Button	Description
Tabular report	Use to have a report of the pressure points generated.
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

HORVATH-KAWAZOE REPORT OPTIONS

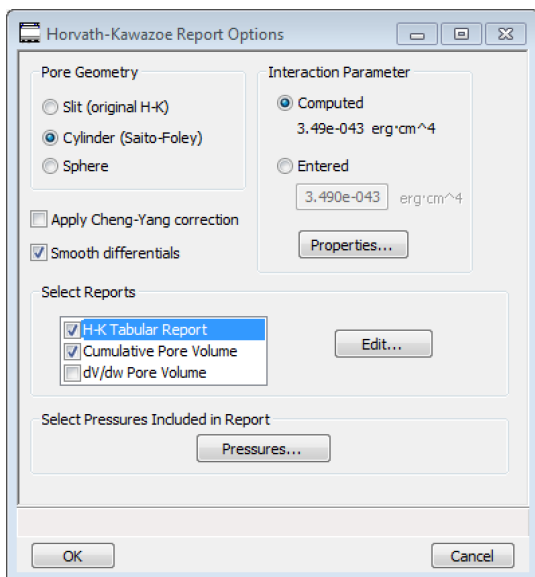


*A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.*



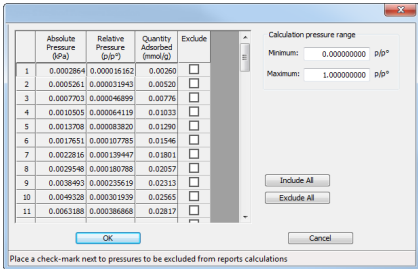
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

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

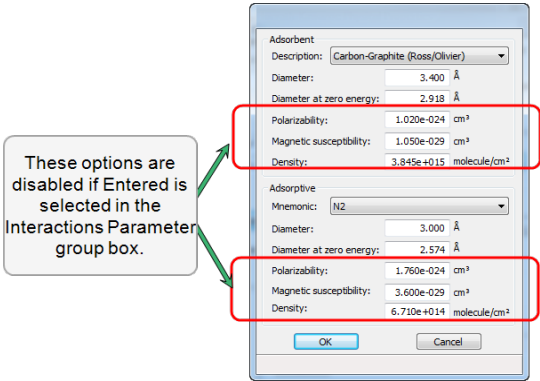


The screenshot shows the "Horvath-Kawazoe Report Options" dialog box. It has several sections: "Pore Geometry" with radio buttons for "Slit (original H-K)", "Cylinder (Saito-Foley)" (selected), and "Sphere"; "Interaction Parameter" with radio buttons for "Computed" (selected) and "Entered", and a text field showing "3.490e-043 erg*cm^4" with a "Properties..." button; "Select Reports" with checkboxes for "H-K Tabular Report" (checked), "Cumulative Pore Volume" (checked), and "dV/dw Pore Volume" (unchecked), and an "Edit..." button; and "Select Pressures Included in Report" with a "Pressures..." button. At the bottom are "OK" and "Cancel" buttons.


Horvath-Kawazoe Report Fields and Buttons Table

Field or Button	Description
Apply Cheng-Yang correction	Use to apply the Cheng-Yang correction to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath-Kawazoe derivation.
Interaction Parameter	<p>Use to determine which interaction parameter will be used in the report. These options are disabled if <i>Sphere</i> is selected in the <i>Pore Geometry</i> group box.</p> <ul style="list-style-type: none"> • Computed. Use to calculate using the parameters on the <i>Horvath-Kawazoe Physical Properties</i> window (click Properties to display the <i>Physical Properties</i> window). The interaction parameter is recalculated each time a parameter in the <i>Physical Properties</i> window is edited. • Entered. Calculates using the value entered in the text box.
Pore Geometry	Select the option that best represents the physical geometry of the micropores in the sample material. When <i>Sphere</i> is selected, options in the <i>Interaction Parameter</i> group box are disabled.
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Properties	Click to view or edit the constants describing the physical properties of the adsorbent and adsorptive.

Horvath-Kawazoe Report Fields and Buttons Table (continued)

Field or Button	Description
	 <p>Adsorbent. Contains the parameters for the sample. If using <i>Computed</i> for the interaction parameter, all fields are enabled. If using <i>Entered</i>, only the values in the <i>Diameter</i> and <i>Diameter at zero energy</i> 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. <p>Adsorptive. Contains the parameters for the adsorptives. If using <i>Computed</i> for the interaction parameter, all fields are enabled. If using <i>Entered</i>, only the values in the <i>Diameter</i> and <i>Diameter at zero energy</i> 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.

Horvath-Kawazoe Report Fields and Buttons Table (continued)

Field or Button	Description
Select Reports	Select the types of reports to generate. Highlight the report, then click Edit to modify report parameters.
Smooth Differentials	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

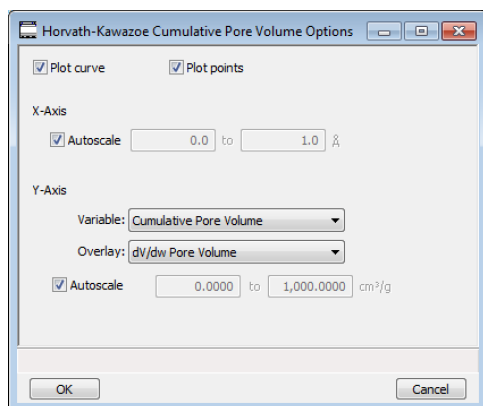
HORVATH-KAWAZOE PLOT OPTIONS



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



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



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

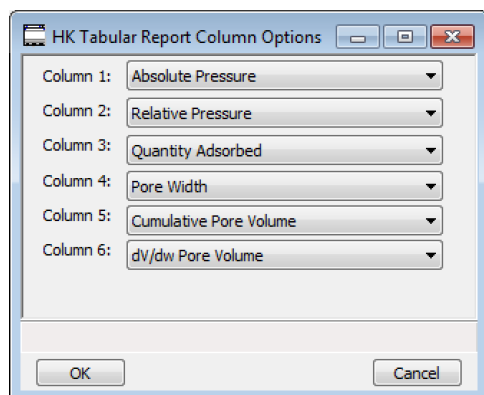
HORVATH-KAWAZOE TABULAR REPORT OPTIONS



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



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



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

ISOTHERM REPORT OPTIONS

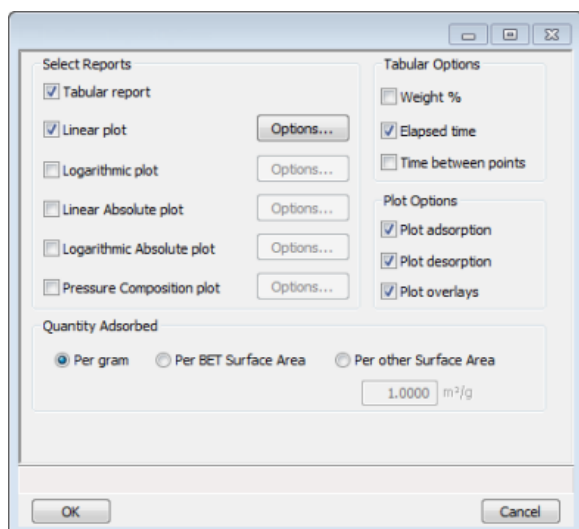


A tutorial is available for this topic. To view the tutorial, click the *Tutorial* tab in *Online Help*.



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


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



Physisorption Isotherm Report Options Fields and Buttons Table

Field or Button	Description
Options	Click to display related linear plot options. All plot windows contain identical fields. <ul style="list-style-type: none"> Plot curve / Plot points. Select to plot points on the graph. Autoscale x-axis. Linear x-axes begin at zero. Logarithmic x-axes begin at an appropriate value. The x-axis field shows the relative or absolute pressure. Autoscale y-axis. The y-axis field shows the quantity of gas adsorbed.
Plot Options	Select the types of isotherm to plot.
Quantity Adsorbed	Select how to report the quantity adsorbed.

Physisorption Isotherm Report Options Fields and Buttons Table (continued)

Field or Button	Description
	<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
Select Reports	Select each option to include on the final report. Click the Options button of a selected item to include plot curve, plot points, and to autoscale x- and y-axes.
Tabular Options	Select the options to include on the report. <ul style="list-style-type: none"> • Weight %. Enter the mass percentage when plotting pressure composition • Elapsed time. Time elapsed during the analysis • Time between points. Time elapsed between points during the analysis
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

LANGMUIR REPORT OPTIONS

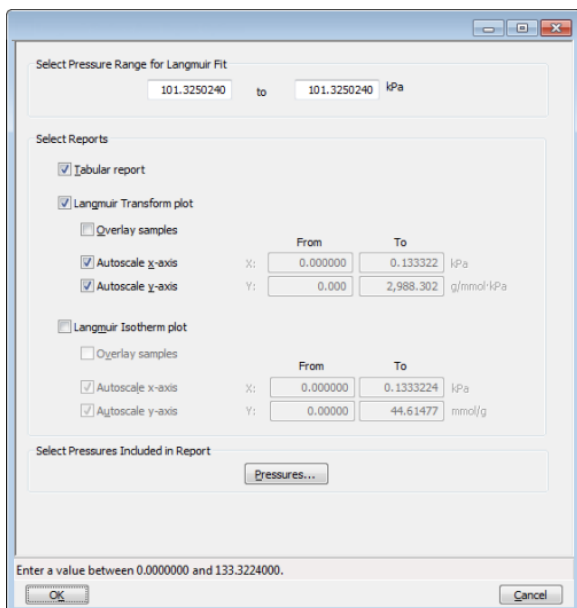


*A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.*



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

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

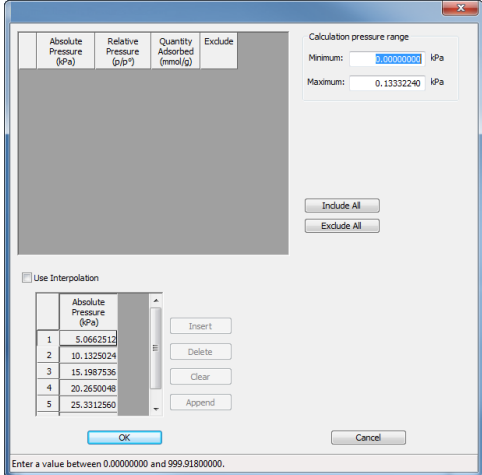


The dialog box is titled "Select Pressure Range for Langmuir Fit". It contains the following sections:


- Select Pressure Range for Langmuir Fit:** Two input fields showing "101.3250240" and "101.3250240" kPa, separated by "to".
- Select Reports:**
 - ☒ Tabular report
 - ☒ Langmuir Transform plot
 - ☐ Overlay samples
 - ☒ Autoscale x-axis: X: 0.000000 To 0.133322 kPa
 - ☒ Autoscale y-axis: Y: 0.000 To 2,988.302 g/mmol kPa
 - ☐ Langmuir Isotherm plot
 - ☐ Overlay samples
 - ☒ Autoscale x-axis: X: 0.000000 To 0.1333224 kPa
 - ☒ Autoscale y-axis: Y: 0.00000 To 44.61477 mmol/g
- Select Pressures Included in Report:** A button labeled "Pressures..."

At the bottom, there is a text field with the prompt "Enter a value between 0.000000 and 133.3224000." and "OK" and "Cancel" buttons.

Langmuir Report Options Fields and Buttons Table

Field or Button	Description
Pressures	<p>This option is available when the sample file has a status of <i>Analyzing</i> or <i>Complete</i>. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Use Interpolation. Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Select Pressure Range for Langmuir fit	Enter values to indicate the fitted pressure range.
Selected Reports	<ul style="list-style-type: none"> • Langmuir Transform Plot. Use to generate a traditional Langmuir surface area plot used to determine monolayer volume constant <ul style="list-style-type: none"> ◦ Autoscale x-axis. Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. ◦ Autoscale y-axis. The y-axis field shows Langmuir transformation. ◦ Overlay samples. Use to overlay sample files on the Lang-

Langmuir Report Options Fields and Buttons Table (continued)

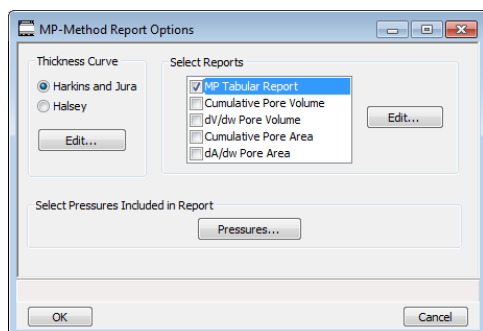
Field or Button	Description
	<p>muir transform plot.</p> <ul style="list-style-type: none">• Langmuir Isotherm Plot. Uses the Langmuir monolayer volume and constant to produce an isotherm.<ul style="list-style-type: none">◦ Overlay samples. Use to overlay sample files on the Langmuir isotherm plot.◦ Autoscale x-axis. Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir.◦ Autoscale y-axis. The y-axis field shows the quantity of gas adsorbed.
	<p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>

MP-METHOD REPORT OPTIONS

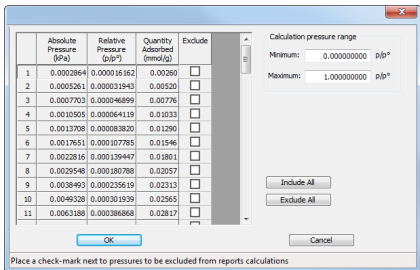



To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

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



MP-Method Report Options Fields and Buttons Table

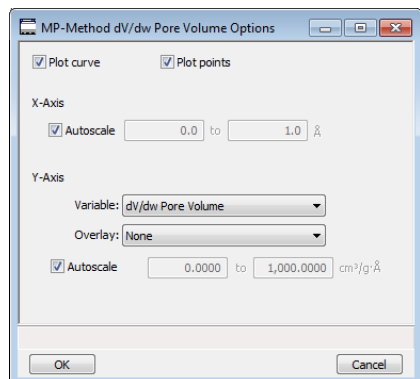
Field or Button	Description
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Select Reports	Select the reports to generate. Highlight the report, then click Edit to modify report options.
Thickness Curve	Select the thickness curve, then click Edit to modify the values in the equation for the selected curve.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

MP-METHOD PLOT REPORT OPTIONS



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

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



MP Method Plot Options Fields and Buttons Table

Field or Button	Description
Overlay drop-down list	Select an option to overlay on the current report.
Plot curve / Plot points	Select to plot points on the graph.
Thickness Curve	Select the thickness curve, then click Edit to modify the values in the equation for the selected curve.
X-Axis	Use to have the x-axis autoscaled or enter beginning and ending values.
Y-Axis	<ul style="list-style-type: none"> Variable. Select a variable. Overlay. Select an option to overlay on the current report. Autoscale. Use to have the y-axis autoscaled or enter beginning and ending values.



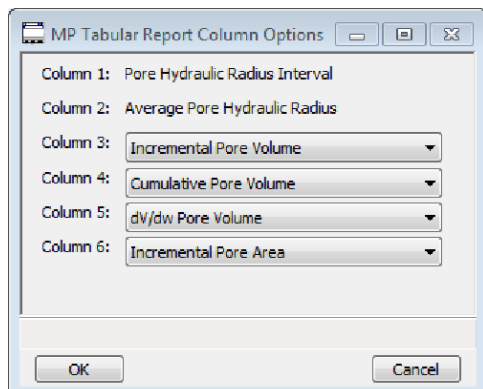
For fields and buttons not listed in this table, see the *Common Fields and Buttons* section of this operator manual.

MP-METHOD TABULAR REPORT OPTIONS



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

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



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

NLDTF ADVANCED PSD REPORT



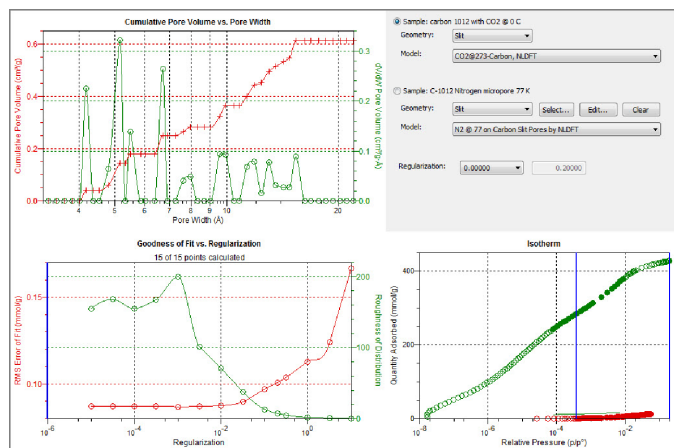
A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.



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

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

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



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

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

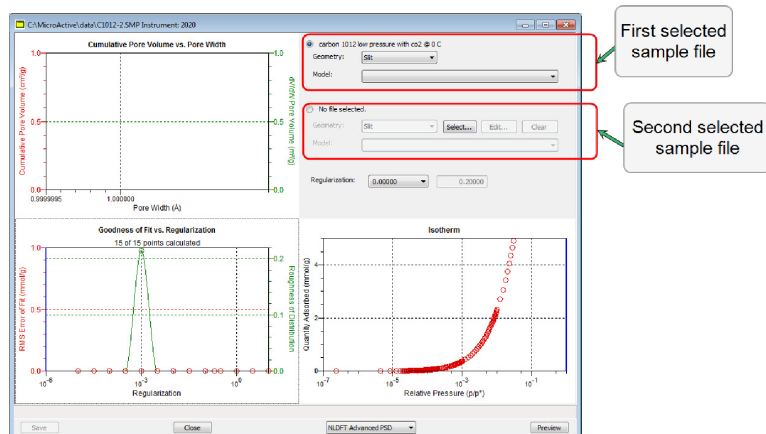
In general, the isotherm for this second sample will be different than the first sample. The advanced DFT calculation takes the data from both sample files and combines all this data into a more accurate calculation of the pore size distribution. More accurate means getting the pore distribution at smaller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



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

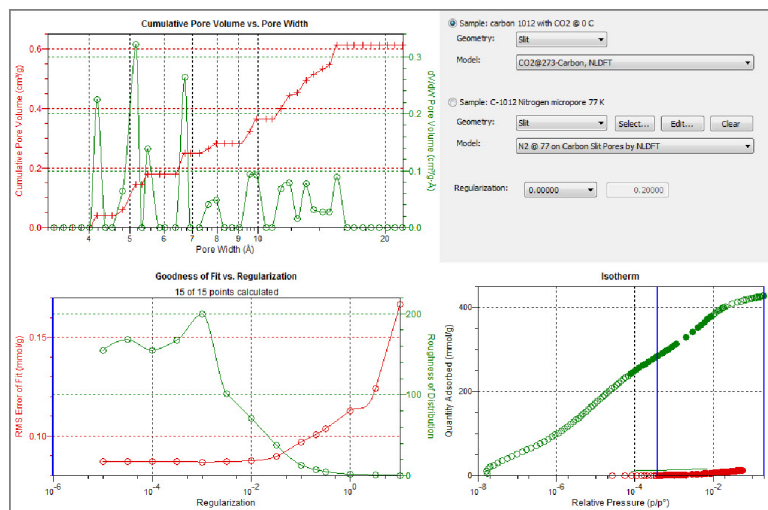
To run the NLDFT report:

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




- a. Select the *Geometry* and *Model* from the drop-down lists for the first sample file.
 - b. To select isotherm data points for calculation for the first sample file, ensure the option to the left of the first sample file description is selected. Slide the two blue bars on the isotherm graph to select data points. Without a second sample selected, the report will perform a single model DFT calculation and show the results in the two left-hand result windows.
3. To calculate data from the second sample file, click **Select** to locate and open the second sample file with a *Complete* status. Graphs for the second sample file display and the sample description displays as the second group box title in the upper right corner of the window.

- Select the *Geometry* and *Model* from the drop-down lists for the second sample file.
- To select isotherm data points for calculation for the second sample file, ensure the option to the left of the second sample file description is selected. Slide the two blue bars on the isotherm graph to select data points. Data are automatically calculated for both sample files.
- Click **Edit** to make any necessary modifications to the second sample file.



NLDTF Advanced PSD Report Fields and Buttons Table

Field or Button	Description
Geometry	Select the pore shape.
Model	Lists the models that meet the specified criteria and match the adsorbate and temperature of the sample data. If no models appear, no models meet the selected criteria. One model must be selected.
Regularization	Select the extent of smoothing to apply to the data. If 0.20000 (user) is selected, enter a number in the text box giving a relative mass for the smoothing during deconvolution. Larger values produce more smoothing.
Select	Use to select the second sample file.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

OPTIONS REPORT

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

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

If *Automatically collected* is selected in the *Type of Data* group box on the *Sample Description* tab, the following information is reported:

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



Options reports cannot be edited.

SAMPLE LOG REPORT

This report provides information on:

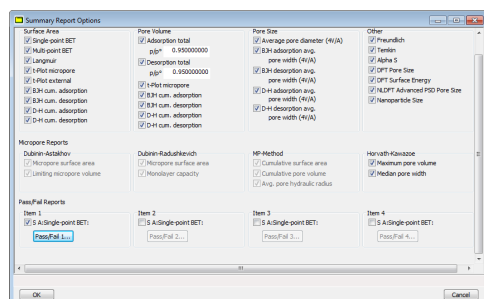
- Manual control operations performed during analysis
- Information entered using *Add Log Entry* on the sample file editor
- Warnings and / or errors which occurred during analysis

SUMMARY REPORT OPTIONS



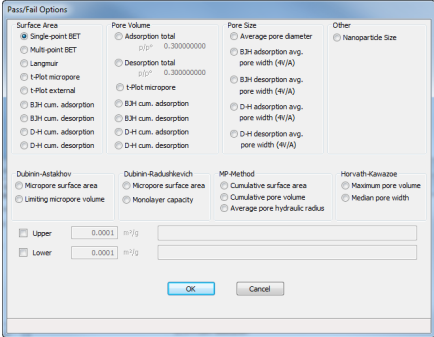

To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

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



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

Summary Report Fields and Buttons Table

Field or Button	Description
Item [n]	<p>Use to enable the first <i>Pass/Fail</i> item. Until the <i>Summary Report</i> is selected, <i>SA Single-point BET</i> will be displayed by default. When selected, click Pass/Fail, then select pass/fail criteria options.</p> <ul style="list-style-type: none"> • S A: Single-point BET. Use to enable Pass/Fail [n] in the <i>Item [n]</i> group box. • Pass/Fail [n]. Click to display the <i>Pass/Fail Options</i> window for selection of pass/fail criteria.  <ul style="list-style-type: none"> • Upper / Lower. Specify upper and lower limits for the selected parameter. A range can be left open by not selecting the limit. In the text box to the right of <i>Upper / Lower</i>, enter operator instructions to be displayed if a failure is encountered.
Select All / Deselect All	Selects (or deselects) all options.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

T-PLOT REPORT OPTIONS

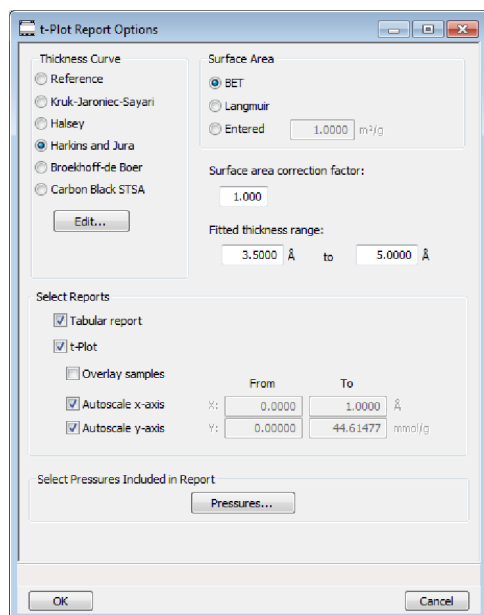


*A tutorial is available for this topic. To view the tutorial, click the **Tutorial** tab in **Online Help**.*



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

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

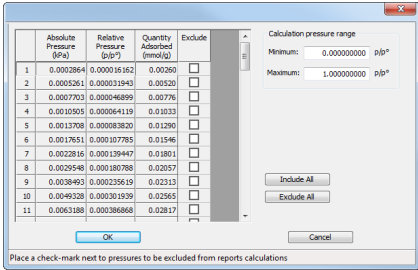


The screenshot shows the 't-Plot Report Options' dialog box. It contains several sections for configuring the report:

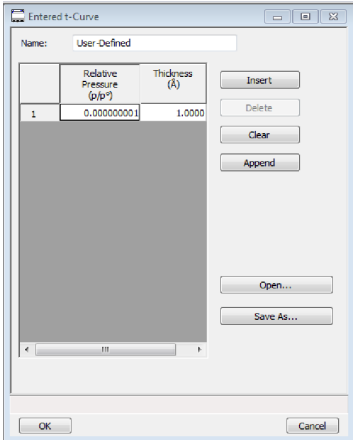

- Thickness Curve:** Radio buttons for Reference, Kruk-Jaroniec-Sayari, Halsey, Harlins and Jura (selected), Broekhoff-de Boer, and Carbon Black STSA. An 'Edit...' button is below.
- Surface Area:** Radio buttons for BET (selected), Langmuir, and Entered. The 'Entered' option has a text field with '1.0000' and units 'm²/g'.
- Surface area correction factor:** A text field with '1.000'.
- Fitted thickness range:** Two text fields for 'From' (3.5000 Å) and 'To' (5.0000 Å).
- Select Reports:** Checkboxes for 'Tabular report' (checked), 't-Plot' (checked), and 'Overlay samples' (unchecked).
- Autoscale:** Checkboxes for 'Autoscale x-axis' (checked) and 'Autoscale y-axis' (checked). Below are 'From' and 'To' ranges for X (0.0000 to 1.0000 Å) and Y (0.00000 to 44.61477 mmol/g).
- Select Pressures Included in Report:** A 'Pressures...' button.

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

t-Plot Report Options Fields and Buttons Table

Field or Button	Description
Fitted thickness range	Enter the minimum and maximum thicknesses (in angstroms or nanometers) to include in the thickness curve. Go to Options > Units to specify default units.
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Selected Reports	<ul style="list-style-type: none"> • Tabular Report. Use to have a tabular report of data generated. • t-Plot. Use to have a graphical representation of data generated. <ul style="list-style-type: none"> ◦ Overlay samples. Use to overlay sample files on the t-plot. ◦ Autoscale x-axis. The X-axis field shows the statistical thickness of the adsorbed film. ◦ Autoscale y-axis. The Y-axis field shows the quantity of gas adsorbed.
Surface area correction factor	Enter the value to correct for surface areas that are not smooth. This brings the values for BET surface area and micropore surface area into accordance. For most samples, the default value of 1.000 is adequate.
Surface Area	Select the surface area value used for thickness calculations. BET is the most commonly used option.
Thickness Curve	<p>Select the thickness curve, then click Edit to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option.</p> <p>Reference. Select <i>Reference</i>, then click Edit to define a t-curve by entering both the relative pressure and thickness values. One predefined</p>

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

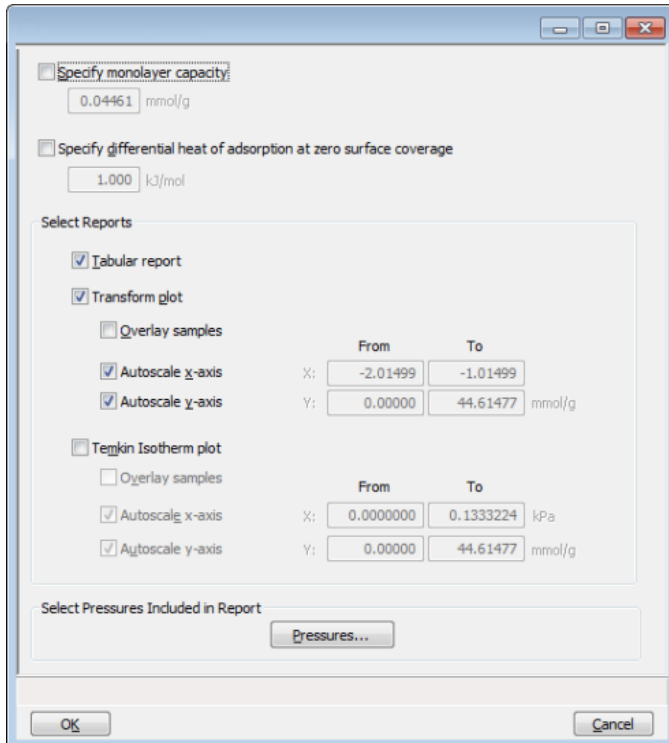
Field or Button	Description
	<p>curve is shipped with the analysis program and is found in the <i>Reference</i> directory.</p>  <p>To import values from an existing thickness curve (.THK file), click Open, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space or a tab.</p> <p>Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA. Select the thickness curve option, then click Edit. Modify the equation for the selected curve as needed.</p>
t-Plot	<p>Use to have a graphical representation of data generated.</p> <ul style="list-style-type: none"> • Overlay samples. Use to overlay sample files on the <i>t</i>-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.
 <p>For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.</p>	

TEMKIN REPORT OPTIONS



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

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

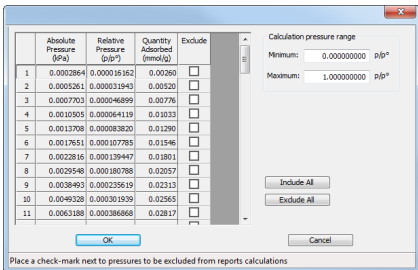


The screenshot shows the 'TEMKIN REPORT OPTIONS' dialog box. It contains several sections for configuring report output:


- Specify monolayer capacity:** A checkbox is checked, and the value is 0.04461 mmol/g.
- Specify differential heat of adsorption at zero surface coverage:** A checkbox is checked, and the value is 1.000 kJ/mol.
- Select Reports:**
 - ☒ Tabular report
 - ☒ Transform plot
 - ☐ Overlay samples
 - ☒ Autoscale x-axis: X: -2.01499 To -1.01499
 - ☒ Autoscale y-axis: Y: 0.00000 To 44.61477 mmol/g
 - ☐ Temkin Isotherm plot
 - ☐ Overlay samples
 - ☒ Autoscale x-axis: X: 0.0000000 To 0.1333224 kPa
 - ☒ Autoscale y-axis: Y: 0.00000 To 44.61477 mmol/g
- Select Pressures Included in Report:** A button labeled 'Pressures...' is present.

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

Temkin Report Options Fields and Buttons Table

Field or Button	Description
Pressures	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <ul style="list-style-type: none"> • Calculation pressure range. Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>. • Include All. Select to include all pressure points in the table. • Exclude All. Select to exclude all pressure points in the table.
Selected Reports	<ul style="list-style-type: none"> • Tabular Report. Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample. • Temkin transform plot. Plots a linear form of the Temkin transform plot. <ul style="list-style-type: none"> ◦ Autoscale x-axis. The X-axis field shows the logarithm of pressure (ln). ◦ Autoscale y-axis. The Y-axis field shows the quantity of gas adsorbed. ◦ Overlay samples. Use to overlay sample files on the transform plot. • Temkin isotherm plot. Overlays the Temkin isotherm with the analysis data. <ul style="list-style-type: none"> ◦ Overlay samples. Use to overlay sample files on the isotherm plot. ◦ Autoscale x-axis. Linear X-axes begin at zero. The X-axis field shows the absolute pressure. ◦ Autoscale y-axis. Y-axes begin at zero. The Y-axis field shows the quantity of gas adsorbed.
Specify differential heat of adsorption	<p>Select and enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all Temkin constants.</p>

Temkin Report Options Fields and Buttons Table (continued)

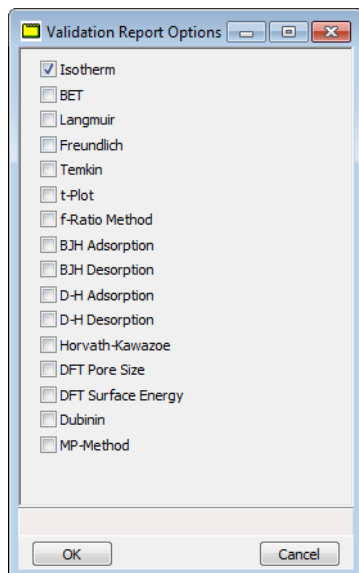
Field or Button	Description
Specify monolayer capacity	Select and enter the monolayer capacity of the sample.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

VALIDATION REPORT OPTIONS



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

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

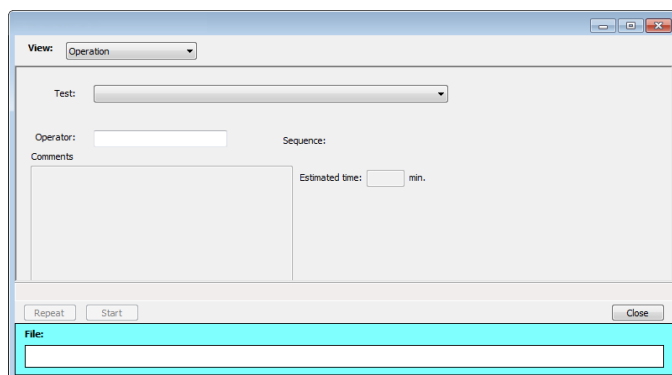


9 DIAGNOSTICS


START DIAGNOSTIC TEST

Unit [n] > Diagnostics > Start Diagnostic Test

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



Start Diagnostic Test Fields and Buttons Table

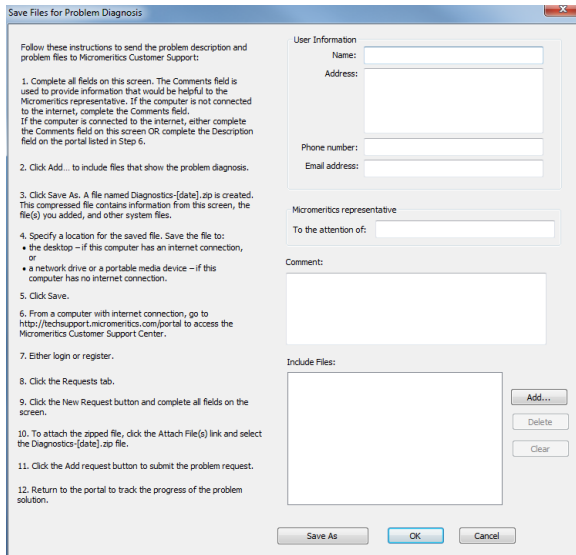
Field or Button	Description
Comments	Displays comments from the selected diagnostic test.
Estimated time (min.)	Approximate time for test completion.
File	Shows a status bar of steps complete once the test begins.
Next	Starts the next test.
Operator	Enter information to identify the person running the service test.
Repeat	Repeats the selected diagnostic test.
Report after test	Automatically generates reports to the selected destination when the test is complete.
Sequence	Sequence number assigned to the test.
Start	Starts the diagnostic test.
Test	Select the diagnostic test to be performed.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

SAVE FILES FOR PROBLEM DIAGNOSIS

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

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

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



Save Files for Problem Diagnosis

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:

Add...

Delete

Clear

Save As **OK** **Cancel**

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

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


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

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

Save Files for Problem Diagnostic Fields and Buttons Table

Field or Button	Description
Comment	Enter information that would be helpful to the Micromeritics representative. If the computer is not connected to the internet, complete this field. If the computer is connected to the internet, this information can be completed on the Micromeritics Customer Support portal.
Include Files	<ul style="list-style-type: none"> • Add. Click to select additional files to send with this problem diagnosis. To select more than one file, hold down the Ctrl key on the keyboard while selecting the files, or hold down the Shift key to select a range of files. • Delete. Select the file in the <i>Include Files</i> box, then click Delete to remove the file from the list. • Clear. Click to clear all files from the <i>Include Files</i> box.
Save As	Click to specify the name and location of the compressed file. Make a note of the file name and location. This file will need to be sent to your Micromeritics representative for problem resolution.

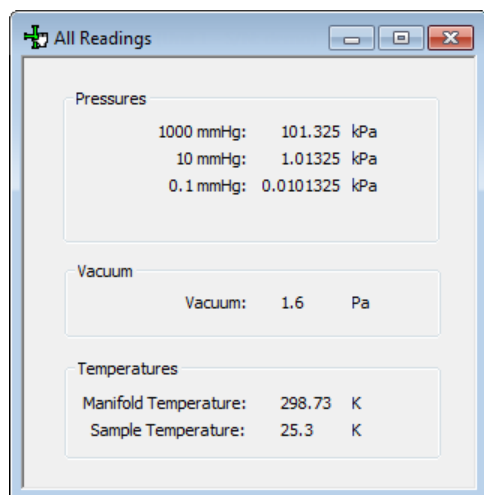
Save Files for Problem Diagnostic Fields and Buttons Table (continued)

Field or Button	Description
Micromeritics representative	Enter the name of your Micromeritics representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).
User Information	Enter information for the person to be contacted by a Micromeritics representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).
	For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.

SHOW ALL READINGS

Unit [n] > Diagnostics > Show All Readings

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



DIAGNOSTIC TEST REPORT

Unit [n] > Diagnostics > Diagnostic Test Report

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

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

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10 ABOUT CALIBRATION

Unit [n] > Calibration

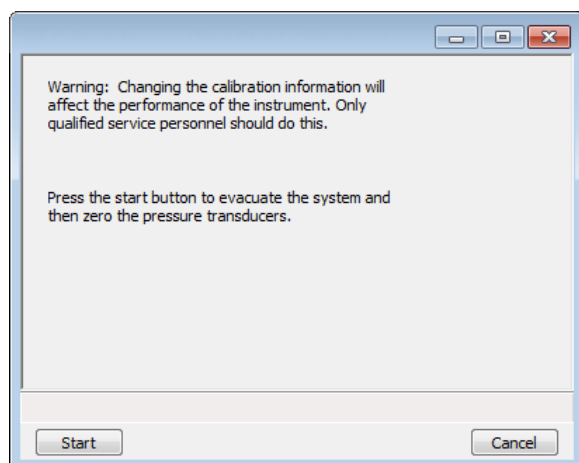
Use to perform system calibrations. Disabled calibration options can be accessed only with the assistance of an authorized Micromeritics service representative. Calibrations can be saved to a file and reloaded later.

CALIBRATE PRESSURE ZERO

Unit [n] > Calibration > Pressure Zero

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

The system automatically zeros the transducers before an analysis begins. Therefore, this procedure may not need to be performed unless operating the system in manual mode.



RESET

Set the pressure transducer and temperature sensor calibrations to nominal values.

Recalibration should be performed by a Micromeritics service representative.

LOAD CALIBRATION FROM FILE

Unit [n] > Calibration > Load from File

Use to load a previously saved calibration file.

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



Changing the calibration may affect the analyzer's performance.

SAVE CALIBRATION TO FILE

Unit [n] > Calibration > Save to File

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

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

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

11 HARDWARE COMPONENTS AND ACCESSORY INSTALLATION

FILL AND INSTALL AN ANALYSIS DEWAR

See [*Step 6 - Fill and Install the Dewar on page 6 - 9*](#)

ENABLE CRYOSTAT OPTION

Unit [n] > Enable Cryostat

A Cryostat is an available option. See [*Parts and Accessories on page 13 - 1*](#).

Select to enable the cryostat, if using this option.

SAMPLE TUBE INSTALLATION

See [*Step 5 - Install the Sample Tube on page 6 - 8*](#)

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12 TROUBLESHOOTING AND MAINTENANCE

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

What Happened	Why	What to Do
Analysis dewar cannot be raised or lowered	Dewar elevator stuck in up position, down position, or somewhere in between.	Check for possible obstruction to elevator movement.
Analysis valves cannot be operated.	Circuit opened by circuit breaker.	Reset breaker (depress breaker button) located on the right side of the instrument beneath the tray cover. See Front Components on page 1 - 2 . If the circuit breaker trips (pops out), call your Micromeritics service representative.
	Cable from computer to the instrument is loose.	Ensure the cable is seated properly.
Furnace cannot be raised or lowered	Elevator is stuck.	Check for possible obstruction to elevator movement.
Vacuum pump gurgles continuously.	Sample tube O-ring or fitting loose.	Tighten fitting. Replace O-ring.
	Sample tube 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.	Go to Unit [n] > Enable Manual Control to ensure a checkmark appears next the Enable Manual Control . Right click on the open valve and select <i>Close</i> .
Vacuum gauge shows reading above 20 μmHg, even after extended pumping through unrestricted valve with analysis port closed.	Filter in sample port is dirty.	Replace filter in port. See Replace Port Filters on page 12 - 10 .
	Leak in vacuum plumbing.	Check and tighten all connections in vacuum plumbing, including cold traps.
	Vacuum pump turned off or unplugged.	Check pump power plug and power switch.
	Dry roughing pump needs to be serviced.	Replace the diaphragms.

GUIDELINES FOR CONNECTING GASES

- Place gas cylinders within 6 feet (2 m) of the gas inlets of the instrument. Using gas line extenders on gas cylinders located in remote areas may degrade gas quality and reduce pressure. Gas lines are typically five to six feet long. Place the cylinders close enough to allow for proper connection at the analyzer inlet.
- Use a retaining strap (or other appropriate tether) to secure the gas cylinder.
- Always use the gas lines provided with the analyzer. It is very important that proper gas lines are used with the analyzer.
 - **Do not use** polymer tubing for the gas line.
 - **Do not use** flexible gas lines. Some flexible lines may appear to be appropriate, such as those with a herringbone covering, but the line may be coated internally with a polymer.
- Long gas lines, such as those used with gas cylinders placed in remote areas, must be evacuated for an extended period of time to remove ambient gases. When possible, avoid placing gas cylinders in remote locations. It is always better to have gas cylinders located near the analyzer.
- Carefully route the gas lines from the cylinder to the analyzer avoiding overlapping or entangling gas lines. This will help avoid confusion when maintenance is required.
- Label the gas line at the analyzer inlet for proper identification and maintenance.
- Replace gas cylinders before gas is depleted. It is best to replace a gas cylinder when the pressure reads approximately 200 psi (1500 kPa) on the high pressure gauge. Contaminants absorbed to the walls of the cylinder will desorb as the pressure decreases.
- Ensure the gas cylinder is closed before connecting to the analyzer.



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

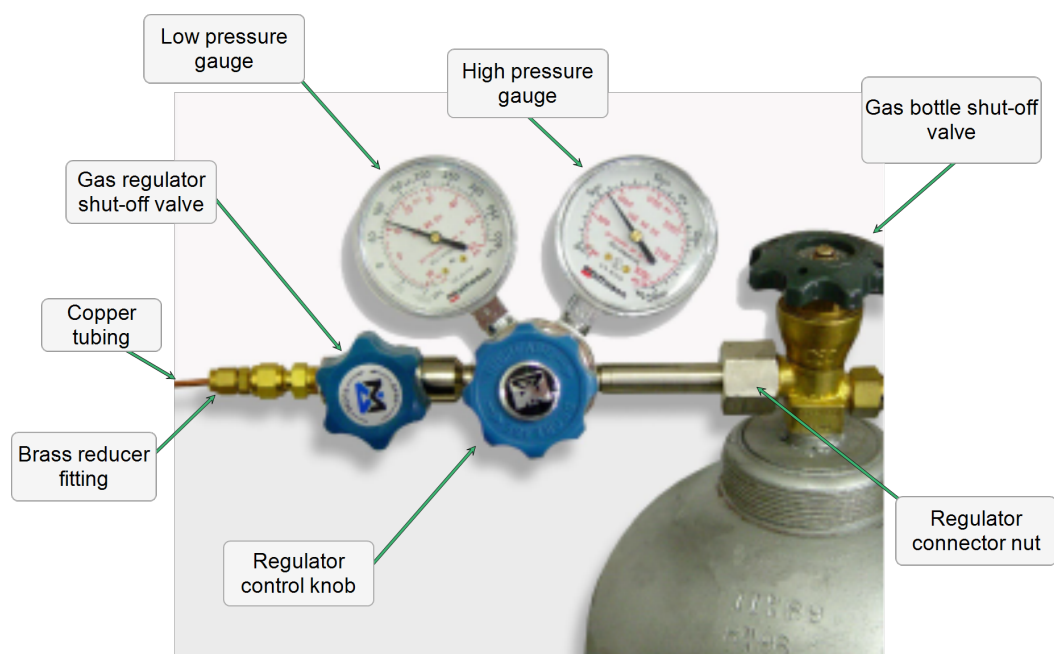
REPLACE A GAS CYLINDER



These instructions apply to working with inert gases only. When working with hazardous gases, follow the safety procedures established by your lab.



A power failure can result in dangerous pressures in the sample chamber. When using toxic or flammable gases, additional venting of the cabinet may be required.



Disconnect the Depleted Gas Cylinder

1. Close the regulator shut-off valve and gas cylinder shut-off valve by turning the knobs clockwise.
2. Disconnect the gas line from the regulator. Gas will be vented from the line. It is not necessary to disconnect the gas line from the analyzer inlet if the cylinder will be replaced immediately with one of the same type.
3. Open the gas regulator shut-off valve by turning the knob counter-clockwise. Gas will be vented from the regulator.
4. Turn the regulator control knob clockwise to open and vent any remaining gas. Both gauges should read at or near zero. If not, make sure the gas regulator shut-off valve is open.
5. Close the regulator by turning the control knob counter-clockwise.

6. Use an appropriate wrench to loosen the nut at the regulator connector nut then remove the regulator from the bottle.
7. Replace the protective cap on the depleted bottle. Disconnect the retaining strap and move the bottle to an appropriate location.

Connect A Gas Cylinder

Regulator Pressure Settings

Analyzer Series	Gauge should indicate
3Flex	15 psig (103 kPag)
AccuPyc	15 psig (103 kPag)
ASAP	15-17 psig (69 kPag)
AutoChem	5 psig (35 kPag)
AutoPore	45 - 50 psig (310 - 345 kPag)
Gemini	19.5 psig (134.4 kPag)
TriStar	15 psig (03 kPag)

Move the replacement bottle close to the analyzer and tether it into place.

1. Use an appropriate cylinder wrench to remove the protective cap from the replacement gas cylinder. Place the protective cap in a secure location. It will be needed to recap the gas cylinder when it is depleted and replaced.
2. Attach the gas regulator to the gas cylinder connector. Hand tighten the nut, then use an appropriate wrench to tighten an additional 3/4 turn.



Over-tightening the fitting may cause a leak.

3. Check for leaks at the high-pressure side of the regulator and in the connector.
 - a. Turn the regulator control knob fully counter-clockwise.
 - b. Slowly open the gas cylinder shut-off valve, then quickly close it.
 - c. Observe the pressure on the high-pressure gauge for approximately one minute.
 - If the pressure is stable, proceed with the next step.
 - If the pressure decreases, tighten the regulator connector nut until it becomes stable. If the pressure does not remain stable, remove the regulator and clean all contacts at the regulator connection, then reinstall the regulator.
4. Connect the gas line to the regulator. Use two 7/16 in. (11 mm) wrenches to tighten the gas line connection. One wrench holds the fitting steady and the other tightens the connector nut.

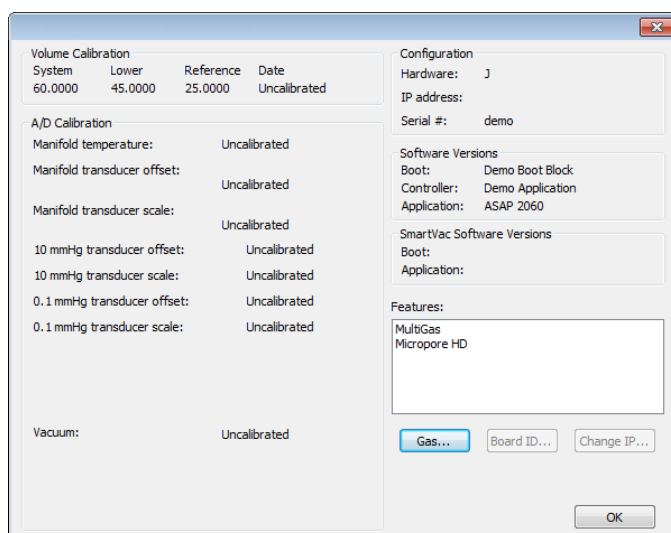
5. Open the gas cylinder shut-off valve.
6. Purge the air from the lines if the regulator or gas lines have been open to atmosphere for more than a short period of time.
 - a. Disconnect the gas line from the analyzer inlet if connected.
 - b. Turn the regulator control knob clockwise until the low-pressure gauge reads approximately 5 psi (30 kPa).
 - c. Open the regulator shut-off valve. Allow gas to flow for 30 seconds.
 - d. Close the regulator shut-off valve.
7. Connect the gas line to the analyzer if not already connected.
8. Open the regulator shut-off valve.
9. Turn the regulator control knob clockwise until the low-pressure gauge indicates the appropriate pressure. See [Regulator Pressure Settings on the previous page](#).

SPECIFY GAS PORTS

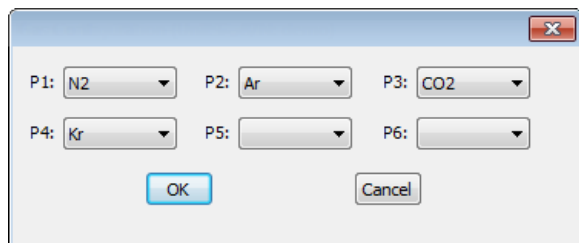
Unit [n] > Unit Configuration

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

The analyzer has gas inlets for up to six analysis gases. The gases connected to the inlets must be specified in the analysis program. If the gas is changed on one of the inlets, the same change must be made on the *Unit Configuration* window. The analysis software must be updated when gases change.




1. Click **Gas**.



2. Use the drop-down arrows to specify the type of gas installed on each port.
3. Click **OK** to close the window and save the changes.

Unit Configuration Fields and Buttons Table

Field or Button	Description
Calibration group boxes	Displays calibration information for analyzer components.
Configuration group box	<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 <i>Unit IP Setup</i> 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 display information from the electronic circuit boards in the instrument.. These parameters cannot be edited.
Features group box	Displays options installed on the analyzer.
Gas button	Displays ports for gas selections.
Software Versions group box	Displays the software versions of the MIC BIOS, controller, and analysis program.
 For fields and buttons not listed in this table, see the <i>Common Fields and Buttons</i> section of this operator manual.	

PREVENTIVE MAINTENANCE

Perform the following preventive maintenance procedures to keep the analyzer operating at peak performance. Micromeritics also recommends that preventive maintenance procedures and calibration be performed by a Micromeritics Service Representative every 12 months.

Maintenance Required	Frequency
Clean the Analyzer	As required or every 6 months
Lubricate elevator screw	As required or every 6 months. Use a light coat of lithium grease.
Check analysis port dewar	Weekly
Replace sample tube O-ring	As required or every 3 months
Replace port filters	Every 30 days
Replace diaphragm in vacuum pump **	Every 12 months
Calibrate manifold temperature sensor	Every 12 months
Calibrate system volume	Every 12 months
Check analyzer outgassing rate	Every 6 months
Test analyzer for leaks	As required or every 12 months
Perform reference material analysis	As required or every 3 months
** After about 12 to 18 months of operation, the diaphragm in the pump will wear out and become completely inoperable. To prevent any instrument downtime due to an inoperable pump, it is recommended that you have the diaphragm replaced by a Micromeritics Service Representative every 12 months.	

CHECK THE DEWAR



Review [Dewar Precautions on page 6 - 1](#) for information on handling dewars.



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

Ice and suspended frost particles may accumulate in the bottom of the analysis port dewar. Particles or deposits exceeding 1/4 in. (0.64 cm) in depth may jam between the bottom of the sample tube 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 tube. 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 on a weekly basis.

1. Remove the dewar from the instrument.
2. Pour out liquid nitrogen into an appropriate cryogenic container. Do not re-use liquid nitrogen.



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

3. Rinse the dewar with warm water to melt any remaining ice accumulation which may remain. Dry thoroughly.
4. Replace the dewar.

CLEAN THE ANALYZER

The exterior casing of the analyzer may be cleaned using a clean cloth, dampened with isopropyl alcohol (IPA), a mild detergent, or a 3% hydrogen peroxide solution. Do not use any type of abrasive cleaner.



- Do not allow liquid to penetrate the casing of the analyzer. Doing so could result in damage to the unit.
- Use only a mild detergent in water to clean safety shields. The use of isopropyl alcohol can damage the shield surface.

LUBRICATE THE ELEVATOR DRIVE ASSEMBLY

The elevator screw is lubricated before it leaves the factory and should not require lubricating. If the elevator starts to vibrate or becomes noisy when traveling, contact a Micromeritics Service Representative for disposition.

Should lubrication become necessary, apply a light coat of lithium grease to the elevator screw, accessed from the rear of the instrument, as needed.

PORT FILTERS

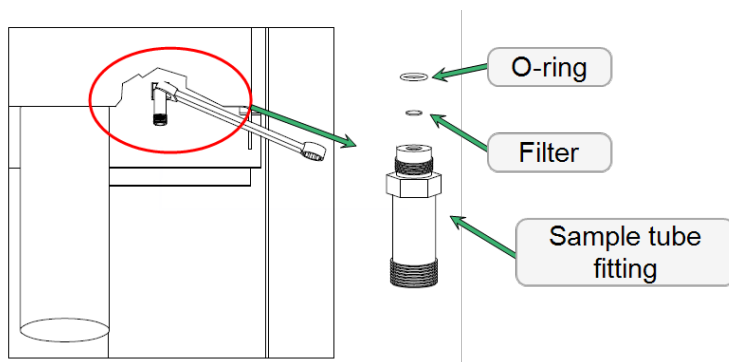
Replace Port Filters

A porous metal filter is located in the analysis port and in each degas port. Using a contaminated filter on the analysis port may extend the time required to achieve a vacuum at that port and the contaminant may also adsorb or desorb during analysis, affecting the analysis results. A contaminated filter on the analysis port may be detected by a leak test (if the contaminant outgasses) or by a free-space reading much lower than normal.

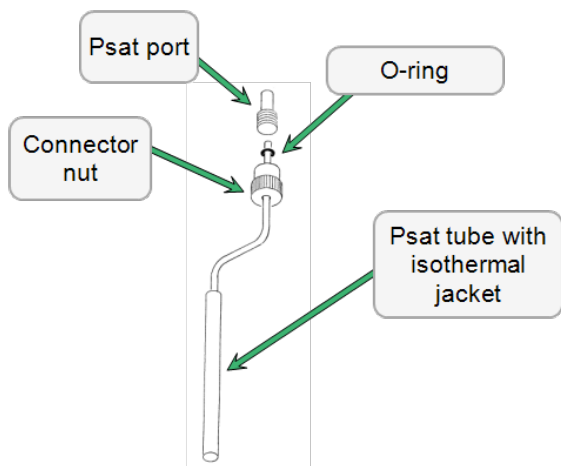
Replace the Analysis Port Filter



To avoid analysis problems, the new filter and O-ring must be clean. Wear gloves when performing this task. Do not touch the parts with bare hands.



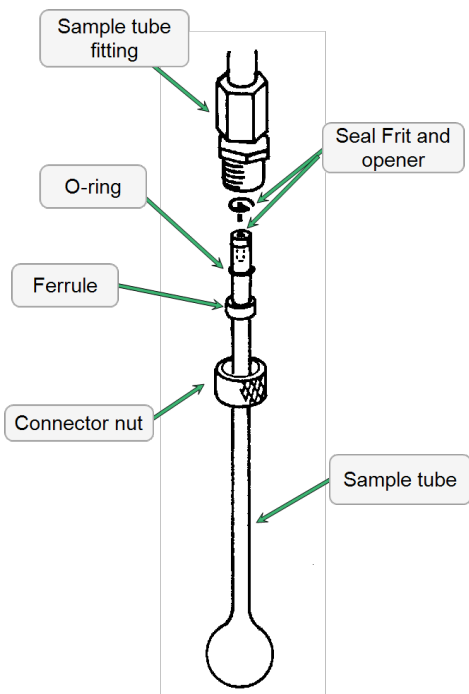
1. Remove the dewar and sample tube (or plug).
2. Close the sample valve.
3. If the O-ring and filter are stuck in the port, use a fingernail or plastic instrument to remove them. Using a metal instrument may scratch the sealing surface, causing a leak.
4. Replace the filter and the O-ring.
5. Carefully reassemble the sample tube fitting, filter, O-ring and manifold connector and hand tighten. Then tighten with a wrench to prevent leaks when evacuated.

REPLACE THE PSAT TUBE O-RING

1. Turn the connector nut counter-clockwise to loosen.
2. Pull the connector nut downward.
3. Gently pull the Psat tube downward to remove it from the Psat port.
4. Remove the O-ring from the Psat tube and replace with a new one.
5. Insert the Psat tube into the Psat port.
6. Slide the connector nut up to the Psat port and turn the connector nut clockwise to tighten.

REPLACE 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 analyzer schematic to verify valve status.

1. Carefully remove the dewar from the elevator. Take care not to bump the sample tube bulbs with the dewar during this process. Place the dewar aside.
2. Hold the sample tube firmly with one hand and loosen the sample tube connector nut by turning counter-clockwise.
3. Carefully pull the sample tube down until it is free from the port. It may be necessary to grasp the sample tube with both hands.
4. Remove the O-ring from the top of the sample tube and replace it with a new one.



If the O-ring remains inside the sample port, use a pair of tweezers or needle-nose pliers to remove it.

5. After the new O-ring is in place, insert the sample tube back into the sample port until it is fully seated.
6. Slide the sample tube connector nut up the tube until it comes in contact with the port fitting (the ferrule and O-ring will move along with the connector nut). Then, turning clockwise, hand tighten the connector nut to the sample connector.

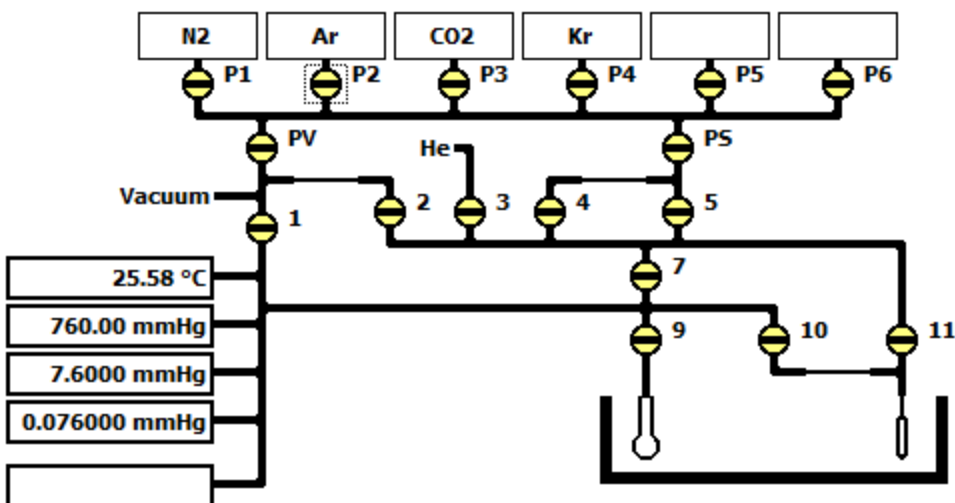
RECOVER FROM A POWER FAILURE

The analyzer saves entered and collected data in case of power failure. File parameters and any other data entered will still be present when power is restored. If an analysis was in progress when the power failure occurred, it will be canceled when the analyzer restarts. Any data collected during the analysis will still be present, but the analysis should be restarted in order to produce complete results.

ENABLE MANUAL CONTROL

Unit [n] > Enable Manual Control





Use to enable the manual control of valves and the elevator. When this option is enabled, a checkmark appears to the left of **Unit [n] > Enable Manual Control**. If the analyzer schematic is not immediately visible, go to **Unit [n] > Show Instrument Schematic**. The schematic should display automatically when manual control is enabled.



Analysis Valve Descriptions

Valve	Description
1	Unrestricted vacuum
2	Restricted vacuum
3	Helium inlet port valve
4	Restricted analysis gas
5	Unrestricted analysis gas
7	Lower manifold isolation
9	Sample port
10	Restricted Psat tube port
11	Unrestricted Psat tube port
P1 through P6	Gas inlet port valves
PS	Supply valve for physisorption gases
PV	Vacuum valve for physisorption gases

Analyzer Schematic Icon Table

Icon or Symbol	Description
	Open Valve. Green indicates an open valve.
	Closed Valve. Yellow indicates a closed valve. When manual control is disabled, closed valves appear white.
	Elevator.
	Sample Tube. Cannot be manually controlled.

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13 PARTS AND ACCESSORIES

Order system components and accessories using one of the following methods:

- Call our Customer Service Department at 1-770-662-3636
- Email orders to Orders@Micromeritics.com
- Contact your local sales representative

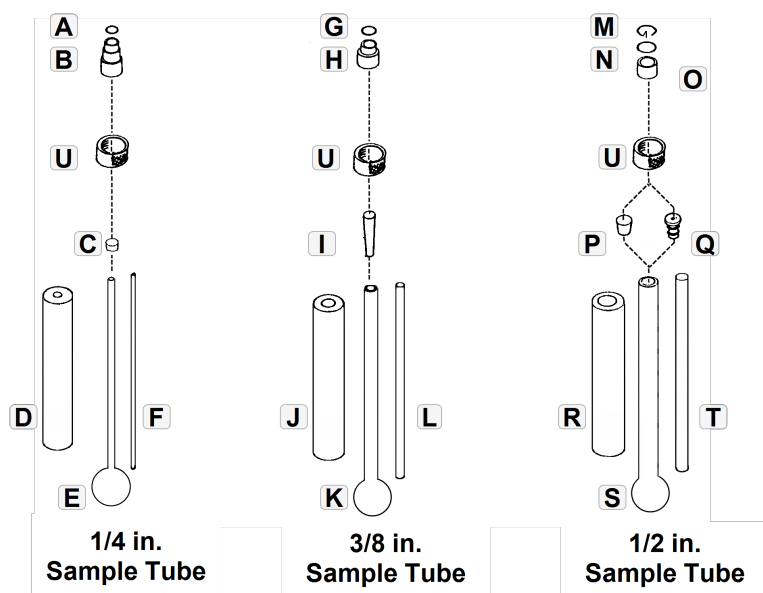
Part Number	Item and Description
Analyzer Optional Equipment	
021-00000-00	Model 021 LN ₂ transfer system, for easy filling of sample dewars; includes 44 L dewar, mobile platform, and pump with 100 cm discharge line
060-00000-24	FlowPrep 060, degasses up to six samples at up to 400 °C with flowing gas. A gas source and regulator are required.
061-00000-24	VacPrep 061, degasses up to six samples at up to 400 °C; using flowing gas or vacuum. A vacuum pump, gas source and regulator are required.
065-00000-24	SmartPrep 065, Windows interface provides programmable ramp and soak rate for degassing up to six samples with flowing gas. A gas source and regulator are required.
246-33010-00	MicroPrep Option, temperature controller and heating mantle for in-situ degassing.
Cables	
003-63801-01	Cable, Ethernet straight-thru
Chiller Dewar Parts and Accessories	
025-25820-00	Hose Assembly (replacement), for connecting the Chiller Dewar to a recirculating bath (2 each required per Chiller Dewar assembly)
025-63801-00	Recirculating Bath Temperature Control RTD, replacement for the RTD cable supplied in the Recirculating Bath Temperature Control RTD Kit
Dewar and Accessories	
025-00202-00	Chiller Dewar Option, permits connection of recirculating bath to cryogen dewar to control analyses at a variety of temperatures.
025-26002-00	Recirculating Bath, -28 °C TO 200 °C, 230V 50HZ, refrigerated and heating circulator for temperature control of the bath in the Chiller Dewar.
025-26004-00	Recirculating Bath, -28 °C TO 200 °C, 115V 60HZ, refrigerated and heating circulator for temperature control of the bath in the Chiller Dewar.

Part Number	Item and Description
025-33001-00	Recirculating Bath Temperature Control RTD Kit, optional RTD mounted in the Chiller Dewar bath and connected to the recirculating bath controller. Includes RTD cable assembly and mounting bracket. Note: The connector on the RTD cable assembly is designed to plug into a Micromeritics supplied recirculating bath.
202-14702-00	Dewar shield, for use with Chiller Dewar Option
202-25850-00	Dewar, 3 liter, analysis
202-31707-00	Dewar lid, analysis
240-25901-00	Dewar depth gauge
Gas Cylinder Accessories and Replacement Parts	
004-25103-00	Ferrule, front, teflon 1/4 in., for use with 1/8 in. tube × 1/4 in. tube reducer
004-25104-00	Ferrule, rear, nylon 1/4 in., for use with 1/8 in. tube × 1/4 in. tube reducer
004-25549-00	Reducer, 1/8 in tube × 1/4 in. tube, brass, accepts 1/8 in. tube, connects to 1/4 in. swage fittings
004-25913-00	1/8 Peek Ferrule Set for sealing gas inlet lines into the gas inlet manifold assemblies
004-33601-00	Expansion Kit, adds an additional outlet to the gas regulator, includes fittings and instructions
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 pressure regulator, CGA 320 fitting (CO ₂), 30 psig
004-62230-35	Gas pressure regulator, CGA 350 fitting (CO, H ₂), 30 psig
004-62230-54	Gas pressure regulator, CGA 540 fitting (O ₂), 30 psig
004-62230-58	Gas pressure regulator, CGA 580 fitting, 30 psig (He, N ₂ , Kr, Ar)
004-62230-326	Gas pressure regulator, CGA 326 fitting (N ₂ O), 30 psig
004-62230-705	Gas pressure regulator, CGA 705 fitting (NH ₃), 30 psig
201-25818-00	Gas Inlet Line, 6 ft, SS
201-25818-01	Gas Inlet Line, 16 ft (5 m), SS
290-25846-00	Gas Inlet Line, 1/8 in × 6 ft, copper
290-25846-01	Gas Inlet Line, 1/8 in × 16 ft (5 m), copper
Heating Mantle	
003-26043-00	Heating mantle with type K thermocouple, 450 °C maximum, 24 V
003-26045-00	Heating mantle with type K thermocouple, 450 °C maximum, 24 V, side-laced to accommodate monolithic & nonstandard sample tubes
230-25808-00	Heating mantle clip

Part Number	Item and Description
MicroPrep Parts and Accessories	
003-26043-00	Heating mantle with type K thermocouple, 450 °C maximum, 24 V
230-25808-00	Heating mantle clip
Operating Supplies and Replacement Parts	
004-25006-00	O-ring, size 006, Buna-N, for P ₀ tube
004-25466-00	O-ring, -010 70 Durometer Buna-N, for sealing frit in sample tube port
004-27041-00	Frit, µm SS 1/4D × 1/16T, for replacement in sample tube port
004-54130-00	Removal tool for CheckSeal opener
200-25840-00	Saturation pressure tube, includes isothermal jacket
206-14701-00	Dewar shield, for use on sample port, cold trap, and degas ports
206-33010-06	Physisorption operating supplies (6 month supply), O-rings, sample port frits, 1/8 Peek ferrule sets, sample tubes, filler rod.
206-33010-12	Physisorption operating supplies (12 month supply), O-rings, sample port frits, 1/8 Peek ferrule sets, sample tubes, filler rods, sample tube brush, isothermal jacket, dewar lid, sample tube stoppers, reference material
Reference Material	
004-16816-00	Reference material, low surface area (krypton)
004-16821-00	Reference material, high surface area
004-16843-00	Reference material, micropore, 13X
004-16844-00	Reference material, micropore, Y-zeolite
Sample Tube Accessories	
004-54609-00	Brush for cleaning sample tube
004-54618-00	Tool, for removing sample port O-ring
202-33052-00	22 mm ID Sample Tube Kit (see table for kit contents)
240-14855-00	Sample tube rack
240-25853-00	Sample tube funnel
240-32805-00	Sample tube support, assists sample weighing
Software and Manuals	
202-33027-00	MicroActive Software Package, interactive data analysis software
206-20800-00	ASAP 2060 Software program
206-33001-00	Software Package, ASAP 2060; includes current version of software and operator manual.
206-42800-00	ASAP 2060 Operator Manual (includes binder and dividers)
Vacuum Pump and Accessories	

Part Number	Item and Description
004-62023-01	Service kit, for Vaccubrand dry forepump
206-33002-00	Vacuum Connection Expansion Kit (2nd or 4th Unit). Tee, flexible stainless steel tubing, centering rings, and clamps required for connecting a 2nd or 4th unit to the free-standing hybrid-turbo vacuum system.
206-33003-00	Vacuum Connection Expansion Kit (3rd Unit). Tee, flexible stainless steel tubing, centering rings, and clamps required for connecting a 3rd unit to the free-standing hybrid-turbo vacuum system.
Valves	
201-22600-00	Valve spring, for Buna-N plunger, for analysis manifold
250-25608-00	Valve gasket, Kel-F, for analysis manifold
250-25627-00	Valve plunger, Buna-N seal, for analysis manifold

SAMPLE TUBES AND COMPONENTS



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

	Part Number	Item and Description
A	004-25466-00	O-ring, size 010, Buna-N, for 1/4 in. sample tube
B	240-25803-00	Ferrule, 1/4 in.
C	004-32604-00	Cap (stopper) for 1/4 In. sample tube (not shown)
D	202-25901-00	Isothermal jacket, 1/4 in.
E	240-61001-00	Sample tube, 1/4 in.

1/4 in., 3/8 in., and 1/2 in. Sample Tubes (continued)

	Part Number	Item and Description
F	240-61014-00	Volume displacement insert, 1/4 in.
G	004-25022-00	O-ring, size 012, Buna-N, for 3/8 in. sample tube
H	240-25802-00	Ferrule, 3/8 in.
I	004-32004-00	Stopper, for 3/8 in. sample tubes
J	202-25902-00	Isothermal jacket, 3/8 in.
K	240-61002-00	Sample tube, 3/8 in.
L	240-61015-00	Volume displacement insert, 3/8 in.
M	260-25891-00	Opener, seal frit, for 1/2 in. sample tube
N	004-25044-00	O-ring, size 013, Buna-N, for 1/2 in. sample tube
O	260-25843-00	Ferrule, 1/2 in.
P	240-32000-00	Stopper, for 1/2 in. sample tube
Q	260-25890-00	Seal Frit with built-in check valve for air-sensitive samples
R	202-25903-00	Isothermal jacket, 1/2 in. sample tube
S	240-61003-00	Sample tube, 1/2 in.
T	240-61016-00	Volume displacement insert, 1/2 in.
U	300-25824-00	Nut, sample tube

22 mm Sample Tube Kit (part number 202-33052-00)

Part Number	Item and Description
004-25609-02	O-ring, -022, 70 Buna-N
004-32230-00	Stopper, rubber, for 22 mm ID tube
202-25868-00	Fitting, sample port, 25 mm sample tube
202-25869-00	Nut, for 25 mm sample tube
202-25870-00	Ferrule for 25 mm sample tube
202-25871-00	Fitting, degas port, 25 mm sample tube
202-25904-00	Isothermal jacket, for 25 mm OD sample tube
202-31709-00	Dewar lid, 25 mm OD sample tube
202-61022-00	Sample tube, 22 mm ID, 25 mm OD
202-61022-01	Filler rod for 22 mm ID sample tube

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A CALCULATE FREE SPACE VALUES FOR MICROPORE ANALYSES

Many microporous materials, such as zeolites and activated carbons, trap and hold helium in their complex pore structures for many hours after being exposed to helium. Helium trapped in micropores can interfere with the analysis at low pressures, causing an “S”-shaped curve at the lower end of the isotherm. For this reason, it is recommended that you enter the warm and cold free-space volumes when performing micropore analyses, therefore avoiding exposure of the sample to helium. Two techniques can be used for determining warm and cold free-space values.

The first method is to perform a short analysis on the sample after partial degassing (one pressure point with no incremental dosing), but prior to final sample preparation. Measure the free space during this analysis. The measured free-space values will be printed on the report and may then be entered into the sample file after more thorough sample preparation.

The second method requires prior tests using empty tubes that will be employed later for the sample analyses. The measured free-space data can be used thereafter on every analysis performed using these sample tubes. This small initial investment of time will save considerable time later. Perform an empty tube analysis on each sample tube you intend to use for micropore analysis. Measure the free space of each sample tube, taking only one pressure point.

1. Create a sample tube file from each empty tube test.
2. Use the Load from Sample File button to read in the measured free space from the empty tube tests.
3. In the sample file for the sample analysis, pick the appropriate sample tube from the sample tube list.
4. Enter the mass and density of the sample.
5. Choose Calculate in the free space options in the analysis conditions.

$$V_{ws} = V_{wm} - \left(\frac{M_s}{\rho} \frac{T_{std}}{T_{amb}} \right)$$

where

M_s	=	mass of sample to be analyzed (in grams)
T_{amb}	=	ambient temperature (in kelvins)
T_s	=	approximate sample true density (in grams/cm ³)
T_{std}	=	standard temperature (273.15 K)
V_{wm}	=	warm free space measured for the empty tube (in cm ³ STP/atm)
V_{ws}	=	calculated warm free space with sample present (in cm ³ STP/atm)

To correct cold free space:

$$V_{cs} = V_{cm} - (M_s T_{std} \div \rho T_{bath})$$

ρ	=	approximate sample true density (in grams/cm ³)
M_s	=	mass of the sample to be analyzed (in grams)
T_{bath}	=	analysis bath temperature (in kelvins)
T_{std}	=	standard temperature (273.15 K)
V_{cm}	=	cold free space measured for the empty tube (in cm ³ STP/atm)
V_{cs}	=	calculated cold free space with sample present (in cm ³ STP/atm)

B CALCULATIONS

ALPHA-S METHOD

The alpha-S curve is calculated from the reference isotherm by dividing each quantity adsorbed by the quantity adsorbed at 0.4 relative pressure.

$$\alpha_s = Q/Q_{0.4}$$

where $Q_{0.4}$ is found by linear interpolation.

A least-squares analysis fit is performed on the (α_p ,Q) pairs. The following are calculated:

- Slope (s cm³/g STP)
- Y-intercept (Q_0 cm³/g STP)
- Uncertainty of the slope (cm³/g STP)
- Uncertainty of the Y-intercept (cm³/g STP)
- Correlation coefficient

Surface area is calculated as:

$$A_s = A_{ref} s / Q_{0.4}$$

where A_{ref} is the entered reference surface area.

Pore size is calculated as:

$$V_p = Q_0 / D$$

BET SURFACE AREA

The BET¹⁾ transformation is calculated as:

$$y = \frac{1}{Q(\rho^0 / \rho - 1)}$$

A least-squares fit is performed on the (P_{rel} , y). The following are calculated:

- Slope ($S \text{ cm}^3/\text{g STP}$)
- Y-intercept ($y_0 \text{ cm}^3/\text{g STP}$)
- Uncertainty of the slope ($u(s) \text{ cm}^3/\text{g STP}$)
- Uncertainty of the Y-intercept ($u(y_0) \text{ cm}^3/\text{g STP}$)
- Correlation coefficient

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

BET Surface Area (m^2/g):

$$A_s = \frac{A_m N_A}{V_{m(s+y_0)}} \times 10^{-18} \frac{\text{m}^2}{\text{nm}^2}$$

BET C value:

$$C = s / y_0 - 1$$

Quantity in the Monolayer ($\text{cm}^3/\text{g STP}$):

$$Q_m = 1 / C y_0 = \frac{1}{s + y_0}$$

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

$$u(A_s) = \frac{\sqrt{u^2(s) + u^2(y_0)}}{s + y_0}$$

¹⁾ Brunauer, S.; Emmett, P.H.; and Teller, E., J. Am. Chem. Soc. 60, 309 (1938).

BJH PORE VOLUME AND AREA DISTRIBUTION

For adsorption data, the relative pressure and quantity adsorbed data point pairs collected during an analysis must be arranged in reverse order from which the points were collected during analysis. All calculations are performed based on a desorption model, regardless of whether adsorption or desorption data are being used.

The data used in these calculations must be in order of strictly decreasing numerical value. Points which do not meet this criterion are omitted. The remaining data set is composed of relative pressure (P), quantity adsorbed (Q) pairs from (P_1, Q_1) to (P_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 Kelvin¹⁾ 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 Halenda²⁾ developed the method (known as the BJH method) which incorporates these ideas. The algorithm used is an implementation of the BJH method.

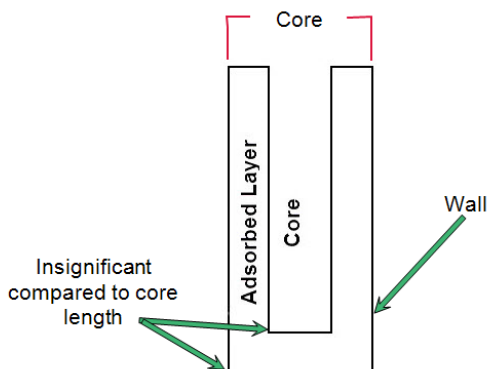
EXPLANATION OF TERMS

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.

¹⁾ Kelvin, J. (published under the name of Sir William Thomson), Phil. Mag. 42, 448-452 (1871).

²⁾ Barrett, E.P.; Joyner, L.S.; and Halenda, P., J. Am. Chem. Soc. 73, 373-380 (1951).



CALCULATIONS

The quantities adsorbed (Q_a) are converted to the liquid equivalent volumes (V_l , cm^3/g):

$$V_l = \frac{Q_i V_{mol}}{22414 \text{ cm}^3 \text{ STP}}$$

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 <i>BJH Adsorptive Options</i> window)
F	=	fraction of pores open at both ends (from the <i>BJH Adsorption Report Options</i> window or the <i>BJH Desorption Report Options</i> 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 on page B - 45](#).

For the last pressure interval from the lowest Pr_i to zero relative pressure, reference the calculations from the equations in [Thickness Curve on page B - 45](#).

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 ($Vd_i = 0$), and the remainder of Step (a) is skipped.

The change in thickness of the wall layer due to desorption from previously opened pores is calculated as:

$$\Delta Tw = Tw_1 - Tw_{i+1}$$

The annular cross-sectional area of the wall layer desorbed is calculated for all previously opened pores:

$$CSA_j = \pi \left[\left(Rc_j + \Delta Tw \right)^2 - Rc_j^2 \right] \left(10^{-16} \frac{cm^2}{\text{\AA}^2} \right)$$

The total volume of gas desorbed from walls of previously opened pores is calculated:

$$Vd_i = \sum_j \left(LP_j \right) \left(CSA_j \right) \quad \text{for all previously opened pores}$$

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:

$$SA_w = \sum_j \pi \left(LP_j \right) \left(D_{avg,j} \right) \left(\frac{10^{-8} cm}{\text{\AA}} \right) \quad \text{for all previously opened pores}$$

where

$D_{avg,j}$ = weighted average pore diameter calculated in Step b.2.

A new layer thickness (ΔTw) that will not overcompensate for the actual volume desorbed in this interval is calculated:

$$\Delta Tw = \frac{\left(VI_i - VI_{i+1} \right) \left(10^8 \frac{\text{\AA}}{\text{cm}} \right)}{SAw_i}$$

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_I to zero relative pressure, no new pore volume is calculated and the rest of Step b is skipped.)

The volume desorbed from newly opened pores in this interval is calculated as:

$$Vc_i = (VI_i - VI_{i+1}) - Vd_i$$

The Kelvin radius for the end of the interval is calculated as:

$$Rc_{k+1} = \frac{-A}{(1+F) \ln(P_{i+1})}$$

All new pores opened in this interval are represented by one pore having a length-weighted average pore diameter and a corresponding length sufficient to account for the required volume of adsorbate. The weighted average pore diameter is calculated as:

$$D_{avg,k} = \frac{2(Rc_k + Rc_{k+1})(Rc_k)(Rc_{k+1})}{Rc_k^2 + Rc_{k+1}^2}$$

$D_{avg,k}$ is the diameter of a pore which would have a surface area that is the average of the areas for pores radius Rc_k and Rc_{k+1} , if its length was the mean of the lengths at those radii.

The relative pressure corresponding to $D_{avg,k}$ is calculated as:

$$P_{avg,k} = \ln^{\exp} \left[\frac{-2A}{(1+F)(D_{avg,k})} \right]$$

The thickness of the adsorbed layer at this pressure is calculated as:

$$Tw_{avg,k} = HP1 \left[\frac{HP2}{\ln(P_{avg,k})} \right]^{HP3}$$

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:

$$\Delta Td = Tw_{avg,k} - Tw_{i+1}$$

The cross-sectional area of the newly opened pores is calculated as:

$$CSAc_k = \left[\frac{D_{avg,k}}{2} + \Delta Td \right]^2 \left(\frac{10^{-16} cm^2}{\text{\AA}^2} \right)$$

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:

$$D_{avg,k,new} = D_{avg,k,old} + 2(\Delta Td)$$

The layer thickness change during the whole interval is added to diameters of previously opened pores as:

$$D_{avg,k,new} = D_{avg,k,old} + 2(\Delta Tdw)$$

(not including $D_{avg,k}$)

The layer thickness change desorbed during this interval also is added to the radii corresponding to the ends of the pressure intervals as:

$$Rc_{j,new} = Rc_{j,old} + \Delta Tw$$

for all except Rc_{k+1} .

Steps a to c are repeated for each pressure interval.

After the above calculations have been performed, the diameters corresponding to the ends of the intervals are calculated as:

$$Dp_j = 2(Rc_j)$$

for all Rc_j including Rc_{k+1} .

The remaining calculations are based on Dp_i , $D_{avg,i}$ and LP_i . These calculations are only done for $D_{avg,i}$ values that fall between the Minimum BJH diameter and the Maximum BJH diameter specified by the operator on the *BJH Adsorption Report Options* window or the *BJH Desorption Report Options* window.

(1) Incremental Pore Volume (Vp_i , cm³/g):

$$Vp_i = \pi \left(LP_i \right) \left[\frac{D_{avg,i}}{2} \right]^2 \left[\frac{10^{16} cm^2}{\text{\AA}^2} \right]$$

(2) Cumulative Pore Volume ($VP_{cum,i}$, cm³/g):

$$VP_{cum,i} = \sum_j Vp_j \text{ for } (J \leq 1)$$

(3) Incremental Surface Area (SA_i , m²/g):

$$SA_i = \pi \left(LP_i \right) \left(\frac{10^{-2} m}{cm} \right) \left(D_{avg,i} \right) \left(\frac{10^{-10} m}{\text{\AA}} \right)$$

(4) Cumulative Surface Area ($SA_{cum,i}$, m²/g):

$$SA_{cum,10} = \sum SA_j \quad \text{for } J \leq 1$$

(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):

$$\frac{dV}{d\log D_i} = \frac{VP_i}{\log\left(\frac{Dp_i}{Dp_{i+1}}\right)}$$

(7) dA/dD pore area $(dA/dD)_i$ m²/g-A):

$$\frac{dA}{dD_i} = \frac{SA_i}{Dp_i - Dp_{i+1}}$$

(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 $(DF_{avg,j})$ A):

$$DF_{avg,j} = \frac{DP_{F_j} + DP_{F_{j+1}}}{2}$$

calculated for all intervals in the fixed pore size table.

For the intervals with between the Minimum BJH diameter and the Maximum BJH diameter.

(2) Cumulative Pore volume $(VPF_{cum,i})$ cm³/g):

$$VPF_{cum,i} = \text{INTERP}(DP_{F_{i+1}})$$

where $\text{INTERP}(x)$ is the value interpolated from the function $X = DP_{j+i}$ and

$Y = VPF_{cum,i}$ using an AKIMA semi-spline interpolation.

(3) Incremental Pore Volume (VPF_i) cm³/g):

$$VPF_i = VPF_{cum,i} - VPF_{cum,i-1}$$

where $VPF_{cum,0} = 0$.

(4) Cumulative Surface Area $(SAF_{cum,i})$ m²/g):

$$SAF_{cum,i} = INTERP(DpF_{i+1})$$

where $INTERP(x)$ is the value interpolated from the function $X = Dp_{j+i}$ and

$$Y = SA_{cum,j}$$

(5) Incremental Surface Area (SAF_i , m²/g):

$$SAF_i = SAF_{cum,i} - SAF_{cum,i-1}$$

where $SAF_{cum,0} = 0$.

(6) dV/dD pore volume ($dV/dDpF_i$, cm³/g-A):

$$\frac{dV}{dDpF_i} = INTERP(DpF_{i+1})$$

where $INTERP(x)$ is the value interpolated from the function $X = D_{avg,j}$ and

$$Y = dV/dD_j$$

(7) $dV/d\log(D)$ pore volume [$dV/d\log(DpF_i)$, cm³/g]:

$$\frac{dV}{d\log(DpF_i)} = INTERP(DpF_{i+1})$$

where $INTERP(x)$ is the value interpolated from the function $X = D_{avg,j}$ and

$$Y = dV/d\log(D)_j$$

(8) dA/dD pore area ($dA/dDpF_i$, m²/g-A):

$$\frac{dA}{dDpF_i} = INTERP(DpF_{i+1})$$

where $INTERP(x)$ is the value interpolated from the function $X = D_{avg,j}$ and

$$Y = dA/dD_j$$

(9) $dA/d\log(D)$ pore area [$dA/d\log(DpF_i)$, m²/g]:

$$\frac{dA}{d\log(DpF_i)} = INTERP(DpF_{i+1})$$

where $INTERP(x)$ is the value interpolated from the function $X = D_{avg,j}$ and

$$Y = dA/d\log(D)_j$$

COMPENDIUM OF VARIABLES

ΔT_d	=	thickness of layer desorbed from walls of newly opened pores (Å)
ΔT_w	=	thickness of adsorbed layer desorbed during interval (Å)
A	=	adsorbate property factor; from the <i>BJH Adsorptive Options</i> window
CSA	=	analysis gas molecular cross-sectional area (nm ²), user-entered on the <i>Adsorptive Properties</i> window
CSA_a	=	annular cross-sectional area of the desorbed layer (cm ²)
CSA_c	=	cross-sectional area of opening of newly opened pores (cm ²)
D_{avg}	=	average pore diameter (Å)
D_p	=	pore (or core) diameter (Å)
F	=	fraction of pores open at both ends; from the <i>BJH Adsorption Report Options</i> window or the <i>BJH Desorption Report Options</i> window
LP	=	length of pore (cm/g)
P	=	relative pressure
Q	=	quantity adsorbed expressed as a volume (cm ³ /g STP)
R_c	=	Kelvin radius (Å) of core
SA_w	=	total surface area of walls exposed (cm ² /g)
T_w	=	thickness of remaining adsorbed wall (Å)
V_c	=	volume desorbed from cores of newly opened pores (cm ³ /g)
V_d	=	volume of gas desorbed from walls of previously opened pores (cm ³ /g)
VI	=	liquid equivalent volume of volume adsorbed (cm ³ /g)
V_{mol}	=	liquid molar volume, from the fluid property information

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 Olivier¹⁾ 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

$$Q(p) = \int da db dc... q(p, a, b, c...) f(a, b, c...) \quad (1)$$

where

$Q(p)$	=	the total quantity adsorbed per unit weight at pressure p ,
$a, b, c, ...$	=	a set of distributed properties,
$f(a, b, c, ...)$	=	the distribution function of the properties, and
$q(p, a, b, c, ...)$	=	the kernel function describing the adsorption isotherm on unit surface of material with fixed properties $a, b, c, ...$

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.

¹⁾ Ross and Olivier, J.P., "On Physical Adsorption," J. Wiley and Sons, New York (1964).

APPLICATION TO SURFACE ENERGY DISTRIBUTION

Under certain conditions, an energetically heterogeneous surface may be characterized by a distribution of adsorptive energies. The conditions are that the sample is not microporous, i.e., that adsorption is taking place on essentially a free surface with no pore filling processes at least to about 0.2 relative pressure. Secondly, that each energetically distinct patch contributes independently to the total adsorption isotherm in proportion to the fraction of the total surface that it represents. This condition is satisfied if the patches are relatively large compared to an adsorptive molecule, or if the energy gradient along the surface is not steep. In mathematical terms, this concept is expressed by the integral equation of adsorption in the following form.

$$Q(p) = \int d\epsilon \, q(p, \epsilon) f(\epsilon) \quad (2)$$

where

$Q(p)$	=	the experimental quantity adsorbed per gram at pressure p ,
$q(p, \epsilon)$	=	the quantity adsorbed per unit area at the same pressure, p , on an ideal free surface of energy ϵ , and
$f(\epsilon)$	=	the total area of surface of energy ϵ in the sample.

The exact form of the energy-dependent term depends on the form of the model isotherms expressed in the kernel function and is provided in the model description.

APPLICATION TO PORE SIZE DISTRIBUTION

Similarly, a sample of porous material may be characterized by its distribution of pore sizes. It is assumed in this case that each pore acts independently. Each pore size present then contributes to the total adsorption isotherm in proportion to the fraction of the total area of the sample that it represents. Mathematically, this relation is expressed by

$$Q(p) = \int dH \, q(p, H) f(H) \quad (3)$$

where

$Q(p)$	=	the experimental quantity adsorbed at pressure p ,
$q(p, H)$	=	the quantity adsorbed per unit area at the same pressure, p , in an ideal pore of size H , and
$f(H)$	=	the total area of pores of size H in the sample.

Numerical values for the kernel functions in the form of model isotherms can be derived from modern statistical mechanics such as density functional theory or molecular simulations, or can be calculated from one of various classical theories based on the Kelvin equation. Several types are found in the models library.

PERFORMING THE DECONVOLUTION

The integrations in equations (2) and (3) are carried out over all surface energies or pore sizes in the model. The functions $q(p, \varepsilon)$ and $q(p, H)$, which we call the kernel functions, are contained in numeric form as model isotherms. Because, in general, there is no analytic solution for equation (1), the problem is best solved in a discrete form; the integral equation for any distributed property Z becomes a summation

$$Q(p) = \sum_i q(p, Z_i) f(Z_i) \quad (4)$$

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.

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.

$$\overline{D}_i = \left(\frac{D_i + D_{i+1}}{2} \right)$$

PORE LENGTH

$$l_i = \frac{A_{p,i} + 10^8}{\pi \overline{D}_i}$$

$$A_{p,i} = \frac{4 \times (10^8 \Delta V_p)}{\overline{D}_i}$$

$$\Delta V_p = C_v \left(D \left(Q_{i-1} - Q_i \right) - \Delta t \times 10^8 \left(A_{p,cum} - 2\pi \bar{t} l_{i,cum} \right) \right)$$

$$C_v = \left(\frac{\overline{D}_i}{2(\overline{r}_k + t(P_i) - t(P_{i+1}))} \right)^2$$

$$\bar{t} = \frac{\overline{D}_i}{2 - \overline{r}_k}$$

$$\overline{r}_k = \frac{(r_{k,i} + r_{k,i+1})}{2}$$

where

$$\Delta V_p = \text{Change in pore volume}$$

$A_{p,i}$	=	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
\overline{r}_k	=	Average Kelvin radius
\overline{t}	=	Average thickness

DUBININ-ASTAKHOV

The Dubinin-Astakhov equation is:

$$\log(Q) = \log(Q_0) - \left(\frac{RT}{\beta E_0}\right) \times \left(\log \frac{P_0}{P}\right)^N$$

where

β	=	the affinity coefficient of the analysis gas relative to the P_0 gas, from the <i>Dubinin Adsorptive Options</i> window
E_0	=	characteristic energy (kJ/mol)
N	=	Astakhov exponent, may be optimized or user entered from the <i>Dubinin Report Options</i> window
P	=	equilibrium pressure
P_0	=	saturation vapor pressure of gas at temperature T
Q	=	quantity adsorbed at equilibrium pressure (cm ³ /g STP)
Q_0	=	the micropore capacity (cm ³ /g STP)
R	=	the gas constant (0.0083144 kJ/mol)
T	=	analysis bath temperature (K)

For each point designated for Dubinin-Astakhov calculations, the following calculations are done:

$$LV = \log(Q)$$

$$LP = \log\left(\frac{P_0}{P}\right)^N$$

A least-squares fit is performed on the (LP, LV) designated pairs where LP is the independent variable and LV is the dependent variable. If the user selected *Yes* for the *Optimize Astakhov Exponent* prompt, a systematic search for the optimum value of N is conducted by recalculating the linear regression and selecting the value of N that gives the smallest standard error of the y-intercept. The exponent N is optimized to within 10^{-4} . If the optimum value for N is not found in this range, an exponent of 2 is used. The following are calculated:

- 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)

- 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):

$$V_i = \frac{Q_i V_{mol}}{22414}$$

where

V_{mol} = liquid molar volume conversion factor from the fluid property information

Limiting Micropore Volume (cm^3/g):

$$V_0 = \frac{Q_0 V_{mol}}{22414 \text{ cm}^3 \text{ STP}}$$

where

V_{mol} = liquid molar volume from the fluid property information

Error of Limiting Micropore Volume (cm^3/g):

$$V_{0, err} = W_0 (10^{YI_{err}} - 1.0)$$

Characteristic Energy (KJ/mol):

$$E = \frac{2.303(RT)}{\beta (2.303 \times S)^{1/N}}$$

Modal Equivalent Pore Diameter (\AA):

$$D_{mode} = 2 \left\{ \left[\frac{3N}{3N+1} \right]^{1/N} \times \left[\frac{10^3 \text{ nm}^3 / \text{\AA}^3}{\beta \cdot E_0} \right] \right\}^{1/3}$$

where

β = affinity coefficient of the analysis gas relative to the P_0 gas from the *Dubinin Adsorptive Options* window

Maximum Differential Pore Volume (cm³/g-Å):

This value is also known as *frequency of the mode*¹⁾.

$$\frac{dV}{dD_{mode}}Max = 0.5 \left(3N + 1 \right) W_0 \left[\frac{3N + 1}{3N} \right]^{1/3N} \left[\frac{\beta \cdot E_0}{\left(\left(10^3 nm^3 \right) / \text{\AA}^3 \right)} \right]^{1/3} \exp \left(- \left[\frac{3N + 1}{3N} \right] \right)$$

Mean Equivalent Pore Width (Å):

$$D_{mean} = 2 \times \frac{\left[\frac{\left(10^3 nm^3 \right) / \text{\AA}^3}{\beta \cdot E_0} \right]^{1/3}}{\Gamma \left(\frac{3N + 1}{3N} \right)}$$

Micropore surface area (m²/g):

$$SDA = 1000 \times 2.0 \times W_0 \times \left[\frac{E_0}{k} \right]^{1/3} \times \Gamma \left(\frac{3N + 1}{3N} \right)$$

Γ is calculated by a polynomial approximation over the domain $0 \leq x \leq 1$:

$$\Gamma(x + 1) = 1 + b_1 x + b_2 x^2 + b_3 x^3 + b_4 x^4 + b_5 x^5 + b_6 x^6 + b_7 x^7 + b_8 x^8 + \epsilon x |\epsilon x| \leq 3(10^{-7})$$

where

$$\begin{aligned} b_1 &= -0.57719\ 1652 \\ b_2 &= 0.98820\ 5891 \\ b_3 &= -0.89705\ 6937 \\ b_4 &= 0.91820\ 6857 \\ b_5 &= -0.75670\ 4078 \\ b_6 &= 0.48219\ 9394 \end{aligned}$$

¹⁾ Ross and Olivier, J.P., "On Physical Adsorption," J. Wiley and Sons, New York (1964).

$$\begin{aligned} b_7 &= -0.19352\ 7818 \\ b_8 &= 0.03586\ 8343 \end{aligned}$$

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):

$$\frac{dV}{dD_i} = 0.5 \times W_0 \times 3N \left(\frac{10^3 \text{ nm}^3 / \text{Å}^3}{\beta \cdot E_0} \right)^N \left(\frac{D_i}{2} \right)^{-(3N+1)} \times \exp \left\{ - \left(\frac{10^3 \text{ nm}^3 / \text{Å}^3}{\beta \cdot E_0} \right)^N \left(\frac{D_i}{2} \right)^{-3N} \right\}$$

DUBININ-RADUSHKEVICH

The Dubinin-Radushkevich¹⁾ 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 ($\text{cm}^3/\text{g STP}$)
Q_0	=	the micropore capacity ($\text{cm}^3/\text{g STP}$)
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 ($S \text{ cm}^3/\text{g STP}$)
- Y-intercept ($YI \text{ cm}^3/\text{g STP}$)
- Error of the slope ($S_{\text{err}} \text{ cm}^3/\text{g STP}$)

¹⁾ Dubinin, M., Carbon 21, 359 (1983); Dubinin, M., Progress in Surface and Membrane Science 9, 1, Academic Press, New York (1975); Dubinin, M. and Astakhov, V., Adv. Chem. Ser. 102, 69 (1971); Lamond, T. and Marsh, H., Carbon 1, 281, 293 (1964); Medek, J., Fuel 56, 131 (1977); Polanyi, M., Trans. Faraday Soc. 28, 316 (1932); Radushkevich, L., Zh. fiz. Kemi. 33, 2202 (1949); Stoeckli, H., et al, Carbon 27, 125 (1989).

- d. Error of the y-intercept (YI_{err} cm³/g STP)
- e. 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):

$$Q_{0, err} = Q_0 (10^{YI, err} - 1.0)$$

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

EQUATION OF STATE

The ideal gas law relates pressure, volume, temperature, and quantity of gas.

$$n = \frac{PV}{RT}$$

where

P = pressure

V = volume

R = a constant that depends on the units of n

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

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

T = temperature

The non-ideality correction is made by replacing P with $P(1 + aP)$, where a is the non-ideality factor. This gives

$$n = \frac{PV}{RT} (1 + aP)$$

The real gas equation of state uses

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

where $z(P, T)$ is the compressibility factor for the gas at the given pressure and temperature.

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.

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

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

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

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

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

¹⁾ Savitzky, A. and Golay, M.J.E., Anal. Chem. 36, 1627 (1964).

F-RATIO METHOD

The *f*-Ratio is the quantity adsorbed divided by the quantity adsorbed in a reference isotherm at the same pressure.

$$f_i = \frac{Q_i}{Q_{ref} P_i}$$

The reference quantity adsorbed is found by spline interpolation of the reference isotherm.

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

$$\log Q = \log Q_m C + \frac{1}{m} \log P$$

HEAT OF ADSORPTION

The adsorption isostere is represented by

$$\ln\left(\frac{P}{P_0}\right) = \frac{q_i}{RT} + C$$

where

q_i	=	isosteric heat of adsorption
C	=	unknown constant
R	=	gas constant
T	=	analysis temperature

The isosteric heat of adsorption, q_i , is determined by finding the slope of $\ln (P/P_0)$ as a function of $1/RT$ for a set of isotherms measured at different temperatures.

HORVATH-KAWAZOE

A relative pressure lower limit is determined such that $L-d_0$ never equals zero. All pressure points less than this limit are discarded. For each collected relative pressure point, values of L are chosen in an iterative manner, and the relative pressure (P/P_0) determined by solving one of the following equations:

- Slit Pore Geometry (original Horvath-Kawazoe)
- Cylinder Pore Geometry (Saito / Foley)
- Sphere Pore Geometry (Cheng / Yang)

SLIT PORE GEOMETRY (ORIGINAL HORVATH-KAWAZOE)

When using the original Horvath-Kawazoe¹⁾ 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.

$$\ln \frac{P}{P_0} = \frac{K}{RT} \frac{IP \times 10^{32}}{\sigma^4 L - 2d_0^3} \frac{\sigma^4}{3L - d_0^3} - \frac{\sigma^{10}}{9L - d_0^9} - \frac{\sigma^4}{3d_0^3} + \frac{\sigma^{10}}{9d_0^9}$$

where

10^{32} = the number of Å⁴ that are equal to 1 cm⁴

α = gas solid nuclear separation at zero interaction energy (Å), $\frac{Z_S + Z_A}{2}$

d_0 = $\frac{D_A + D_S}{2}$

D_A = molecular diameter (Å) from the *Horvath-Kawazoe Physical Properties* window

D_S = diameter of sample atom (Å) from the *Horvath-Kawazoe Physical Properties* window

IP = interaction parameter (erg-cm⁴) from the *Horvath-Kawazoe Report Options* window

K = Avogadro Constant (N_A)

L = pore width (nucleus to nucleus) (Å)

K = Avogadro Constant (N_A)

P = equilibrium pressure

¹⁾ Horvath, G. and Kawazoe, K., J. Chem. Eng. Japan 16(6), 470 (1983).

- P_0 = saturation pressure
 R = gas constant (8.31441×10^7 erg/mol K)
 T = analysis bath temperature (K), from an entered or calculated value on the P_0 and *Temperature Options* window

where:

- Z_s = sample equilibrium diameter at zero interaction energy (Å) from the *Horvath-Kawazoe Physical Properties* window
 Z_A = zero interaction energy diameter from the *Horvath-Kawazoe Physical Properties* window

CYLINDER PORE GEOMETRY (SAITO/FOLEY)

When using the Saito/Foley¹⁾ method, the following equation is solved for each value of P . The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{P_0}\right) = \frac{3\pi K}{4RT} \times \frac{IP \times 10^{32}}{d_0^4} \times \sum_{k=0}^{\infty} \left[\frac{1}{k+1} \left(1 - \frac{d_0}{r_p}\right)^{2k} \times \left\{ \frac{21}{32} \alpha_k \left(\frac{d_0}{r_p}\right)^{10} - \beta_k \left(\frac{d_0}{r_p}\right)^4 \right\} \right]$$

where

$$10^{32} = \text{the number of } \text{\AA}^4 \text{ that are equal to } 1 \text{ cm}^4$$

$$\beta_k = \left(\frac{-1.5-k}{k}\right)^2 \beta_{k-1}, \beta_0 = 1.0$$

$$\alpha_k = \left(\frac{-4.5-k}{k}\right)^2 \alpha_{k-1}, \alpha_0 = 1.0$$

$$d_0 = \frac{D_A + D_S}{2}$$

where

$$D_A = \text{molecular diameter (}\text{\AA}\text{) from the } \textit{Horvath-Kawazoe Physical Properties} \text{ window}$$

$$D_S = \text{diameter of sample atom (}\text{\AA}\text{) from the } \textit{Horvath-Kawazoe Physical Properties} \text{ window}$$

$$IP = \text{interaction parameter (} 10^{-43} \text{ erg-cm}^4 \text{) from the } \textit{Horvath-Kawazoe Report Options} \text{ window}$$

$$K = \text{Avogadro Constant (} N_A \text{)}$$

¹⁾ Saito, A. and Foley, H. C., *AIChE Journal* 37(3), 429 (1991).

L	=	pore width (nucleus to nucleus) (Å)
P	=	equilibrium pressure
P _o	=	saturation pressure
R	=	gas constant (8.31441 × 10 ⁷ erg/mol K)
r _p	=	radius of the cylindrical pore, $\frac{L}{2}$
T	=	analysis bath temperature (K), from an entered or calculated value on the <i>Po and Temperature Options</i> window

SPHERE PORE GEOMETRY (CHENG/YANG)

When using the Cheng / Yang¹⁾ method, the following equation is solved for each value of *P*. The value of *L* is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{P_0}\right) = \frac{6N_1\epsilon_{12}^* + N_2\epsilon_{22}^* L^3 \times 10^{32}}{RTL - d_0^3} \left[-\left(\frac{d_0}{L}\right)^6 \left(\frac{T_1}{12} + \frac{T_2}{8}\right) + \left(\frac{d_0}{L}\right)^{12} \left(\frac{T_3}{90} + \frac{T_4}{80}\right) \right]$$

where

$$10^{32} = \text{the number of cm}^4 \text{ that are equal to } \text{\AA}^4$$

$$\epsilon_{12}^* = \frac{\text{\AA}_S}{4d_s^6}, \text{ where } \text{\AA}_S = \frac{6(mc^2)a_S a_A}{\frac{a_S}{X_S} + \frac{a_A}{X_A}}$$

$$\epsilon_{22}^* = \frac{A_A}{4D_A^6}, \text{ where } \text{\AA}_A = \frac{3(mc^2)(a_A)(X_A)}{2}$$

$$d_0 = \frac{D_A + D_S}{2}$$

where

$$D_A = \text{molecular diameter (\AA) from the Horvath-Kawazoe Physical Properties window}$$

$$D_S = \text{diameter of sample atom (\AA) from the Horvath-Kawazoe Physical Properties window}$$

$$L = \text{pore width (nucleus to nucleus) (\AA)}$$

$$N_1 = 4\pi L_2 N_s, \text{ where } N_s = \text{number of sample atoms/cm}^2 \text{ at monolayer}$$

¹⁾ Cheng, Linda S. and Yang, Ralph T., Chemical Engineering Science 49(16), 2599-2609 (1994).

N_2	=	$4\pi (L-d_0)^2 N_A$, where N_S = number of gas molecules/cm ²
P	=	equilibrium pressure
P_0	=	saturation pressure
R	=	gas constant (8.31441×10^7 erg/mol K)
T	=	analysis bath temperature (K), from an entered or calculated value on the P_0 and Temperature Options window

$$T_1 = \frac{1}{(1-S)^3} - \frac{1}{(1+S)^3}$$

$$T_2 = \frac{1}{(1+S)^2} - \frac{1}{(1-S)^2}$$

$$T_3 = \frac{1}{(1-S)^9} - \frac{1}{(1+S)^9}$$

$$T_4 = \frac{1}{(1+S)^8} - \frac{1}{(1-S)^8}$$

where

$$S = \frac{L-d_0}{L}$$

CHENG/YANG CORRECTION

This factor corrects for the nonlinearity of the isotherm. It adds an additional term to the equations for the different geometries:

$$\ln\left(\frac{P}{P_0}\right) = G(L) - \left[1 - \frac{1}{\theta} \ln\left(\frac{1}{1-\theta}\right)\right]$$

where

$G(L)$	=	one of the Horvath-Kawazoe equations given above
θ	=	degree of void filling; θ is estimated by first computing the monolayer capacity (Q_m) with the Langmuir equation over the range of data points from relative pressure 0.02 to 0.2 or the maximum relative pressure included in the Horvath-Kawazoe analysis. θ is computed as the quantity adsorbed over Q_m .

INTERACTION PARAMETER

The interaction parameter (IP) results from the following calculations:

The Kirkwood-Muller dispersion coefficients

$$A_S = \frac{6mc^2 \alpha_S \alpha_A}{\frac{\alpha_S}{X_S} + \frac{\alpha_A}{X_A}}$$

$$A_A = \frac{3mc^2 \alpha_A x_A}{2}$$

where

α_A	=	polarizability of gas molecule (cm ³)
α_S	=	polarizability of sample atoms (cm ³)
mc^2	=	kinetic energy of electron (0.8183 × 10 ⁻⁶ erg)
X_A	=	diamagnetic susceptibility of gas molecule (cm ³)

$$IP = (N_A A_A) + (N_S A_S)$$

N_A = number of gas molecules/cm² at monolayer from the *Horvath-Kawazoe Physical Properties* window

N_S = number of sample atoms/cm² from the *Horvath-Kawazoe Physical Properties* window

X_S = diamagnetic susceptibility of sample atom (cm³)

See [Interaction Parameter Components Table on page B - 33](#) for recommended values.

ADDITIONAL CALCULATIONS

Based on the previous calculations, the following can be calculated:

Adjusted Pore Width (Å):

(Shell to Shell)

$$AL_i = L_i - D_S$$

Cumulative Pore Volume (cm³/g):

$$V_{cum,i} = \frac{Q_i V_{mol}}{22414 \text{ cm}^3 \text{ STP}}$$

where

V_{mol} = liquid molar volume from the fluid property information

dV/dD Pore Volume (cm³/g-Å):

$$\frac{dV}{dD_i} = \frac{V_{cum,i} - V_{cum,i-1}}{AL_i - AL_{i-1}}$$

Median Pore Width (Å):

$$V_{half} = \frac{V_{cum,n}}{2}$$

$$D_{med} = \exp \left(\ln D_i + (\ln D_g - \ln D_i) \frac{\ln V_{half} - \ln V_l}{\ln V_g - \ln V_l} \right)$$

where

D_l	=	pore width (L_i) that corresponds to V_l
D_g	=	pore width (L_i) that corresponds to V_g
$V_{cum,n}$	=	total cumulative pore volume ($V_{cum,i}$) for points designated for Horvath-Kawazoe calculations
V_g	=	cumulative pore volume ($V_{cum,i}$) for first point greater than V_{half}
V_{half}	=	50% of total cumulative pore volume
V_l	=	cumulative pore volume ($V_{cum,i}$) for first point less than V_{half}

Interaction Parameter Components Table

Gas	Bath Temperature (K)	Sample Type	Interaction Parameter Calculated Value *
Argon	87.3	Carbon (Ross/Olivier value)	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

* The interaction parameter is entered in the *Horvath-Kawazoe Report Options* window in the following field:

Interaction parameter: (calculated value) × 10⁻⁴³ erg-cm⁴

The following values were used to calculate the values in the previous table.

Carbon-Graphite

$$\begin{aligned}
 D_S &= 3.40 \\
 N_S &= 3.845 \times 10^{15} \\
 X_S &= 1.05 \times 10^{-29} \text{ (Ross/Olivier)} \\
 &\quad 13.5 \times 10^{-29} \\
 &\quad \text{(Horvath/Kawazoe, implicit)} \\
 \alpha_s &= 1.02 \times 10^{-24}
 \end{aligned}$$

Zeolite

$$\begin{aligned}
 D_S &= 3.04 \\
 N_S &= 3.75 \times 10^{15} \\
 X_S &= 1.94 \times 10^{-29} \\
 \alpha_s &= 0.85 \times 10^{-24}
 \end{aligned}$$

Nitrogen

$$\begin{aligned}
 D_A &= 3.00 \\
 N_A &= 6.71 \times 10^{14} \\
 X_A &= 3.6 \times 10^{-29}
 \end{aligned}$$

Argon

$$\begin{aligned}
 D_A &= 2.95 \\
 N_A &= 7.608 \times 10^{14} \\
 X_A &= 3.22 \times 10^{-29}
 \end{aligned}$$

$$\alpha_A = 1.76 \times 10^{-24}$$

$$\alpha_A = 1.63 \times 10^{-24}$$

Carbon Dioxide

$$D_A = 3.23$$

$$N_A = 4.567 \times 10^{14} \text{ (25 °C)}$$

$$5.45 \times 10^{14} \text{ (0 °C)}$$

$$7.697 \times 10^{14} \text{ (-78 °C)}$$

$$X_A = 5.0 \times 10^{-29}$$

$$\alpha_A = 2.7 \times 10^{-24}$$

D_A values are from van der Waal's constant.

N_A values are from liquid densities.

x and a values are derived from data found in Ross and Olivier¹⁾.

The physical parameters referenced in Saito/Foley are:

Aluminophosphate

$$D_S = 2.60$$

$$N_S = 1.48 \times 10^{15}$$

$$X_S = 1.3 \times 10^{-29}$$

$$\alpha_s = 2.5 \times 10^{-24}$$

Aluminosilicate

$$D_S = 2.76$$

$$N_S = 1.31 \times 10^{15}$$

$$X_S = 1.3 \times 10^{-29}$$

$$\alpha_s = 2.5 \times 10^{-24}$$

¹⁾ Ross and Olivier, J.P., "On Physical Adsorption," J. Wiley and Sons, New York (1964)

LANGMUIR SURFACE AREA

For each point designated for surface area calculations, the Langmuir¹⁾ transformation is calculated as:

$$L = \frac{P_{rel}}{N_{ads}}$$

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

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

- Slope (S g/cm^3 STP)
- Y-intercept (Y_{int} g/cm^3 STP)
- Error of the slope (S_{err} g/cm^3 STP)
- Error of the y-intercept (YI_{err} g/cm^3 STP)
- Correlation coefficient

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

Langmuir Surface Area (m^2/g):

$$SA_{Lan} = \frac{CSA \times N_A}{\left(22414 \text{ cm}^3 \text{ STP}\right) \left(\frac{10^{18} \text{ nm}^2}{\text{m}^2}\right) S}$$

where

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

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

$$Q_m = \frac{1}{S}$$

Langmuir b Value:

$$b = Y_{int} V_m$$

¹⁾ Langmuir, I., J. Am. Chem. Soc. 38, 2267 (1916); J. Am. Chem. Soc. 40, 1361 (1918); Phys. Rev 8, 149 (1916).

Error of the Langmuir Surface Area (m²/g):

$$LAN_{err} = \frac{SA_{Lan} S_{err}}{S}$$

MP-METHOD

With the (t_i, Q_i) data pairs¹⁾, the Akima semi-spline interpolation method is used to interpolate quantity adsorbed values based on thickness values that are evenly spaced 0.2 angstrom apart starting at the first outlier point. Outliers are defined as those points that have the maximum instantaneous slope within an iteratively shrinking subset of all points. The remaining pore surface area calculation result is the slope of the line defined by two consecutive interpolated points. The slopes of each pair of consecutive points from the origin to the last point must be monotonically decreasing and non-negative. With the interpolated points set the following can be calculated:

Average pore hydraulic radius (Å):

$$R_i = \frac{t_i + t_{i-1}}{2}$$

Remaining pore surface area for the ith point (m²/g):

$$S_i = \frac{Q_i - Q_{i-1}}{t_i - t_{i-1}} \frac{V_{mol}}{22414 \text{ cm}^3 \text{ STP}} \times 10^4$$

where

10^4 = unit conversions

V_{mol} = liquid molar volume from the fluid property information

Incremental pore surface area occluded for the ith point (m²/g):

$$S_{inc,i} = S_{i-1} - S_i$$

Cumulative pore surface area occluded for the ith point (m²/g):

$$S_{cum_i} = S_{inc,i} + S_{inc,i-1} + \dots + S_{inc,i}$$

dA/dR pore surface area for the ith point (m²/g-Å):

$$\frac{dA}{dR_i} = \frac{S_{inc_i}}{t_i - t_{i-1}}$$

Incremental pore volume occluded for the ith point (cm³/g):

¹⁾ Mikhail, R., Brunauer, S. and Bodor, E., J. Colloid and Interface Sci. 24, 45 (1968).

$$V_{inc,i} = S_{inc,i} R_i \times 10^{-4}$$

Cumulative pore volume occluded for the ith point (cm³/g):

$$V_{cum,i} = V_{inc,i-1} + V + \dots + V_{inc,i}$$

dV/dR pore volume for the ith point (cm³/g-Å):

$$\frac{dV}{dR_i} = \frac{V_{inc,i}}{t_i - t_{i-1}}$$

RELATIVE PRESSURE

Relative pressure is defined as:

$$P_{rel,i} = \frac{P_i}{P_{0,i}}$$

$P_{0,i}$ depends on the method selected in the P_0 and Temperature options

If P_0 was measured for each point, $P_{0,i}$ is the saturation pressure measured when P_i was equilibrated.

If P_0 was measured at intervals, $P_{0,i}$ is found by linear interpolation.

$$P_{0,i} = P_{0,i-1} + \left(P_{0,i+1} - P_{0,i-1} \right) \frac{t_i - t_{i-1}}{t_{i+1} - t_{i-1}}$$

The last measured P_0 is only for points taken after the last P_0 measurement.

If P_0 was calculated from measured Psat or entered temperature the calculated P_0 is used for all data points.

If P_0 was entered, the entered P_0 is used for all data points.

SATURATION PRESSURE

Saturation pressure (P_0) is selected on the P_0 and Temperature Options window. It may be entered or measured in the P_0 tube. The analyzer uses the following methods to get P_0 :

1. P_0 is measured in the P_0 tube for each isotherm point. This method can be used only if the instrument has a dedicated P_0 transducer.
2. P_0 is measured at a specified time interval using the 1000 mmHg manifold transducer.
3. The saturation pressure of a gas is measured in the P_0 tube for prior to data collection. The bath temperature is found by looking up the temperature for the measured saturation pressure in the Psat vs. T table. P_0 is found by looking up the calculated bath temperature in the adsorptive properties Psat vs. T table.
4. P_0 is found by looking up the entered bath temperature in the adsorptive properties Psat vs. T table.
5. P_0 is entered.

SPC REPORT VARIABLES

REGRESSION CHART VARIABLES

The line of best fit for the Regression Chart is calculated by the usual least squares method. ^{1)} If there is only a single point or all N points have the same x -value, there can be no line of best fit in the standard form.

$$\bar{x} = \frac{\sum x_i}{N}$$

$$\bar{y} = \frac{\sum y_i}{N}$$

$$\text{Slope} = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{\sum (x_i - \bar{x})^2}$$

$$\text{Intercept} = \bar{y} - \text{Slope} \cdot \bar{x}$$

The coefficient of correlation for this line is also calculated in the usual way. ^{2)}

$$\sigma_x = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N}}$$

$$\sigma_y = \sqrt{\frac{\sum (y_i - \bar{y})^2}{N}}$$

$$\text{Cov}(x, y) = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{N}$$

$$\text{Correlation Coeff} = \frac{\text{Cov}(x, y)}{\sigma_x \sigma_y}$$

^{1)} BASIC Scientific Subroutines Vol II, by F.R. Ruckdeschel, Copyright 1981 BYTE Publications/McGraw Hill, p. 16.

^{2)} Mathematical Handbook for Scientists and Engineers, G.A. Korn and T.M. Korn, McGraw Hill, Sec. 18.4. (1968)

CONTROL CHART VARIABLES

$$\text{Mean} = \frac{\sum y_i}{N}$$

$$\text{Standard Deviation} = \sqrt{\frac{\sum (y_i - \text{Mean})^2}{N - 1}}$$

$$\text{C. V.} = \frac{\text{StdDev}}{\text{Mean}}$$

$$+ n \sigma = \text{Mean} + n \cdot \text{Standard Deviation}$$

$$- n \sigma = \text{Mean} - n \cdot \text{Standard Deviation}$$

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:

- Slope (S cm³/g-Å STP)
- Y-intercept (Y_{int} cm³/g STP)
- Error of the slope (S_{err} cm³/g-Å 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:

External Surface Area (m²/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 screen

V_{mol} = liquid molar volume, from the fluid property information

Micropore Surface Area (m²/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 (cm³ liquid/g):

$$\frac{Y_{int} V_{mol}}{22414 \text{ cm}^3 \text{ STP}}$$

TEMKIN ISOTHERM

The Temkin isotherm has the form

$$\frac{Q}{Q_m} = \frac{RT}{q_0 \alpha} \ln(A_0 P)$$

where

A_0	=	adjustable constant
α	=	adjustable constant
P	=	equilibrium pressure measured by gauge at temp T_{amb}
q_0	=	the differential heat of adsorption at zero surface coverage
Q	=	quantity of gas adsorbed
Q_m	=	quantity of gas in a monolayer
R	=	molar gas constant $8.31441 \times 10^{-3} \frac{kJ}{molK}$
T	=	bath temperature

In terms of quantity adsorbed

$$Q = \frac{RTQ_m}{q_0 \alpha} \left[\ln A_0 + \ln \left(\frac{P}{P_0} \right) \right]$$

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 $\ln A_0 \frac{RTQ_m}{q_0 \alpha}$.

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 (P/C/P) is greater than or equal to 0.99.

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

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

$$F = \frac{1}{\alpha Y^2 + \beta Y + \mu}$$

$$P = \left(1 - F \left(1 - \sqrt{\frac{T_{bath}}{T_{amb}}} \right) \right)$$

where

α	=	Weber's coefficient, 0.033
β	=	Weber's coefficient, 0.245
F, Y, 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 <i>Adsorptive Properties</i> window
P	=	equilibrated collected pressure measured by gauge at temp T_{amb}
SD	=	inside diameter of sample tube (mm), from the <i>Report Options</i> window
T	=	average temperature $\frac{T_{amb} + T}{2}$
T_{amb}	=	room temperature (298 K)
T_{bath}	=	analysis bath temperature (K), from the P_0 and <i>Temperature Options</i> window

THICKNESS CURVE

For each point designated, the following parameters are used in thickness curve calculations:

C_1	=	parameter #1
C_2	=	parameter #2
C_3	=	parameter #3
$P_{rel, i}$	=	relative pressure for the i^{th} point (mmHg)
t_i	=	thickness for i^{th} point

REFERENCE

Interpolated from table.

KRUK-JARONIEC-SAYARI

$$t = \left(\frac{C_1}{C_2 = \log(P_{rel, i})} \right)^{C_3}$$

HALSEY

$$t_i = C_1 \left[\frac{C_2}{\ln(P_{rel, i})} \right]^{C_3} \quad \text{Halsey}^1)$$

HARKINS AND JURA

$$t_i = \left[\frac{C_1}{C_2 - \log(P_{rel, i})} \right]^{C_3} \quad \text{Harkins and Jura}^2)$$

¹⁾ Halsey, G., J. Chem. Phys. 16, 931-937 (1948).

²⁾ Harkins, W.D. and Jura, G., J. Chem. Phys. 11, 431 (1943).

BROEKOFF-DE BOER

$$\log(P_{rel,i}) = \frac{C_1}{t_{i,i}^2} + C_2 \exp(c_3 t_i)$$

CARBON BLACK STSA

$$t_i = C_1(P_{rel,i})^2 + C_2(P_{rel,i}) + C_3$$

C DFT MODELS

Theories are developed by scientists in an attempt to explain a class of observed behavior. In the experimental physical sciences, theories are often expressed in terms of a model that can be visualized and described mathematically. Early models of physical adsorption were quite simple, both conceptually and mathematically, for very practical reasons — hand computations were required. Today we can explore complex models that describe adsorption systems on the atomic scale of size and sub-picosecond time frame. This is not because scientists are smarter, but because of available tools. The DFT models are created by classical approaches to adsorption as well as models based on modern statistical thermodynamics.

MODELS BASED ON STATISTICAL THERMODYNAMICS

Included in this group are methods that model the adsorption system in terms of forces acting between individual molecules.

THEORETICAL BACKGROUND

Traditional adsorption theories attempt to describe experimental adsorption isotherms with an isotherm equation containing a small number of parameters. At a minimum, these parameters include the extent of the surface, such as the monolayer capacity (V_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:

$$U(s) = 4\epsilon \left(\frac{\sigma}{s} \right)^{12} - \left(\frac{\sigma}{s} \right)^6$$

where

ϵ = a characteristic energy of the adsorptive,
 σ = the diameter of the adsorptive molecule, and
 s = the separation distance.

MOLECULAR SIMULATION METHODS

Two simulation techniques are commonly used to determine the distribution of gas molecules in a system in equilibrium: the molecular dynamics method and the Monte Carlo method. Both of these are used as reference methods because their results are considered exact.

MOLECULAR DYNAMICS METHOD

In the molecular dynamics method, the position and velocity of individual gas particles are calculated over time at very short intervals. This method takes into account both the forces acting between the gas particles themselves and those acting between the gas particles and the atoms of the simulated surface. As the simulated particles collide with each other and with the surface, the average concentration of particles in the space near the surface is calculated; this calculation yields the amount of gas adsorbed.

This method can be thought of as a way to determine the chronological record of the movement of each particle in the system using time steps of 10-14 seconds. Although the mathematics are simple, the number of calculations required for a system of even a few hundred particles is astronomical and challenges even the fastest computers.

Monte Carlo Method

In the Monte Carlo method, determination of the system equilibrium distribution begins with an assumption (which may be only approximate) about the initial configuration of particles in the system. The system is “equilibrated” through a process of randomly selecting one particle and conditionally moving it a random distance in a random direction.

If the move results in a configuration of *lower total energy*, then the move is completed and another particle is randomly selected to be moved.

If the move results in a configuration of *higher energy*, a probability for that event is calculated, and a random number between zero and one is generated. If the generated number is smaller than the probability of the event, then the move is accepted; otherwise, another particle is selected and the process is repeated. This process continues until the average total energy of the system no longer decreases; at this point, average configuration data are accumulated to yield the mean density distribution of particles in the system.

Monte Carlo simulations require considerably less computation time than molecular dynamic simulations and can yield the same results; however, neither method provides a really practical way to calculate complete isotherms.

Density Functional Formulation

Density functional theory offers a practical alternative to both molecular dynamic and Monte Carlo simulations. When compared to reference methods based on molecular simulation, this theory provides an accurate method of describing inhomogeneous systems yet requires fewer calculations. Because the density functional theory provides accuracy and a reduced number of calculations, it is the basis embodied in the DFT models.

The system being modeled consists of a single pore represented by two parallel walls separated by a distance H . The pore is open and immersed in a single component fluid (adsorptive) at a fixed temperature and pressure. Under such conditions, the fluid responds to the walls and reaches an equilibrium distribution. In this condition (by the definition of equilibrium), the chemical potential at every point equals the chemical potential of the bulk fluid. The bulk fluid is a homogenous system of constant density; its chemical potential¹⁾ 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.

¹⁾ Chemical potential may be thought of as the energy change felt by a probe particle when it is inserted into the system from a reference point outside the system. It can also be defined as the partial derivative of the grand potential energy with respect to density (or concentration).

As noted previously, at equilibrium, the whole system has a minimum (Helmholtz) free energy, known thermodynamically as the grand potential energy (GPE). Density functional theory describes the thermodynamic grand potential as a functional of the single-particle density distribution; therefore, calculating the density profile that minimizes the GPE yields the equilibrium density profile. The calculation method requires the solution of a system of complex integral equations that are implicit functions of the density vector. Since analytic solutions are not possible, the problem must be solved using iterative numerical methods. Although calculation using these methods still requires supercomputing speed, the calculation of many isotherm pressure points for a wide range of pore sizes is a feasible task. The complete details of the theory and the mathematics can be found in the papers listed under [DFT Model References on page C - 16](#).

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

Figure 1 shows the density profile for argon at a carbon surface as calculated by density functional theory for a temperature of 87.3 K and a relative pressure of about 0.5.

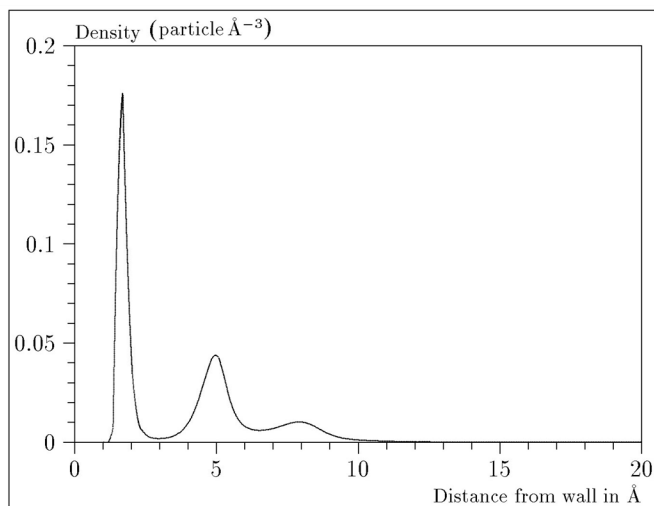


Figure 1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

This figure represents a cross-section of the region near the surface. Note the layerwise distribution of adsorbate; the first monolayer is sharply defined and a third layer can be distinguished. The area under the profile curve represents the amount adsorbed per unit area at this pressure. The positions of the maxima are separated by a distance determined by the size of the adsorptive atom.

Given the density profile, the amount adsorbed at the stated pressure can be easily calculated as the integral over the profile. Repeating this calculation over a range of pressures yields the adsorption isotherm for the model. If the value of H is very large, the isotherm obtained corresponds to that of an external, or *free*, surface. If H is smaller, a range of pressures is reached where two minima exist for the grand potential, showing the presence of two metastable phases having different density distributions but the same chemical potential. The phase with the lower GPE is the stable one. As the pressure is increased, a point is reached where the other phase becomes the stable one. This phase transition reflects condensation of adsorbate in the pore; the pressure at which it occurs is called the *critical pore-filling pressure*. This pressure is analogous to the condensation pressure predicted by the Kelvin equation in the classical model of pore filling.

Figure 2 shows how the profiles change with pressure for a model pore with $H = 40$ angstroms. The inset shows the density profiles for the corresponding points of the isotherm.

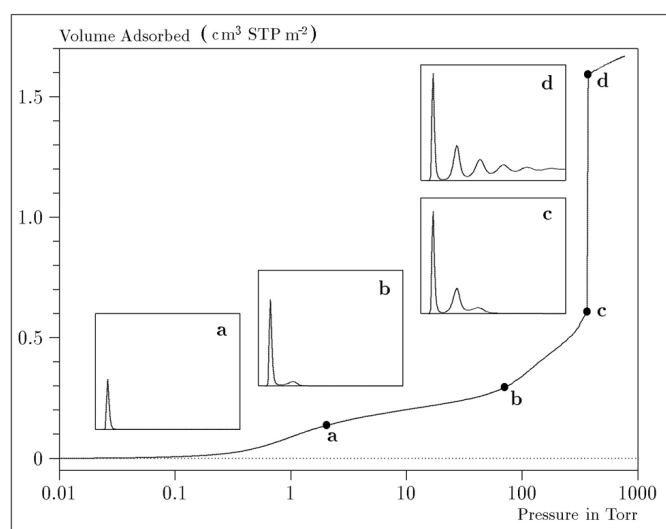


Figure 2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

The profiles show the density distribution from one wall to the center of the slit; the other half of the distribution is a mirror image of the profile shown.

As the pressure is first increased from zero, almost all the adsorbed atoms occupy a position close to the surface.

- Inset **a** shows the profile corresponding to point **a** on the isotherm where the surface is about half covered.
- At point **b**, the first layer is so full that it is more favorable for atoms to start a new layer.
- At point **c**, a third layer is forming. Point **c**, for this size slit, is the critical pore-filling pressure. In inset **c**, the profile shows the density decreasing to near zero (actually the bulk gas density) at 4 or 5 molecular diameters from the surface.

- Inset *d* shows the profile converging on a density similar to that of bulk liquid argon in the center of the pore, indicating a phase transition.

Note that the adsorption isotherms for pores larger than the one shown in the previous graph is identical up to point *c*. The lower branch of the isotherm simply continues to a higher pressure for larger pores. This trend is illustrated in the Figure 3, where isotherms for some larger size pores are shown. It is clear that pore size is uniquely characterized by a corresponding critical pore-filling pressure. At large pore sizes, density functional theory produces results for the critical filling pressures that are in good agreement with those produced by the Kelvin equation.

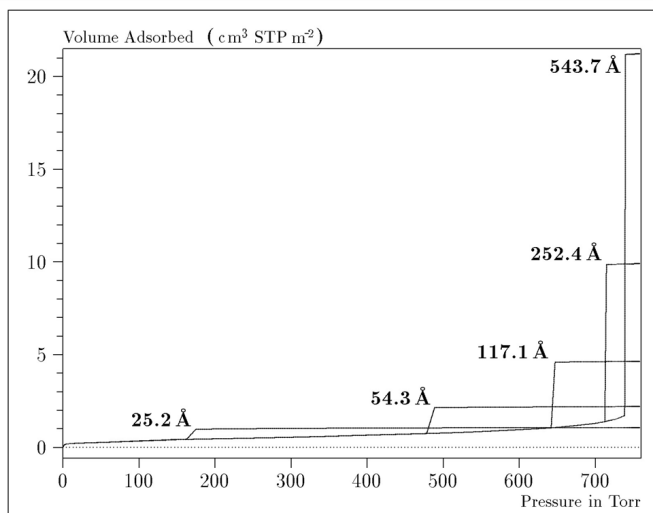


Figure 3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

Figure 4 shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.

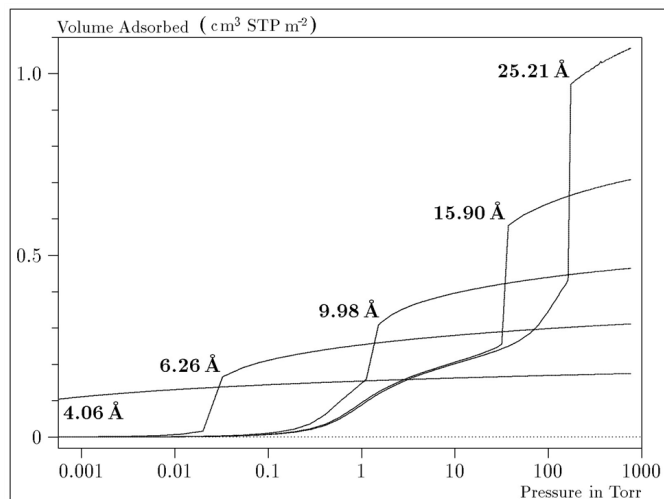


Figure 4. Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

Pores of 4 Å width, barely larger than the argon atom (3.38 Å), fill at pressures below 1 millitorr. Pores below 15 Å fill before a monolayer is completed on the surface of the larger pores. In the micropore size range, the pore volume fills more gradually with pressure and the total shape of the isotherm is important in characterizing the pore size.

Models Included

Non-Local Density Functional Theory with Density-Independent Weights

N2 - DFT Model

AR - DFT Model

Geometry:	Slit
Substrate:	Carbon (graphite)
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Using the methods of non-local density functional theory, two sets of isotherms have been calculated to serve as kernel functions for the characterization of porous solids from adsorption data. The model isotherms are stored in binary format files. These models assume a *slit-like pore geometry*. The pore size range from 4.0 to 4000 Å is covered in 91 classes in a geometric progression. The class intervals are rounded to the nearest 0.02 molecular diameters. A model for the free or external surface is included to account for unfilled pores. Each of the 92 model isotherms has been calculated at 181 pressure points from near 1×10^{-6} to near 1.00 relative pressure.

These models are identical to those supplied with the original DOS version of DFT software. Some slight difference from the DOS results may be noted when they are applied to the same data due to improvements in the deconvolution algorithm and better regularization of the current software.

Non-Local Density Functional Theory with Density-Dependent Weights

N2 - Modified Density Functional

Geometry:	Free surface
Substrate:	Surface energy
Method:	Nitrogen at 77K

Using the modified Tarazona prescription described by Olivier (see [DFT Model References on page C - 16](#) [items 3 and 4]), model isotherms were calculated for a wide range of adsorptive energies to a relative pressure of 0.6. The model makes no provision for pore filling in the micropore region. If the sample solid contains small mesopores, the isotherm data should be truncated (using the *Select Data Points* window) to a suitably low relative pressure to avoid trying to fit this region; mesopore filling reports as a large area of low energy in the calculated distribution of adsorptive potential.

The surface energy is reported in terms of the effective Lennard-Jones interaction parameter, ie, for the adsorptive / adsorbent pair divided by Boltzmann constant. The units are therefore Kelvin.

N2 - Cylindrical Pores - Oxide Surface**AR - Cylindrical Pores - Oxide Surface**

Geometry:	Cylinder
Substrate:	Oxide
Category:	Porosity
Method:	Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using a combination of statistical mechanical calculations and experimental observations for macroporous silicas and MCM-41 mesoporous silicas as well as zeolites. The pore-filling pressures were determined as a function of the pore size from adsorption isotherms on MCM-41 materials characterized by X-ray and other techniques. The variation of the pore fluid density with pressure and pore size has been accounted for by density functional theory calculations. The N2 model reports pore sizes ranging from 3.8 to 387 Å and the AR model from 3.8 to over 500 angstroms.

References: M. Jaroniec, M. Kruk, J.P. Olivier, and S. Koch, "A New Method for the Accurate Pore Size Analysis of MCM-41 and Other Silica-Based Mesoporous Materials," Proceedings of COPS-V, Heidelberg, Germany (1999).

N2 – Cylindrical Pores – Pillared Clay Surface (Montmorillonite)

Geometry:	Cylinder
Substrate:	Crystalline Silicate
Category:	Porosity
Method:	Nitrogen at 77K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFT) calculations and experimental isotherms for reference samples of 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 angstroms.

References: Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V*, Unger, et al, Eds, Elsevier, Amsterdam, 2000.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of a Pillared Interlayered Clay (PILC) from a Hybrid Density Functional Theory (DFT) Method," *The Journal of Physical Chemistry B*; 2001, 105(3),

623-629.

M. L. Occelli, J. P. Olivier, J. A. Perdigon-Melon, and A. Auroux, "Surface Area, Pore Volume Distribution, and Acidity in Mesoporous Expanded Clay Catalysts from Hybrid Density Functional Theory (DFT) and Adsorption Microcalorimetry Methods," *Langmuir* 2002, 18, 9816-9823.9b.

James P. Olivier, "The Importance of Surface Heterogeneity in Developing Characterization Methods." *6th International Symposium on the Characterization of Porous Solids*, Studies in Surface Science and Catalysis 144, Elsevier, 2002.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of Pillared Rectorite Catalysts from a Hybrid Density Functional Theory Method," *Microporous and Mesoporous Materials* 2003, 57, 291-296.

C02 - DFT Model

Geometry: Slit
Substrate: Carbon
Category: Porosity
Method: Carbon dioxide at 273 K

Model isotherms were calculated using the non-local prescription of Tarazona, employing molecular parameters derived from the known bulk properties of carbon dioxide.

AR - Modified Density Functional Model

Geometry: Free surface
Substrate: Any
Category: Surface energy
Method: Argon at 87K

This model was produced in the same manner as the N2 Modified Density Functional model listed earlier, except applicable to argon adsorbed at 87.3 K.

N2 - Tarazona NLDFT, Esf = 30.0K

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a cylindrical pore geometry. The wall potential used is $k = 30$ K, typical for a silica or alumina surface.

This model file is particularly useful for sizing zeolites or zeolite containing materials that have substantial micropore volume. The reported pore size range is 3.8 to 387 angstroms.

- References:** P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Slit Pores by NLDFT

Ar - Carbon Slit Pores by NLDFT

- Geometry:** Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a slit-like pore geometry. These models are slightly different from N2-DFT and Ar-DFT models that were calculated using NLDFT with density independent weighting functions.

The reported pore size range is from 3.5 to 1000 angstroms.

- References:** P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Finite Pores, As=6, 2D-NLDFT

Ar - Carbon Finite Pores, As=6, 2D-NLDFT

- Geometry:** Finite Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions assuming 2D model of finite slit pores having a diameter-to-width aspect ratio of 6.

This model is particularly useful for microporous carbon materials. The reported pore size range is from 3.5 to 250 angstroms

- References:** Jacek Jagiello and James P. Olivier. "A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure

analysis.,” The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N₂ - Carbon Finite Pores, As=12, 2D-NLDFT**Ar - Carbon Finite Pores, As=12, 2D-NLDFT**

Geometry:	Finite Slit
Substrate:	Carbon
Category:	Porosity
Method:	Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the same methods and assumptions that were used in the model above except in this model, the aspect ratio is equal to 12.

These two finite pore models may be used as a research tool in conjunction with independent analytical techniques such as high-resolution transmission electron microscopy (HRTEM) and / or X-ray diffraction (XRD) to obtain comprehensive information about the structure of studied carbon material.

References: Jacek Jagiello and James P. Olivier. “A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.,” The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N₂ - Carbon Cylinder, single-wall nanotube by NLDFT**Ar - Argon Cylinder, single-wall nanotube by NLDFT**

Geometry:	Cylinder
Substrate:	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the graphitic surface of an infinitely long cylinder.

This model is particularly useful for characterizing carbon single-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N₂ - Carbon Cylinder, multi-wall nanotube by NLDFT**Ar - Argon Cylinder, multi-wall nanotube by NLDFT**

Geometry:	Cylinder
Substrate:	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and multiple concentric graphitic surfaces of infinitely long cylinders.

This model is particularly useful for characterizing carbon multi-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References:	P. Tarazona, Phys. Rev. A 31: 2672 (1985).
	Idem, Phys. Rev. A 32: 3148 (1985).
	P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987)

Ar - Zeolites H-Form by NLDFT

Geometry:	Cylinder
Substrate:	Zeolite
Category:	Porosity
Method:	Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is particularly useful for characterizing oxides and H⁺ and (NH₄)⁺ exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

Ar - Zeolites Me-Form by NLDFT

Geometry:	Cylinder
Substrate:	Zeolite
Category:	Porosity
Method:	Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is similar to the model above, but it more appropriate is for characterizing alkali metal exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

MODELS BASED ON CLASSICAL THEORIES

Both surface energy distribution and pore size distribution may be evaluated using classical approaches to model kernel functions for use with equation (1) of the DFT Theory in [*DFT \(Density Functional Theory\) on page B - 12*](#). Be aware that the deconvolution method only provides a fitting mechanism; it does not overcome any inherent shortcomings in the underlying theory.

SURFACE ENERGY

The use of classical theories to extract adsorptive potential distribution is mostly of historical interest. At a minimum, the equation must contain a parameter dependent on adsorption energy and another dependent on monolayer capacity or surface area. This is sufficient to permit the calculation of the set of model isotherms that is used to create a library model. The Langmuir equation has been used in the past, as have the Hill-de Boer equation and the Fowler-Guggenheim equation. All of these suffer from the fact that they only describe monolayer adsorption, whereas the data may include contributions from multilayer formation.

PORE SIZE

It is well established that the pore space of a mesoporous solid fills with condensed adsorbate at pressures somewhat below the prevailing saturated vapor pressure of the adsorptive. When combined with a correlating function that relates pore size with a critical condensation pressure, this knowledge can be used to characterize the mesopore size distribution of the adsorbent. The correlating function most commonly used is the Kelvin equation. Refinements make allowance for the reduction of the physical pore size by the thickness of the adsorbed film existing at the critical condensation pressure. Still further refinements adjust the film thickness for the curvature of the pore wall.

The commonly used practical methods of extracting mesopore distribution from isotherm data using Kelvin-based theories, such as the BJH method, were for the most part developed decades ago and were designed for hand computation using relatively few experimental points. In general, these methods visualize the incremental decomposition of an experimental isotherm, starting at the highest relative pressure or pore size. At each step, the quantity of adsorptive involved is divided between pore emptying and film thinning processes and exactly is accounted for. This computational algorithm frequently leads to inconsistencies when carried to small mesopore sizes. If the thickness curve used is too steep, it finally will predict a larger increment of adsorptive for a given pressure increment than is actually observed; since a negative pore volume is non-physical, the algorithm must stop. Conversely, if the thickness curve used underestimates film thinning, accumulated error results in the calculation of an overly large volume of (possibly nonexistent) small pores.

The use of equation (1) represents an improvement over the traditional algorithm. Kernel functions corresponding to various classical Kelvin-based methods have been calculated for differing geometries and included in the list of models.

MODELS INCLUDED**Kelvin Equation with Halsey Thickness Curve****N2 - Halsey Thickness Curve**

Geometry:	Slit
Substrate:	Average
Category:	Porosity
Method:	Nitrogen 77 K

The kernel function is calculated using the Halsey equation with standard parameters:

$$t = 3.54 \left(\frac{-5.00}{\ln(P/P_0)} \right)^{1/3}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension =	8.88 dynes cm ⁻¹
Molar density =	0.02887 g cm ⁻³

N2 - Halsey Thickness Curve

Geometry:	Cylinder
Substrate:	Average
Category:	Porosity
Method:	Nitrogen 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

Reference: G. Halsey, J. Chem. Phys 16, 931 (1948).

Kelvin Equation with Harkins and Jura Thickness Curve**N2 - Harkins and Jura Thickness Curve**

Geometry:	Slit
Substrate:	Average
Category:	Porosity
Method:	Nitrogen 77 K

The kernel function is calculated using the Harkins and Jura equation with standard parameters:

$$t = \left(\frac{13.99}{0.034 - \log(P/P_0)} \right)^{1/2}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm⁻¹

Molar density = 0.02887 g cm⁻³

N2 - Harkins and Jura Thickness Curve

Geometry: Cylinder

Substrate: Average

Category: Porosity

Method: Nitrogen 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

References: W. D. Harkins and G. Jura, J.A.C.S. 66, 1366 (1944).

J. H. DeBoer et al., J. Colloid and Interface Sci. 21, 405 (1966).

Kelvin Equation with Broekhoff-de Boer Thickness Curve

N2 - Broekhoff-de Boer Model

Geometry: Cylinder

Substrate: Average

Category: Porosity

Method: Nitrogen 77 K

The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

$$\log\left(p/p^0\right) = \frac{-16.11}{t^2} + 0.1682^{-0.1137t}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm⁻¹

Molar density = 0.02887g cm⁻³

N2 - Broekhoff-de Boer Model

Geometry:	Cylinder
Substrate:	Average
Category:	Porosity
Method:	Nitrogen 77 K

The calculation is similar to the above except that cylindrical geometry is assumed, and the film thickness depends on pore size (see reference).

References: Specifically, equations 20 and 21 in: J.C.P. Broekhoff and J.H. de Boer, "The Surface Area in Intermediate Pores," Proceedings of the International Symposium on Surface Area Determination, D.H. Everett, R.H. Ottwill, eds., U.K. (1969).

DFT MODEL REFERENCES

The papers listed below provide additional information on DFT models:

1. "Determination of Pore Size Distribution from Density Functional Theoretic Models of Adsorption and Condensation within Porous Solids," J.P. Olivier and W.B. Conklin, Micromeritics Instrument Corp; presented at the International Symposium on the Effects of Surface Heterogeneity in Adsorption and Catalysts on Solids, Kazimierz Dolny, Poland (July 1992).
2. "Classification of Adsorption Behavior: Simple Fluids in Pores of Slit-shaped Geometry," Perla B. Balbuena and Keith E. Gubbins, *Fluid Phase Equilibria*, 76, 21-35, Elsevier Science Publishers, B.V., Amsterdam (1992).
3. "Modeling Physical Adsorption on Porous and Nonporous solids Using Density Functional Theory," J.P. Olivier, *Journal of Porous Materials*, 3, 9-17 (1995).
4. "The Determination of Surface Energetic Heterogeneity Using Model Isotherms Calculated by Density Functional Theory," J.P. Olivier; presented at the Fifth International Conference on the Fundamentals of Adsorption, Pacific Grove, CA (1995).

D ERROR MESSAGES

If the *Action* response indicates to contact a Micromeritics service representative, record the error message, then make backup copies of any files involved in the operation.

1023 | Preparation failed in primary data.

Cause: The data preparation failed for a report for either the selected sample file or an overlay used by the report

Action: If this message references an overlay file, the report may still be printed for the selected sample file.

1026 | Calculations failed for [n] overlay data.

Cause: Calculations failed for overlays in the BJH report.

Action: Continue printing the report for the primary data.

1026 | Calculations failed for [n] primary data.

Cause: Calculations failed for the selected sample file in the BJH report.

Action: Select a different sample file.

1043 | Isotherm calculation called with no points available.

Cause: Not enough valid data points are available for the Isotherm report.

Action: Select a different sample file.

1043 | Fewer than 2 points available for [n,n] calculations.

Cause: Calculations failed for overlays in the BJH report.

Action: Continue printing the report for the primary data.

1044 | No [n,n] reports. Fewer than 2 points meet [n] criteria.

Cause: Not enough data points are found with pressures and volumes Adsorbed monotonically increasing (adsorption) or monotonically decreasing (desorption) for the BJH report.

Action: Select a different sample file.

2401 | FATAL ERROR.

Cause: An internal processing and / or hardware error has occurred during communication with the analyzer.

Action: Contact your Micromeritics service representative.

2430 | Error accessing file [n], error code = [n].

Cause A: Media may be damaged.

Action A: Clean the media drive. If this does not eliminate the problem, attempt operation

using a backup copy of the file.

Cause B: Hard disk may be damaged.

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. Do not use characters such as * or ?. Refer to the operating system manual.

2431 | Error writing file [n], error code = [n].

Cause A: User logged onto a computer that does not have Write permissions.

Action A: Contact your IT department to change user permissions.

Cause B: Insufficient hard disk to perform the operation.

Action B: Copy files not used regularly to the hard disk external media. Delete them from the hard disk, and then try the operation again.

2432 | Invalid response from MMI 'FILE_READ' request.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact a Micromeritics service representative if this error message continues.

2433 | New entries have been found in this directory. Refresh the directory information?

Cause: Several analyzer files (sample information, analysis conditions, adsorptive properties, or report options) have been added to this directory by some function other than the analyzer program.

Action: Click **Yes** to update the directory information with data from each new file. This operation may take a minute.

Click **No** to locate the file manually. This option may be feasible if a large number of files have been copied into the directory and the file name is known.

2434 | File [n] — Subset [n] wrote wrong [n] of data, expected [n] bytes.

Cause: An internal processing and/or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2436 | Path specification [n] is invalid.

Cause: An invalid path name and / or extension was entered.

Action: Type a valid path name (including the proper extension), then press **Enter**.

2437 | File name [n] does not exist.

Cause: The overlay file selected in the report option does not exist.

Action: Enter an existing file specification, or select a file name from the list box.

2437 | Overlay file [n] does not exist.

Cause: The overlay file selected in Report Options does not exist.

Action: Enter an existing file specification, or select a file name from the list box.

2438 | Cannot open scheduling diagnostic data save file: [n].

Cause: The file necessary for *Unit [n] > Diagnostics > Schedule Diagnostic Test* (...\\Hardware\\scheddiagtests.dat) could not be read. It is likely corrupted.

Action: Exit the application. Delete or rename the file mentioned in the error message and restart the software. A new file will be created.

2439 | Could not register file.

Cause: An unexpected error occurred when trying to access a data file.

Action: Contact your Micromeritics service representative.

2440 | Subset not found.

Cause: An unexpected error occurred when trying to access a data file.

Action: Contact your Micromeritics service representative.

2441 | Seek within file failed.

Cause: An unexpected error occurred when trying to access a data file.

Action: Contact your Micromeritics service representative.

2442 | Bad header in subset file.

Cause: An unexpected error occurred when trying to access a data file.

Action: Contact your Micromeritics service representative.

2443 | Subset owner denied access.

Cause: An unexpected error occurred when trying to access a data file.

Action: Contact your Micromeritics service representative.

2444 | Not a valid file format.

Cause: An unexpected error occurred when trying to access a data file.

Action: Contact your Micromeritics service representative.

2445 | Subset wrote the wrong amount of data.

Cause: An unexpected error occurred when trying to access a data file.

Action: Contact your Micromeritics service representative.

2446 | Error reading data.

Cause: An unexpected error occurred when trying to access a data file.

Action: Contact your Micromeritics service representative.

2447 | Error writing data.

Cause: An unexpected error occurred when you tried to access a data file.

Action: Contact your Micromeritics service representative.

2448 | The library directory [n] does not exist. Please re-install to make use of Windows 7 libraries.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Basic Mode default parameter file directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default Adsorptive Properties directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default convert sample file directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default parameter file directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default report options directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default sample file directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default script test file directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | File directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the *Directories* list to move to the correct directory.

2448 | Problem diagnostic directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The export data file directory [n] cannot be used. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The library directory [n] cannot be used. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The library directory [n] does not exist. Please re-install to make use of Windows 7 libraries.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The reference file directory [n] cannot be used. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The z-table file directory [n] cannot be used. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | User python script directory [n] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2449 | This field does not contain a valid file specification.

Cause: An invalid file name was entered.

Action: See the description of file naming conventions in a Windows manual, then re-enter the name.

2450 | Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?

Cause: An automatic analysis (an analysis in which sample files are created using the defaults) was processing while editing the defaults.

Action: Finish the edit session of the defaults, close the window, then restart the automatic analysis.

2451 | The specified folder does not exist.

Cause: Path entered in the file selector does not exist.

Action: Enter the correct file path, or browse for the correct file path.

2452 | The instrument is busy performing an operation of which this application is unaware. Do you want to cancel?

Cause: The instrument is performing an operation which is not recognized by the applic-

ation.

Action: Click **Yes** to have the application cancel the operation. Click **No** to allow the operation to continue.

2456 | Insufficient file handles available. Application cannot continue.

Cause: More than 50 files are open at the same time.

Action: Refer to an operating system manual then set the limit for open files to 50 or greater. Alternatively, close any unused open files.

2458 | An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause: An attempt was made to exit the application while the instrument was performing a critical operation. This operation must be completed before the application can be exited.

Action: Wait a short time and attempt to exit the application again.

2459 | An instrument is busy. A delay in restarting this application could result in loss of new data. Continue with program Exit?

Cause: An attempt was made to exit the application while an analysis was in progress. While this is possible, the data collected when the application is inactive will not be permanently recorded until the application is re-started. A power failure to the instrument could cause some data to be lost.

Action: If not concerned with the potential for loss of data should a power failure occur, click **Yes** to continue; otherwise, click **No**.

2460 | Fatal Communications error on [n].

Cause: There was a fatal error in communication between the application and the software in the instrument. All displays for that instrument will be closed.

Action: Ensure that the analyzer is connected to the computer on the communications port configured in the *Setup* program. Stop and restart the analyzer software. Contact your Micromeritics service representative.

2461 | No instruments are in operation. This application will unconditionally terminate.

Cause: At least one instrument must be active for the application to operate. The initialization of all of the instruments configured with the Setup program has failed. The application stops.

Action A: Usually this message is preceded by another message giving the reason for the instrument's failure to initialize. Refer to the instructions for that message.

Action B: Ensure that the instrument is attached to the computer on the communications port configured with the *Setup* program. Verify that the instrument's power switch is in the ON position and that the light on the front panel is illuminated. Contact a Micromeritics service representative if this error message continues.

2471 | [n] has an invalid communications port specified. It cannot be initialized.

Cause: The communications port specified for this analyzer during installation (or configuration) is invalid.

Action: Use the *Setup* program to change the analyzer configuration.

2474 | [n] communications port [n] specified in the program control files is already in use. Unit cannot initialize.

Cause: The communications port assigned to the indicated unit is in use by another program.

Action: End the program using the busy port or use the *Setup* program to change the communications port assigned to this program.

2475 | [n] communications port [n] specified in the program control files cannot be accessed. Unit cannot initialize.

Cause: The operating system has prevented the application from accessing the communications port.

Action: Review the hardware configuration of the computer. Ensure no other application is using the port. Contact a Micromeritics service representative if this error message continues.

2476 | [n] startup failed. Cannot initialize the communications port.

Cause: The communications port specified during installation is invalid.

Action: Use the *Setup* program to change the analyzer configuration. Contact a Micromeritics service representative if this error message continues.

2477 | [n] did not properly initialize.

Cause: The software was unable to initialize this instrument. This is usually caused by one of the conditions listed in the previous error messages.

Action A: Run the *Setup* program and ensure that a valid port is specified; if not, specify a valid one when prompted.

Action B: Reinstall the software, then restart application.

Action C: Contact a Micromeritics service representative if this error message continues.

2478 | Error copying sequential data segment.

Cause: An internal processing and / or hardware error occurred while accessing a portion of a sample file.

Action: Confirm that the media being accessed does not contain errors.. Contact your Micromeritics service representative.

2479 | Cannot open [n] because it is a [n] file.

Cause: The file cannot be opened because it came from a different Micromeritics application

Action: Select a different file.

2480 | File [n] cannot be analyzed. It is currently being edited.

Cause: An attempt was made to start an analysis using a file that is open for editing.

Action: Finish editing the file, save and close it, then start the analysis.

2481 | Error accessing the sample information file [n].

Cause: An unexplained error prevented access to this file.

Action: The hard disk drive may be corrupt. Run diagnostics.

2482 | File cannot be opened for writing.

Cause: An attempt was made to open a file currently being used.

Action: Locate the application using the file (in the Micromeritics application, use the Windows menu item to get a list of all open windows, one of which may contain this file).

2483 | An analysis cannot be performed on [n]. It is open for editing and contains errors.

Cause: An attempt was made to use a sample file containing errors that is currently open.

Action: Go to the window containing the file, correct the errors, then save it.

2484 | The edit session for [n] must be saved before the analysis. Save changes and continue with the analysis?

Cause: An attempt was made to start an analysis using a file that contains unsaved changes and is open for editing.

Action: Click **Yes** to save the changes, then proceed with the analysis. Click **No** to cancel the analysis, then continue editing the Sample Information file.

2485 | The sample file [n] has an invalid status and cannot be used for this operation.

Cause: The selected file has a status other than *No Analysis*.

Action: Select a different sample file, or create a new one and use **Replace All** to copy parameters from the file originally selected.

2485 | The service test file has an invalid status and cannot be used for this analysis.

Cause: The selected file has a status other than *No Analysis*.

Action: Select a different service test file, or create a new one and use **Replace All** to copy parameters from the file originally selected.

2486 | Could not construct [n] report type. Program will terminate.

Cause A: Full rights to the application's folders and files is required.

Action A: Contact a system administrator to have full rights granted.
Cause B: An internal processing and / or hardware error has occurred.
Action B: Contact your Micromeritics service representative.

2487 | Could not start report generator. Error code [n]. Program will terminate.

Cause A: Full rights to the application's folders and files is required.
Action A: Contact a system administrator to have full rights granted.
Cause B: An internal processing and / or hardware error has occurred.
Action B: Contact your Micromeritics service representative.

2488 | File [n] cannot be opened for editing.

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

2489 | File [n] cannot be opened for writing.

Cause: The specified file in a *Save As* operation is already open for edit.
Action: Select a different file for the *Save As* operation.

2490 | No '.INI' file present. Application will terminate.

Cause: The ASCII .INI file containing initialization information and system options information used during program startup does not exist.
Action: Restore the .INI file from back-up, or re-install the software.

2491 | Highlighted fields contain errors. Please correct the errors before dialog box.

Cause: The fields highlighted in red contain invalid entries. The window cannot be closed until all errors are corrected.
Action: Check the entries, correct the errors, then close the window.

2492 | This field's entry is invalid.

Cause: The highlighted field contains an invalid entry.
Action: Check the entry and correct the error.

2493 | An entry is required for this field.

Cause: This field requires a valid entry to proceed.
Action: Enter or select an appropriate value.

2494 | Value is out of the valid range.

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

2495 | Value is out of the valid range. Enter a value between [n] and [n].

Cause: The entered value in the highlighted field is outside the valid range of values.

Action: Check the entry, then either enter or select an appropriate value.

2496 | Invalid number.

Cause: An invalid number was entered in the highlighted field.

Action: Check the entry, then either enter or select a valid number.

2497 | This field contains an invalid character.

Cause: An invalid character was entered in the highlighted field.

Action: Check the entry, then enter valid characters.

2498 | The requested change to the Sample's status is invalid at this time.

Cause: A request to change the file's status, for example, from *automatically collected* to *manually entered* could not be done.

Action: Contact your Micromeritics service representative. Record the name of the sample file in which the problem occurred.

2499 | Sequence number must contain at least 3 digits.

Cause: An attempt was made to enter a sequence number that did not contain at least three digits.

Action: Enter a sequence number that contains at least three digits.

2500 | All sample file names that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause: No more sample information files can be created using the currently entered file name sequence number.

Action: Go to **Options > Default Method**, then enter another sequence number.

2501 | System resources have reached a dangerously low level. Please close some windows to avoid the loss of data.

Cause: A large number of windows are open and consuming the system resources available to all applications.

Action: Close one or more windows. Contact your Micromeritics service representative.

2502 | Error writing to file [n] during print. Error code [n].

Cause: An error occurred in the file being written to during a print operation.

Action: Ensure there is sufficient space on the drive containing the file.

2505 | Error logger cannot be initialized. Error code [n]. Program will exit.

Cause: An internal processing error has occurred.

Action: Contact your Micromeritics service representative.

2506 | Sample file [n] has a *No Analysis* status and cannot be used for this operation.

Cause: The selected sample file does not have collected data and cannot be used for operations, for example, reporting.

Action: Enter the name of a file with a status of *Complete*, *Analyzing*, or *Entered*. Alternatively, select a sample file from the list box.

2507 | The sample has an invalid status and cannot be used for degassing.

Cause: A sample file has been selected which does not have a *No Analysis* or *Prepared* status.

Action: Select a different file with a status of *No Analysis* or *Prepared*.

2508 | The selected file [n] does not contain a valid script.

Cause: The selected service test file does not contain any steps or the file has become corrupted.

Action: If the service test file has no steps, open the service test file and add steps to the file using the *Service Test Script* tab .

2513 | Unable to read the calibration file [n].

Cause: An invalid calibration file was selected or cannot be read.

Action: Ensure the media containing the calibration file has no problems.

2514 | Unable to write the calibration file [n].

Cause: An attempt to save calibration data has failed due to possible media problems.

Action A: Ensure the destination location has no problems.

Action B: Choose an alternate media to save the calibration data.

2515 | Warning: Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?

Cause: The process of performing a calibration operation was started.

Action: Calibration operations should only be done by or under the direction of qualified service personnel.

2516 | Warning: Keeping a backup copy of the calibration data is recommended by Micromeritics. Would you like to do so now?

Cause: A calibration operation was performed and a backup copy is recommended.

Action: Go to **Unit [n] > Calibration > Save to File** to perform a calibration save operation.

2517 | Canceling this dialog will reset the calibration state to what it was when this dialog was first opened. Are you sure you want to cancel?

Cause: The calibration has not been accepted.

Action: If the calibration operation was successful, click [Accept](#).

2520 | No data points available for reporting.

Cause: The selected sample file does not have collected data and cannot be used for reporting.

Action: Select a different sample file.

2521 | Unable to program controller.

Cause: A hardware malfunction has occurred.

Action: Contact your Micromeritics service representative.

2522 | Invalid controller application file.

Cause: The application's control file has been corrupted or deleted.

Action: Reinstall the analysis program.

2523 | Programming controller failed.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2524 | CRC check failed on programming controller.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2525 | Unknown error programming controller.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2526 | Controller download was not successful.

Cause: An internal processing and / or hardware error has occurred.

Action A: Contact your Micromeritics service representative.

2527 | Controller CRC error on boot block.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2528 | Controller DRAM error.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2529 | Controller Com 1: error.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2530 | Controller Com 2: error.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2531 | Controller debug port error.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2532 | The instrument contains a different software version. Do you want to reset it?

Cause: The application has discovered a different version of software operating in the analyzer.

Action: If there are no analyzers other than the one connected to the computer, click **Yes**, then allow the updated software to load.

2533 | Analyzer initialization failed.

Cause: An internal processing and / or hardware error has occurred.

Action: Contact your Micromeritics service representative.

2534 | Unable to establish the TCP connection with [n].

Cause: There was a problem establishing communication with the analyzer.

Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection.

Ensure that no other Micromeritics application is initializing another instrument. If there is another Micromeritics application open and initializing an instrument, wait until the instrument initialization completes or is canceled.

Contact a Micromeritics service representative if this error message continues.

2548 | System status [n] [n].

Cause: There was a problem establishing communication with the analyzer.

Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2549 | Error accessing online manual file [n].

Cause: The operator's manual file could not be located.

Action A: Reinstall the application.

Action B: Copy the contents of the manual folder from the setup CD to the application directory.

2550 | Attempts to acquire the instrument's status timed out.

Cause: There was a problem establishing communication with the analyzer.

Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2551 | Cannot access web page [n].

Cause: The Micromeritics web page for DFT models cannot be accessed. This could be caused by an ISP problem of high internet traffic.

Action: Try the operation later.

2552 | Configured serial number does not match instrument.

Cause: An instrument was substituted without properly changing the instrument serial number.

Action: Use the installation program to add or move devices as necessary.

2553 | Dialog ID [n] can not be created!

Cause: A required window could not be found by the software.

Action: Re-install the software.

2554 | File [n] does not contain any report data.

Cause: The selected sample file has no reports selected for printing.

Action: Edit the sample file and select reports to print in Report Options.

2556 | File [n] cannot be opened. It is currently selected for an analysis.

Cause: The sample file is currently selected and is undergoing a critical operation.

Action: Open the sample file after the critical operation has completed.

2557 | Directory [n] does not exist.

Cause: The directory entered in a file selector is not valid.

Action: Enter a valid directory.

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.

2559 | SPC directory [n] does not exist.

Cause: A directory in one or more of the files selected in a SPC Report is invalid.

Action: Click **Samples** in the SPC report and verify the locations of all sample files.

2560 | File [n] cannot be created or opened. It has an unrecognized extension.

Cause: The extension specified in the file you are trying to create is not one which is recognized by the application.

Action: Change the extension of the file in the file name field of the file selector.

2563 | Cannot write. File or directory [n] read only.

Cause: The specified file name is marked as read-only

Action: Select a different file name.

2564 | Directory database [n] error [n] .

Cause: There is a problem creating the directory file used in file selectors.

Action: Verify the directory specified in this message is not marked read-only.

2570 | Instrument log database could not be opened: [n].

Cause: There is a problem opening the instrument log file [SN.LOG].

Action: Exit the software. Go to the \HARDWARE subdirectory of the instrument's program directory and rename the [SN.LOG] file where SN is the serial number of the instrument. A new log file will be created.

2571 | Instrument log database [n] error [n].

Cause: There is a problem opening the instrument log file [SN.LOG].

Action: Exit the software. Go to the \HARDWARE subdirectory of the instrument's program directory and rename the [SN.LOG] file where SN is the serial number of the instrument. A new log file will be created.

2572 | File [n] already selected for the analysis.

Cause: The file selected for analysis on one port has already been specified for another port.

Action: Choose a different sample file for each port.

2573 | A maximum of [n] files may be selected.

Cause: When using one file selector to select samples for all ports, the number of samples selected exceeds the number of ports on the instrument.

Action: Choose the proper number of samples or pick the sample for each port individually.

2574 | No samples, or completed samples selected.

Cause: When using one file selector to select samples for all ports, either no samples were selected or a file with *Complete* status was selected.

Action: Select samples that do not have a *Complete* status.

2576 | The instrument [n] is not calibrated.

Cause: The analyzer application is in the process of initializing the instrument and is unable to locate the calibration files.

Action A: Click **OK**. Go to **Unit [n] > Calibration > Load from File**, then select a file containing calibration data.

Action B: Click **OK**. Close the application, then use the *Setup* program to reinstall calibration files.

2577 | The python directory is missing or some of its contents have been removed.

Cause: When using Advanced reports, a necessary component is missing.

Action: Re-install the software.

2578 | Not enough available resources. Application will have reduced functionality. Reduce the number of open files or report windows.

Cause: There is not enough memory available to open the requested window.

Action: Close any unnecessary applications, then try the operation again.

2579 | The selected file has a status that is not compatible with the operation requested.

Cause: The selected sample file cannot be used for the selected operation. For example, a sample file with a *Complete* status cannot be used for analysis.

Action: Select a different sample file.

2580 | Problem encountered trying to load dbghelp.dll.

Cause: Files necessary for the application are missing or have been corrupted.

Action: Re-install the software.

2581 | Problem encountered trying to create the trace file.

Cause: Files necessary for the application are missing or have been corrupted.

Action: Re-install the software.

2582 | Bad MiniDumpWrite function found in dbghelp.dll.

Cause: Files necessary for the application are missing or have been corrupted.

Action: Re-install the software.

2583 | Error writing trace.

Cause: Files necessary for the application are missing or have been corrupted.

Action: Re-install the software.

2584 | The application encountered an unexpected error and will be halted.

Cause: Files necessary for the application are missing or have been corrupted.

Action: Re-install the software.

2585 | The following libraries are missing: [n]

Cause: This message is triggered on application start up if any of the library files used by an application, do not exist on disk.

Action: Add the library into the libraries.

2586 | Sample file [n] has no pressure table entries.

Cause: Trying to analyze a sample file with no pressure entries in *Analysis Conditions*.

Action: Either edit the *Analysis Conditions* and enter the pressure points to be used for analysis, or choose a sample file that already has the pressure points entered.

2588 | Sample file [n] is a [n] file and can not be analyzed on this instrument.

Cause: The specified sample file is from another Micromeritics application which is not usable with this application.

Action: Select a different sample file.

2589 | Sample file [n] can not be analyzed on this instrument.

Cause: The file specified for analysis cannot be used.

Action: Select a different file.

2590 | The default sample file [n] cannot be selected.

Cause: The default sample file (default method) cannot be used in this operation (e.g., as a sample file for analysis).

Action: Select a different sample file from the data directory.

2590 | The default sample file [n] can not be overwritten.

Cause: The default sample file (default method) cannot be used in this operation (e.g., as a sample file for analysis).

Action: Select a different sample file from the data directory.

2590 | An error occurred accessing file or directory [n].

Cause: The default sample file (default method) cannot be used in this operation (for example — as a sample file for analysis).

Action: Select a different sample file.

2591 | Cannot open problem diagnostic data save file: [n]

Cause: The file necessary for *Unit [n] > Diagnostics > Schedule Diagnostic Test* (\Hardware\probdiag.dat) could not be read. It is likely corrupted.

Action: Exit the application. Delete or rename the file mentioned in the error message and restart the software. A new file will be created.

2592 | The selected file has an extension that is not supported by this operation.

Cause: The selected file does not have a supported file extension.

Action: Open the adsorptive properties file. Open the FPI file selector and select another file with a supported file extension.

2593 | Warning: The selected file uses Unicode [n] or n[] encoding with non-ASCII characters.

Cause: The Advanced report is not saved in ASCII format, which is required by Python.

Action: Edit the file and save it in ASCII (ANSI) format.

2594 | The selected file uses Unicode [n] or [n] encoding and could not be read.

Cause: The Advanced report is not saved in ASCII format, which is required by Python.

Action: Edit the file and save it in ASCII (ANSI) format.

2595 | The selected file is too large (maximum allowed size is [n] MB).

Cause: The *Advanced* report is too large.

Action: Edit the file and reduce the size.

4012 | Psat gas in sample file does not match any gas in the unit.

Cause: If using *Measure psat of a gas in P_o and T* options in *Analysis Conditions*, the selected gas is not one of the selected gases in *Unit Configuration*.

Action A: If the incorrect psat was selected, change the psat gas.

Action B: If the gas was recently connected to the instrument, update the *Unit Configuration*.

4013 | The Incremental Dosing option is not available on the selected unit.

Cause: The MicroPore option was selected but the instrument does not have the micropore option.

Action: Install the MicroPore option before starting the analysis.

4014 | File [n] is not a valid file for conversion.

Cause: The file selected for conversion is not a valid file.

Action: Select only files that have been created by the proper program.

4015 | Error creating export file for sample [n].

Cause: A file error occurred during creation of an export output file.

Action: The output file name may be invalid. Ensure that the target directory exists and is not full or write protected. The target disk drive may be damaged or inoperative. Verify that other files may be created on the same drive. Contact your Micromeritics service representative.

4016 | Sample [n] has no data for export.

Cause: The file selected for export has a status of *No Analysis*. No export file will be created.

Action: Select a file which contains analysis data.

4017 | Damage to the instrument will result if the sample [n] has not been manually evacuated. Have you evacuated the sample?

Cause: *Backfill sample at start of analysis* was not selected on the *Sample Backfill Options* window. The sample tube is normally at atmospheric pressure when an analysis is started, and it must be backfilled before the analysis begins to prevent sample material from being drawn into the manifold.

Action: If the sample tube has been manually evacuated, click **Yes**. If not, click **No**, either perform a manual evacuation or go to the *Sample Backfill Options* window, then select *Backfill sample at start of analysis*.

4020 | Disabling this option may damage the instrument. Are you sure that the sample should not be backfilled?

Cause: *Backfill sample at start of analysis* was not selected on the *Sample Backfill Options* window. The sample tube is normally at atmospheric pressure when an analysis is started; it must be backfilled before the analysis begins to prevent sample material from being drawn into the manifold.

Action: To manually evacuate the sample prior to the start of the analysis, click **Yes**. Otherwise, click **No**, go to the *Sample Backfill Options* window, then select *Backfill sample at start of analysis*.

4021 | The entered [n] value [n] and Temperature Option of the Analysis Conditions) is outside the range of the pressures listed in the Psat vs Temperature Table (Adsorptive Properties).

Cause: The entered P_0 value is not within the range of pressures selected for analysis.

Action A: Enter a new P_0 value.

Action B: Add more pressures and corresponding temperatures to the *Analysis Conditions* pressure table to include the presently selected P_0 value.

4022 | The entered bath temperature value ([n] and Temperature Options of the Analysis Conditions) is outside the range of the temperatures listed in the Psat vs Temperature Table (Adsorptive Properties).

Cause: The entered bath temperature is outside of the range of temperatures specified in the *Adsorptive Properties*.

Action A: Change the entered temperature.

Action B: Change the adsorptive.

Action C: Add more temperatures and corresponding pressures to *Adsorptive Properties*.

4023 | The file [n] cannot be prepared for analysis. It is open for editing and contains errors.

Cause: An attempt was made to start an analysis using a file that contains errors and is open for editing.

Action: Finish editing this file, save and close it, then start the analysis.

4024 | Backfill gas in sample file does not match any gas in the unit.

- Cause:* The backfill gas specified in the sample information file does not match the analysis gas entered in the *Unit Configuration*.
- Action A:* If the wrong backfill gas was selected in the sample information file, change the backfill gas in the file.
- Action B:* If necessary, attach the appropriate gas cylinder, then enter the gas in the *Unit Configuration*.

4025 | There is no [n] attached to the unit.

- Cause:* The gas shown in the error message is specified in Analysis Conditions but is not connected to the unit.
- Action:* Go to **Unit [n] > Unit Configuration**, then enter the gas.

4026 | Cannot calculate Dubinin-Astkhov: bad least squares data.

- Cause:* Less than two selected data points are within the fitted pressure range.
- Action:* Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4027 | Fewer than two sample files have data suitable for heat of adsorption reports.

- Cause:* Less than two of the selected sample files for heat of adsorption reports contain appropriate data.
- Action:* Edit the *Quantity Adsorbed* table, or select other sample files.

4028 | Dubinin calculations cannot be performed because the affinity coefficient of the analysis gas is zero.

- Cause:* Dubinin values could not be calculated because the affinity coefficient of the analysis gas is zero.
- Action:* Access the *Dubinin Report Adsorptive* options in the sample file, then enter an appropriate value for the analysis gas.

4029 | At least two fitted data points are needed for Alpha-S calculations.

- Cause:* Fewer than two data points fall within the selected Alpha-s range.
- Action:* Edit either the calculation pressure in the fitted Alpha-s range, or use a different reference curve.

4030 | Preparations failed in primary data.

- Cause:* Appropriate data were not available to generate the report.
- Action:* This message was preceded by a different error message. Refer to the cause/action of the preceding message.

4030 | P_0 cannot be measured at intervals when dosing from the Psat tube.

Cause: The dosing option in *Adsorptive Properties* conflicts with the P_0 option.
Action: Either edit *Adsorptive Properties* and change the dosing option, or edit the P_0 option in *Analysis Conditions*.

4031 | Not enough points with a relative pressure in the range $[n,n]$.

Cause: Fewer than two data points selected for the Dubinin report fall within the selected relative pressure range.
Action: Edit the calculation pressure range or the fitted relative pressure range.

4032 | The Psat tube cannot be for dosing or for measuring saturation pressure when the cryostat is used.

Cause: The dosing option in *Adsorptive Properties* cannot be used in conjunction with the cryostat.
Action: Edit *Adsorptive Properties* and change the dosing option.

4033 | Not enough points to generate Dubinin Tabular Report.

Cause: There are fewer than two valid data points available for the Dubinin tabular reports.
Action: At least two micropore pressures must be selected for inclusion in the Dubinin report. Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4034 | Fewer than 2 points available for Dubinin calculations.

Cause: There are fewer than two valid data points available for Dubinin reports in one of the sample files selected for overlaying.
Action: At least two micropore pressures must be selected for inclusion in the Dubinin report. Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4035 | Cannot calculate optimized Astakhov exponent.

Cause: There are fewer than two valid data points in the relative pressure range specified. Astakhov reports will not be produced.
Action: At least two pressures must be selected for inclusion in the Astakhov report. Edit the selection of data points on the Astakhov interactive editor or on the *Astakhov Pressures* window.

4036 | Fewer than 2 points available for Horvath-Kawazoe calculations.

Cause: At least two data points must be selected for inclusion in the Horvath-Kawazoe analysis. No report will be produced.
Action: Edit the selection of points on the Horvath-Kawazoe interactive editor or on the *Horvath-Kawazoe* window.

4037 | Computations failed while processing the primary data set. No reports will be produced.

Cause: The preparation of data for reporting could not be successfully completed. No Horvath-Kawazoe reports will be produced. This message will always be preceded with another one containing additional information.

Action: Refer to the error message number which preceded this one for an explanation.

4038 | Fewer than 2 points available for the Langmuir Qm computation.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm must include at least two points above 0.02 relative pressure for the Langmuir equation to be applied.

Action: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the *Horvath-Kawazoe Report Options* window to prevent this message from appearing on future reports.

4039 | The isotherm does not meet the constraints of the Cheng/Yang assumption.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm does not correlate to the Langmuir assumption with a coefficient of 0.98 or more. The correction is not applicable to this isotherm or to the range of the data points selected.

Action A: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the *Horvath-Kawazoe Report Options* window to prevent this message from appearing on future reports.

Action B: Generate the Langmuir report for the same data points selected for the Horvath-Kawazoe report. If the Langmuir correlation coefficient can be brought above 0.98 by removing some points at high relative pressure, remove them, then reproduce the Horvath-Kawazoe reports.

4040 | The value of Qm computed from the Langmuir equation is too low.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The computed value is less than the volume adsorbed at the largest relative pressure included in the analysis. The correction is not applicable to this isotherm or to the range of the data points selected.

Action: The analysis will be performed and the Cheng/Yang correction will be applied to all points with a volume adsorbed less than the value of 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 [n].

Cause: The Cheng/Yang correction is usually inappropriate for any P/P_0 above the isotherm knee. In some instances, the computed pore sizes may decrease above the knee. While it is possible to include these relative pressures (usually above 0.1 P/P_0) in the analysis, the computed pore sizes for these pressures are usually meaningless.

Action: Change the data points selected for the Horvath-Kawazoe report to include only relative pressures at or below the knee of the isotherm, or change the Horvath-Kawazoe report options so that the Cheng/Yang correction is not applied.

4042 | 0.0 cannot be a starting or ending pressure for a geometric progression from low pressure.

Cause: An attempt was made to generate a pressure table from a geometrically progressing range.

Action: Change the 0.0 entered value.

4043 | 1.0 cannot be a starting or ending pressure for a geometric progression toward saturation.

Cause: An attempt was made to generate a pressure table from a geometrically progressing range.

Action: Change the 1.0 entered value.

4044 | Points in the Langmuir report pressure table lie outside the collected data.

Cause: Calculation pressure range is not being used. More than one of the report pressure table points is above the range of the collected data and more than one is below.

Action: Change the report pressure table to be more consistent with the collected data.

4045 | Points in the report pressure table lie outside the collected data.

Cause: Calculation range is not being used. More than one of the report pressure table points is above the range of the collected data and more than one is below.

Action: Change the report pressure table to be more consistent with the collected data.

4046 | [n] could not be opened for reading.

Cause: A thickness curve file could not be opened.

Action: If the problem persists, restart the computer, then optionally perform a media integrity check.

4047 | Warning: An error occurred while reading [n].

Cause: An error happened during a read operation of a thickness curve file.

Action: If the problem persists, restart the computer, then optionally perform a media integrity check.

4048 | Warning: An error occurred while restoring the heat of adsorption report editor.

Cause: The state of the heat of adsorption report editor could not be restored. Default settings will be used.

Action: No action.

4049 | The sample [n] does not have enough data. A minimum of two adsorption points is required.

Cause: A sample file has been included in the Heat of Adsorption report that does not have enough data.

Action: Remove the file from the selected file list.

4050 | None of the requested quantities adsorbed is within the range of the collected data of more than one sample file.

Cause: The *Heat of Adsorption* report failed because the specified quantities are not within the range of the collected data.

Action: Edit the quantities adsorbed so that they are within the range of the collected data, or select other sample files.

4051 | The sample [n] does not have any data in the range of the requested quantities adsorbed.

Cause: The sample's data cannot be interpolated to any of the quantities adsorbed.

Action: Edit the quantities adsorbed so that they are within the range of the collected data.

4052 | Fewer than three points are selected for this report.

Cause: At least three points are required for the BET calculations.

Action: Edit the calculation range in the BET report.

4053 | At least two data points must be selected for t-Plot calculations.

Cause: At least two points are required for the t-Plot calculations.

Action: Edit the calculation range for the t-Plot report.

4054 | Fewer than two data points are inside the fitted thickness range.

Cause: At least two points must be within the fitted thickness range for the *t*-Plot calculations.

Action A: Edit the calculation range for the *t*-Plot report.

Action B: Edit the fitted thickness range in the *t*-Plot report editor.

4055 | A positive BET surface area was not calculated. Please check your BET Report.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.

Action A: Assign more points to the surface area calculation.

Action B: Select a different surface area in the *t*-Plot report editor.

4056 | A positive Langmuir surface area was not calculated. Please check your Langmuir report.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.

Action A: Assign more points to the surface area calculation.

Action B: Select a different surface area in the t-Plot report editor.

4057 | At least two data points are needed for Freundlich calculations.

Cause: Less than two data points have been selected for the Freundlich report; at least two are required.

Action: Edit the selection of points on the Freundlich interactive editor or on the *Freundlich Pressures* window.

4058 | At least two data points are needed for Temkin calculations.

Cause: Less than two data points have been selected for the Temkin report; at least two are required.

Action: Edit the selection of points on the Temkin interactive editor or on the *Temkin Pressures* window.

4059 | Fewer than 2 points available for MP-Method calculations.

Cause: At least two points are required for the MP-Method calculations.

Action: Edit the calculation range for the MP-Method report.

4060 | Sample [n] contains no data points.

Cause: An attempt was made to save a sample without collected data as a t-curve or alpha-S curve.

Action: Repeat the *Save As t-curve* or *Save As alpha-S* operation after opening a sample that has collected data.

4061 | The t-curve must contain at least 2 points.

Cause: At least two points are required in a thickness curve definition.

Action: Edit the thickness curve.

4062 | Error during report preparation.

Cause: An internal processing error has occurred.

Action: Contact your Micromeritics service representative.

4063 | No subreports selected.

Cause: A report (e.g. BJH) comprised of several reports has no subreport selected.

Action: Edit the report in Report Options and select at least one subreport.

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.

4069 | Dosing method choice is invalid - the Analysis Conditions choice of *Absolute pressure dosing* requires that the Adsorptive Properties *Dosing Method* is set to *Normal*.

Cause: There is an incompatibility between the analysis conditions choice of *Absolute pressure dosing* and the adsorptive properties dosing method.

Action: Edit one of the choices.

4070 | Unable to load deconvolution model [n].

Cause: The list of available models was corrupted; therefore, the model selected could not be loaded for the deconvolution.

Action: Exit the application. Reinstall the software, then try again.

4071 | The selected pressures points do not form a valid set for deconvolution.

Cause: The data points selected for analysis do not contain enough information to allow a DFT data reduction.

Action: At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction. Edit the selection of data points on the DFT interactive editor or on the *DFT Pressures* window.

4072 | The range of pressures selected is too small to deconvolute using this model.

Cause: A null result was found using the selected model.

Action: At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction. Edit the selection of data points on the DFT interactive editor or on the *DFT Pressures* window.

4073 | The analysis gas [n] does not match the model gas [n].

Cause: The model assumes a specific gas, and the sample file uses a different one.

Action: Select a model that assumes the same gas.

4074 | The analysis temperature [n] does not match the model temperature [n].

Cause: The temperature for the selected model did not match the analysis temperature.

Action: Select a different model.

4075 | The models cannot be located in the models folder. Reinstall the software.

Cause: The models could not be located. They may have been inadvertently deleted or moved.

Action: Reinstall the software.

4076 | Invalid sample file - Adsorptive Properties *Non-condensing adsorptive* cannot be used unless Analysis Conditions *Absolute pressure dosing* is selected.

Cause: *Non-condensing adsorptive* is selected in Analysis Conditions / Adsorptive Properties and the *Absolute pressure dosing* check box is not selected in Analysis Conditions.

Action: If *Absolute pressure dosing* is needed, select *Absolute pressure dosing* on the Analysis Conditions window. Otherwise, edit the Analysis Conditions / Adsorptive Properties and deselect *Non-condensing adsorptive*.

4077 | Cannot get surface area for: [n]

Cause: The Isotherm report for the named overlay file has Per gram selected for the *Volume Adsorbed*, and the Isotherm report for the primary file has a surface area option selected for the *Volume Adsorbed*.

Action A: Edit the Isotherm report for the named overlay file, then select a surface area option for *Volume Adsorbed*.

Action B: Click **Overlays** on the *Report Options* window of the primary file, then remove the named overlay file from the list.

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

Cause: The Langmuir report cannot be generated from the selected points.

Action: Edit the calculation pressure range in the Langmuir report pressure window.

4080 | The compressibility factor table has [n] pressure entries. Only [n] are allowed.

Cause: The fluid property information file has too many rows of compressibility information.

Action: Delete rows for pressures that are not likely to be encountered in the analysis.

4081 | In the compressibility factor table for [n], the 4 temperatures below [n] and 7 temperatures above must span at least 10 [n].

Cause: The fluid property information file does not have enough columns of compressibility information for temperatures near the analysis temperature.

Action: Add rows for temperatures near the analysis temperature.

4082 | Compressibility factors must be greater than zero for temperatures near ambient and pressures up to the maximum manifold pressure.

Cause: Fluid property information indicates that the analysis gas will condense.

Action A: Check that the correct fluid properties are being used.

Action B: Change the pressure or temperature range for the analysis.

4083 | All rows of [n] do not have the same number of entries. Some rows will be filled or truncated.

Cause: The compressibility table does not have the same number of temperature entries for each pressure.

Action: Check the fluid property file for errors.

4084 | [n] does not have the correct format. Please load a fluid property information file.

Cause: The selected file was not a fluid property file.

Action: Select a file with fluid property information.

4085 | The temperatures of [n] are not strictly ascending.

Cause: The temperatures for the columns of compressibility factors are not in increasing order.

Action: Order the columns of compressibility factors by increasing temperature.

4086 | The pressures of [n] are not strictly ascending.

Cause: The pressures for the rows of compressibility factors are not in increasing order.

Action: Order the rows of compressibility factors by increasing pressure.

4087 | Error reading [n].

Cause: An error occurred while reading the selected fluid property file.

Action: The file is likely corrupted. Restore the file from backups if possible. If the file was one provided with the software, re-install the software.

4088 | Error writing [n].

Cause: An error occurred while writing the selected fluid property file.

Action: Check the disk and ensure there is plenty of unused space. Scan the disk for bad sectors, then retry the operation.

4089 | Can not open [n] for reading.

Cause: The selected fluid property information file could not be opened.

Action: The file is likely corrupted. Restore the file from backups if possible. If the file was one provided with the software, re-install the software.

4090 | Can not open [n] for writing.

Cause: The selected fluid property information file could not be opened.

Action: Check the disk and ensure there is plenty of unused space. Verify if the file has been marked as read-only. Restore the file from backups if possible. If the file was one provided with the software, re-install the software.

4090 | The compressibility factor table for [n] is empty.

Cause: The fluid property information in the file selected for analysis does not have any compressibility factors.

Action: Update the adsorptive properties files using a different fluid properties file.

4091 | Can not load [n] because the fluid property table is too large.

Cause: The fluid property information file is too large to be stored in a sample file.

Action A: Verify that the selected file is really a fluid property information file.

Action B: Delete unneeded rows and columns from the compressibility factor table.

4093 | Pressure targets at or above [n,n] will be excluded to prevent condensation in the manifold. Continue with the analysis?

Cause: The pressure table contains some points that would require dosing above the adsorptive's saturation pressure.

Action: Click **No** and modify the pressure table before restarting the analysis. Click **Yes** and perform the analysis omitting the points that would cause condensation.

4094 | All pressure targets were excluded. Analysis canceled.

Cause: All target pressures would require dosing above the adsorptive's saturation pressure.

Action A: Verify that the saturation pressures for the adsorptive are correct.

Action B: Use lower target pressures in the pressure table.

4095 | The compressibility file [n] is obsolete. Please load the fluid property information file (.FPI) for the adsorptive.

Cause: The sample file includes obsolete compressibility information from a *.DAT file.

Action: Load the *.FPI file for the adsorptive.

4096 | The Analysis Conditions dosing option Low pressure incremental dose mode cannot be used with the Adsorptive Properties dosing method Selective Adsorption.

Cause: There is a conflict between *Analysis Conditions* selections and *Adsorptive Properties* selections.

Action: Change the *Analysis Conditions* dosing option or do not use selective adsorption.

4097 | The Analysis Conditions dosing option Maximum volume increment cannot be used with the Adsorptive Properties dosing method Selective Adsorption.

Cause: There is a conflict between *Analysis Conditions* selections and *Adsorptive Properties* selections.

Action: Change the *Analysis Conditions* dosing option or do not use selective adsorption.

4098 | The Analysis Conditions dosing option First pressure fixed dose cannot be used with the Adsorptive Properties dosing method Selective Adsorption.

Cause: There is a conflict between *Analysis Conditions* selections and *Adsorptive Properties* selections.

Action: Change the *Analysis Conditions* dosing option or do not use selective adsorption.

4099 | The *Analysis Conditions* dosing option *Selective Adsorption* requires the *Analysis Conditions* choice of *Absolute pressure dosing*.

Cause: There is a conflict between *Analysis Conditions* selections and *Adsorptive Properties* selections.

Action: Change the *Analysis Conditions* to use absolute pressure dosing, or do not use selective adsorption.

4100 | The *Analysis Conditions* choice of *Absolute pressure dosing* requires that the *Adsorptive Properties* dosing method is set to *Normal* or *Selective Adsorption*.

Cause: There is a conflict between *Analysis Conditions* selections and *Adsorptive Properties* selections.

Action: Change the *Analysis Conditions* to use relative pressure dosing, or change the *Adsorptive Properties* dosing method.

4112 | Hard-sphere diameter, molecular weight, and mass flow constant have been updated from the fluid property information.

Cause: A new fluid property information file was loaded. The indicated fields have been updated with values from the file.

Action: This message is informational; no action is required.

4113 | An old version of the fluid property information was read. Please upgrade.

Cause: An older version of a fluid property information file was detected.

Action: Newer versions of the fluid property information files were installed with the software. Locate a newer file in the *Param* directory.

4114 | The backfill gas [n] in sample file [n] does not match any gas in the unit.

Cause: The selected backfill gas for the *Analysis Conditions* is not one of the gases connected to the instrument. Go to **Unit [n] > Unit Configuration** and verify the gas.

Action: Edit the *Analysis Conditions* backfill gas and change the gas to one which is connected to the instrument.

4115 | The P_0 [n] in sample file [n] does not match any gas in the unit.

Cause: The selected gas for the P_0 and T for the *Analysis Conditions* is not one of the gases connected to the instrument. Go to **Unit [n] > Unit Configuration** and verify the gas.

Action: Edit the *Analysis Conditions* P_0 and T and change the gas to one which is connected to the instrument.

4116 | The adsorptive [n] in sample [n] is different from port [n] adsorptive [n].

Cause: The adsorptive selected for one analysis port is different from the adsorptive that is in the sample file selected for another port. All ports must use the same adsorptive.

Action: Edit the *Analysis Conditions* and change the adsorptive to match the other ports.

4117 | Gas [n] in sample file [n] does not match any gas in the unit.

Cause: The adsorptive selected for the analysis is not one of the gases connected to the instrument. Go to **Unit [n] > Unit Configuration** and verify the gas.

Action: Edit the *Analysis Conditions* and change the adsorptive to one which is connected to the instrument.

4117 | Analysis gas in sample file [n] is not specified.

Cause: The adsorptive selected for the analysis is not one of the gases connected to the instrument. Go to **Unit [n] > Unit Configuration** and verify the gas.

Action: Edit the *Analysis Conditions* and change the adsorptive to one which is connected to the instrument.

4118 | The pre-analysis backfill gases differ. Each port must use the same backfill gas.

Cause: The pre-analysis gas selected for one port in the analysis is different from the adsorptive that is in the sample file selected for another port. All ports must use the same adsorptive.

Action: Edit the *Analysis Conditions* and change the pre-analysis gas to match the other port(s).

4119 | The post-analysis backfill gases differ. Each port must use the same backfill gas.

Cause: The post-analysis gas selected for one port in the analysis is different from the adsorptive that is in the sample file selected for another port. All ports must use the same adsorptive.

Action: Edit the *Analysis Conditions* and change the post-analysis gas to match the other port(s).

4120 | The analysis gases differ. Each port must use the same analysis gas.

Cause: The -analysis gas selected for one port in the analysis is different from the gas that is in the sample file selected for another port. All ports must use the same gas.

Action: Edit the *Analysis Conditions* and change the analysis gas to match the other port(s).

4121 | The Psat gases differ. Each port must use the same gas for P₀ or Psat measurements.

Cause: The gas selected for Psat measurement in the analysis is different from the adsorptive that is in the sample file selected for another port. All ports must use the same gas.

Action: Edit the *Analysis Conditions* and change the Psat measurement to match the other port(s).

4122 | The dosing source gases differ. When dosing from a sample port or the Psat tube and the

dosing source is charged from a gas inlet, each port must use the same dosing source gas.

Cause: The gas selected for dosing for one port in the analysis is different from the adsorptive that is in the sample file selected for another port. All ports must use the same gas.

Action: Edit the *Analysis Conditions* and change the gas selected for dosing to match the other port(s).

4123 | Helium adsorptive properties could not be found in the adsorptive properties library.

Cause: There is no *Adsorptive Properties* file for helium available to the application.

Action: An *Adsorptive Properties* file for helium was installed with the software. It was likely deleted. Either re-install the software, or create an *Adsorptive Properties* file for helium.

4124 | Dosing from port [n] cannot be done because a sample is selected for analysis on that port.

Cause: The *Analysis Conditions* of a sample file calls for dosing from a port which has a sample file assigned to it for analysis.

Action: If the *Analysis Conditions* are correct for the currently selected sample file, clear the sample file selection from the port used for dosing. Otherwise, select a different sample file.

4125 | Determination of P_0 from the dosing source can only be done if dosing is done from the Psat tube, sample port, or vapor source.

Cause: If measuring Psat over the source, one of the dosing options listed in the error message must be selected.

Action: Select one of the dosing options from the error message.

4126 | Warning: Measured ambient temperature is not available for calculating the maximum manifold pressure. Analysis will continue using [n].

Cause: Measured ambient temperature is not available. Analysis will continue with a reasonable room temperature

Action: Displayed as a warning. Continue the analysis.

4127 | The compressibility factor table has [n] temperature entries. Only [n] are allowed.

Cause: The fluid property information file has too many columns of compressibility information.

Action: Delete columns for temperatures that are not likely to be encountered in the analysis.

4128 | Error reading from [n].

Cause: There was an error importing data from the Clipboard using copy/paste.

Action: Retry the operation.

4132 | Unable to read model data from directory: [n].

Cause: A DFT model file is either missing from the *Models* subdirectory or has become corrupted.

Action: Restore models from a backup or re-install the software to provide the DFT models.

4133 | Unable to open model file: [n].

Cause: A DFT model file is either missing from the *Models* subdirectory or has become corrupted.

Action: Restore models from a backup or re-install the software to provide the DFT models.

4135 | HOA file [n] does not exist.

Cause: The sample file in the *Heat of Adsorption* report list does not exist.

Action: Go to **Report > Heat of Adsorption**. Click [Add Samples](#), then select the sample file.

4136 | HOA file [n] is corrupt.

Cause: The sample file in the *Heat of Adsorption* report list is corrupt.

Action: Go to **Report > Heat of Adsorption**. Select the corrupt sample file, then click [Remove Sample](#). Rerun the *Heat of Adsorption* report.

4137 | Measured P_0 is not allowed with a cryostat.

Cause: In an analysis using a cryostat, Measured P_0 was specified in *Analysis Conditions*.

Action: Edit the P_0 and T options in *Analysis Conditions* and choose a different option.

4138 | Only entered temperature is allowed with a cryostat.

Cause: In an analysis using a cryostat, the analysis conditions specify something other than entered temperature in the P_0 and T options.

Action: Edit the existing file and change the P_0 and T options, or choose a different sample file.

4139 | Dosing from Psat tube is not allowed with cryostat.

Cause: In an analysis using a cryostat, dosing from Psat tube was specified in the *Adsorptive Properties* in *Analysis Conditions*.

Action: Edit *Analysis Conditions / Adsorptive Properties* and choose a different option.

4400 | The computer does not have the communications port specified for the SmartPrep(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.

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.

4403 | Cannot communicate with SmartPrep Unit [n].

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: [n] 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.

4404 | The application version of the SmartPrep Unit [n] 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].

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 restart the application. Contact a Micromeritics service representative if this error message continues.

4406 | A maximum of [n] files may be selected.

Cause: Too many files were selected for the SmartPrep start degas operation.

Action: Retry the operation with the proper number of files (1 per port).

4409 | A free IP address on the same subnet as [n] could not be found.

Cause: All IP addresses on the network for the Ethernet card specified during installation are in use by other Micromeritics applications on this computer.

Action A: Uninstall unused Micromeritics applications.

Action B: Configure a different Ethernet card for use by the application using the application installer.

4411 | Error dosing.

Cause: The backfill timed out.

Action: Ensure there is gas available and the pressure regulator is set to the appropriate pressure. Also ensure that the gas supply regulator shutoff valve is open.

4412 | Error calibrating the servo.

Cause: Calibration results are out of range.

Action: Follow standard calibration procedures and try again. Contact a Micromeritics service representative if this error message continues.

4413 | Error overheating on port [n] . Current = [n,n], Target = [n,n], Limit = [n,n].

Cause: The temperature of the indicated mantle exceeded the maximum allowed value.

Action: Ensure the power and thermocouple connectors for the mantle are properly installed. Contact a Micromeritics service representative if this error message continues.

4414 | Error thermocouple unplugged on port [n]. Target = [n,n].

Cause: The thermocouple is unplugged or has malfunctioned.

Action: Ensure the thermocouple is plugged in. Contact a Micromeritics service representative if this error message continues.

4415 | Degas transducer zero calibration failed. Current Offset = [n] counts, Current Pressure = [n,n], New Offset = [n] counts, Nominal = [n] counts.

Cause: The pressure transducer offset exceeds the recommended limit.

Action: Ensure that the vacuum pump is on. Repeat the pressure offset calibration..Contact a Micromeritics service representative if this error message continues.

4416 | Degas transducer scale calibration failed. Reference = [n,n], Current = [n,n], New Scale = [n,n] / count, Nominal = [n,n] / count.

Cause: The transducer scale calibration was rejected.

Action: Contact your Micromeritics service representative.

4417 | Degas vacuum gauge low point calibration failed. Reference = [n,n] / count, Current = [0] counts.

Cause: The transducer offset calibration was rejected.

Action: Contact your Micromeritics service representative.

4418 | Degas vacuum gauge high point calibration failed. Reference = [n,n], Current = [0] counts.

Cause: The vacuum gauge calibration was rejected.

Action: Contact your Micromeritics service representative.

4419 | Error reading servo DAC.

Cause: There is a problem with the servo DAC timing out.

Action: Contact your Micromeritics service representative.

4422 | The file [n] does not exist.

Cause: The selected file does not exist on the disk drive.

Action: Select an existing file. Ensure that the file has been created before use.

4423 | The sample [n] is already selected on port [n].

Cause: The selected sample file is already selected for use on a different port.

Action: Select another sample file for this port.

4424 | The file [n] on port [n] could not be opened. Check if the sample file is already in use for editing or analysis.

Cause A: The selected sample file is already open by this or another application.

Cause B: The selected sample file is damaged.

Action: Select another sample file.

4425 | The sample [n] on port [n] has an invalid status and cannot be used for degassing.

Cause: The status of the file is not consistent with the current operation.

Action: Select a sample file that has not been used for an analysis. Only sample files with a status of *No Analysis* or *Prepared* may be selected.

4426 | Port [n] is currently in use. Operation cannot be started.

Cause: The current operation cannot be completed because the port is already in use.

Action: Wait for port to terminate operation or perform the desired operation on an unused port.

4430 | Pressure level is out of range.

Cause: The reference gauge reading is not valid for scaling the manifold pressure transducer.

Action: Adjust the manifold pressure until the reference gauge reading is in the recommended scale calibration range.

4431 | Pressure is out of range.

Cause: The reference gauge reading is not valid for scaling the manifold pressure transducer.

Action: Adjust the manifold pressure until the reference gauge reading is in the recommended scale calibration range.

6500 | Failed to evacuate manifold to VAC SET in [n] seconds. Calibration canceled.

Cause A: The vacuum set point is set too low.

- Action A:* Reset *VAC SET* point to 5.0 mmHg. If the *VAC SET* point is already at 5.0 mmHg or above, the vacuum gauge may need servicing.
- Cause B:* Leak in manifold.
- Action B:* Locate the leak and repair it. Restart calibration.
- Cause C:* Valve failure.
- Action C:* Identify the leaking valve. Contact your Micromeritics service representative.

6240 | Leak test failed on port [n].

- Cause:* With the sample port valve closed, the sample pressure increased by 0.15 mmHg before the leak test duration was completed.
- Action:* Check sample tube fitting and ensure that it is securely attached to the port. Then restart the analysis.

6241 | Maximum time limit exceeded before the port [n] elevator reached the UP position.

- Cause A:* The maximum time for the analysis was exceeded before the elevator reached the UP position. Ice may be present in the bottom or the neck of the dewar.
- Action A:* Check the dewar and remove ice if necessary. Then restart the analysis.
- 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.

6241 | Maximum time limit exceeded before the port [n] elevator reached the DOWN position.

- Cause A:* The maximum time for the analysis was exceeded before the elevator reached the UP position. Ice may be present in the bottom or the neck of the dewar.
- Action A:* Check the dewar and remove ice if necessary. Then restart the analysis.

6242 | Time limit exceeded while evacuating Psat tube.

- Cause:* The maximum time allotted for the Psat tube evacuation operation in a 2020 Physisorption analysis was exceeded.
- Action:* Contact your Micromeritics service representative.

6243 | Time limit exceeded while dosing Psat tube to 925 mmHg.

- Cause:* The sample was at a pressure greater than 0.5 mmHg at the start of the leak test. A leak test can not be performed unless the sample has been fully evacuated.
- Action:* Insert an evacuation task immediately before the leak test. If an evacuation task is already present, ensure that the evacuation is being performed at the same temperature as the test and that the duration of the evacuation is adequate.

6501 | The 1000 mmHg transducer offset exceeds recommended limits: [n]

- Cause:* The *Pressure Gauge Calibration* operation showed the transducer offset exceeds the recommended limit.
- Action:* Repeat the *Pressure Gauge Calibration* operation. Contact a Micromeritics service representative if this error message continues.

6502 | The 10 mmHg transducer offset exceeds recommended limits: [n]

Cause: The *Pressure Gauge Calibration* operation showed the transducer offset exceeds the recommended limit.

Action: Repeat the *Pressure Gauge Calibration* operation. .Contact a Micromeritics service representative if this error message continues.

6503 | The 0.1 mmHg transducer offset exceeds recommended limits: [n]

Cause: The *Pressure Gauge Calibration* operation showed the transducer offset exceeds the recommended limit.

Action: Repeat the *Pressure Gauge Calibration* operation. .Contact a Micromeritics service representative if this error message continues.

6504 | Unable to write the calibration file [n].

Cause: A *Save to File* operation failed.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted. Contact a Micromeritics service representative if this error message continues.

6505 | Unable to read the calibration file [n].

Cause: A *Load from File* operation failed.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted. Contact a Micromeritics service representative if this error message continues.

6506 | Calibration file for [n] is invalid.

Cause: A *Load from File* operation failed.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted. Contact a Micromeritics service representative if this error message continues.

6516 | Sample pressure greater than [n,n] is not allowed.

Cause: An absolute pressure greater than (pressure) mmHg was attained during low pressure dosing (either fixed dose mode or incremental dose mode).

Action: The analysis was canceled. All previously collected data were stored.

6517 | Total volume dosed greater than [n,n] is not allowed.

Cause: The maximum total volume dosed in a 2020 Physisorption analysis was exceeded.

Action: Contact your Micromeritics service representative.

6518 | Pressure of [n,n] exceeds the maximum manifold pressure of [n,n].

Cause: The maximum pressure in a 2020 Physisorption analysis was exceeded.

Action: Contact your Micromeritics service representative.

6519 | Psat gas is not condensing.

- Cause A:* The working dewar does not contain enough bath liquid.
Action A: Retry the operation after filling the dewar.
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.

6520 | Power failure detected.

- Cause:* A power failure occurred in the specified unit, and any analyses in progress were terminated.
Action: If the program was running and an Uninterruptible Power Supply (UPS) was attached, data points were collected and stored in the sample file. If enough data points were not collected, create another sample information file and start a new analysis.

6521 | Transducer overrange detected.

- Cause:* The transducer in the specified unit has detected a pressure equal to or greater than 1000 mmHg. The exhaust port on the sample tube may be blocked.
Action A: Inspect for and clear any blockage.
Action B: Observe caution when operating the analyzer manually. Contact a Micromeritics service representative if this error message continues.

6522 | Time limit exceeded while evacuating manifold.

- Cause A:* Maximum manifold evacuation time was exceeded before the vacuum set point was achieved. Vacuum pump may be turned off.
Action A: Turn on vacuum pump switch. Then restart the analysis.
Cause B: The vacuum pump oil level is low.
Action B: Check the vacuum pump oil level and add more oil if necessary. Then restart the analysis.
Cause C: The manifold is contaminated or leaking.
Action C: Correct the problem. Refer to the *Testing for Leaks* section in this manual. Then restart the analysis.

6523 | Analysis canceled: Time limit exceeded while evacuating sample (unrestricted).

- Cause:* The maximum time for evacuating the sample through the unrestricted valve was exceeded. Possible causes are a leak in the sample tube fitting or a crack in the sample tube.
Action: Check the sample tube and the sample tube fitting; ensure that the tube is securely attached to the port. Then restart the analysis.

6524 | Analysis canceled: Time limit exceeded while evacuating sample (restricted).

Cause: The maximum time allowed for evacuating the sample through the restricted valve was exceeded.

Action: Check the sample tube and the sample tube fitting to ensure that the tube is securely attached to the port. Verify that the sample is properly degassed. Then start the analysis again.

6525 | Power failure lasted too long.

Cause: A failure on an analyzer which has an Uninterruptible Supply attached has lasted for an hour or more; therefore, the analysis has been canceled.

Action: Determine the cause of the failure and correct it.

6526 | Time limit exceeded while backfilling manifold to [n,n] with [n].

Cause A: The maximum time was exceeded before the target pressure point was reached. The gas regulator may be set too low or turned off.

Action A: Set the gas regulator to 10 psig (0.7 bar). Then resume the analysis.

Cause B: The gas bottle is empty.

Action B: Connect a new gas bottle. Then resume the analysis.

6527 | Time limit exceeded while dosing manifold to [n,n] with [n].

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 10 psig (0.7 bar) then resume the analysis.

Cause B: The analysis gas cylinder is empty.

Action B: Connect a new analysis gas cylinder then resume the analysis

6528 | Low pressure gauge offset too high [n,n].

Cause: A check of the 10 mmHg or 0.1 mmHg gauge's offset during an automatic operation indicated it was too high.

Action: Contact a Micromeritics service representative if this error message continues.

6529 | 1000 mmHg pressure gauge offset is too high [n,n].

Cause: A check of the 1000 mmHg gauge's offset during an automatic operation indicated it was too high.

Action: Contact a Micromeritics service representative if this error message continues.

6530 | Volume calibration canceled due to failure (code [n]).

Cause: A problem occurred during volume calibration.

Action: Contact your Micromeritics service representative.

6531 | The gas configuration file for [n] is invalid.

Cause: The contents of the gas configuration file are not valid.

Action: Confirm there is sufficient free space on the media receiving the file and that the media is not corrupted). Contact a Micromeritics service representative if this error message continues.

6534 | Instrument [n] is not calibrated.

Cause: Calibration information for various analyzer components are missing.

Action: Run the application *Setup* program and reinstall calibration information for the specified unit.

6535 | Problem encountered evacuating to target [n,n], last pressure = [n,n], elapsed time = [n,n].

Cause: Evacuation during the manifold dosing operation did not come within the allowed range of the target.

Action: Check that the outlet stage of the gas regulator is within specification. If the problem occurs frequently, contact a Micromeritics service representative

6538 | Power failure detected. The sample is in an unknown condition. A run termination will be performed for safety.

Cause: A total power failure occurred and an Uninterruptible Power Supply was not connected while an analysis was in progress.

Action: Allow the analysis to terminate.

6546 | Desorption sample pressure less than [n,n] is not allowed.

Cause: A desorption target pressure less than 0.050 mmHg was requested. A target absolute pressure less than 0.050 mmHg can correspond to various target relative pressures depending on the value of P_0 . For example, if $P_0 = 760$ mmHg, a relative pressure less than $0.050 / 760.0 = 0.000658$ will result in an error. The analysis will terminate.

Action: Remove the low target relative pressure from the pressure table.

6547 | Vacuum group is not configured.

Cause: The vacuum group is incorrectly configured, or a 2060 attempted to access a vacuum system belonging to another vacuum group.

Action: Open the *Vacuum Groups* window. Ensure that only instruments shown in this dialog are currently powered on and physically connected to the same network. If other 2060s are on the same network, ensure that they are correctly configured or powered off.

6548 | A request to access the shared vacuum system was rejected

Cause: The vacuum group is incorrectly configured, or a 2060 attempted to access a vacuum system belonging to another vacuum group.

Action: Open the *Vacuum Groups* window. Ensure that only instruments shown in this dialog are currently powered on and physically connected to the same network. If

other 2060s are on the same network, ensure that they are correctly configured or powered off.

6549 | The shared vacuum system is not responding to requests for access.

Cause: The instrument managing a vacuum group is either powered off or is not connected to the same network as another instrument in the same vacuum group.

Action: Open the *Vacuum Groups* window. Ensure that all instruments shown in this window are currently powered on and physically connected to the same network.

6550 | Opening this valve will prevent an analysis on Unit [n] Serial [n] from proceeding. Open this valve?

Cause: A manual vacuum valve operation was requested on an instrument while another instrument in the same vacuum group was using the shared vacuum system.

Action: Press **Yes** to interrupt the current vacuum system operation and actuate the vacuum valve. Press **No** to allow the current vacuum system operation to continue; the manual vacuum valve operation will not be performed.

6551 | The vacuum system is in use by Unit [n] Serial [n]. Open this valve?

Cause: A manual vacuum valve operation was requested on an instrument while another instrument in the same vacuum group was using the shared vacuum system.

Action: Press **Yes** to interrupt the current vacuum system operation and actuate the vacuum valve. Press **No** to allow the current vacuum system operation to continue; the manual vacuum valve operation will not be performed.

6551 | The vacuum system is purging. Open this valve?

Cause: A manual vacuum valve operation was requested on an instrument while another instrument in the same vacuum group was using the shared vacuum system.

Action: Press **Yes** to interrupt the current vacuum system operation and actuate the vacuum valve. Press **No** to allow the current vacuum system operation to continue; the manual vacuum valve operation will not be performed.

6551 | The vacuum system may be in use by another instrument. Open this valve?

Cause: A manual vacuum valve operation was requested on an instrument while another instrument in the same vacuum group was using the shared vacuum system.

Action: Press **Yes** to interrupt the current vacuum system operation and actuate the vacuum valve. Press **No** to allow the current vacuum system operation to continue; the manual vacuum valve operation will not be performed.

6552 | The vacuum system is not responding.

Cause: The instrument managing the vacuum system was unavailable when an analysis was started on another instrument in the same vacuum group.

Action: Open the *Vacuum Groups* window. Ensure that all instruments shown in this window are currently powered on and physically connected to the same network .

6553 | Another instrument is currently using the vacuum system for a manual vacuum operation.

Cause: An analysis was started on one instrument while another instrument in the same vacuum group had a vacuum valve open either due to a manual vacuum valve operation or to a manifold over-pressure event .

Action:

6554 | Vacuum system client has been reset.

Cause: An instrument in a vacuum group is failing to respond to a periodic network connectivity check.

Action: Open the *Vacuum Groups* window. Ensure that all instruments shown in this window are currently powered on and physically connected to the same network.

6554 | Vacuum system server has been reset.

Cause: An instrument in a vacuum group is failing to respond to a periodic network connectivity check.

Action: Open the *Vacuum Groups* window. Ensure that all instruments shown in this window are currently powered on and physically connected to the same network.

E PYTHON MODULE - ADVANCED REPORTS

The mic Python module is automatically imported when running a user supplied script. The module provides access to primary and overlay isotherm data and provides support for summary, tabular, and graphical reports.

- **Summary reports.** Consist of summary sections, each containing a two-column table of label and value pairs. Summary reports are created with the *mic.summary* call.
- **Tabular reports.** Consist of one or more tables each containing one or more labeled columns of data. Tabular reports are created with the *mic.table* call.
- **Graphical reports.** Consist of a single graph with one or more curves on one or two y-axes. Graphical reports are created with the *mic.graph* call.

Calls for accessing the sample file data can be found in the *Mic Module Python Calls* section of this appendix. More advanced example python scripts are included in the analyzer software. Application specific discussions can be found on <http://www.micro-report.com>



The examples in this topic are also included as a part of the Micromeritics installation process and are located in the *Scripts* sub-directory.

RUN A SCRIPT

1. Open a sample file with a *Complete* file status.
2. Select *Advanced* in the drop-down list at the bottom of the window.
3. Select the *Report Options* tab.
4. Highlight *Advanced* in the *Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add**. Locate and select one or more python scripts then click **Select**. The selected scripts become a part of the drop-down list in the *Available Scripts* section of the *Advanced Report Options* window.
6. In the *Selected Reports* section, select up to five Advanced reports in the drop-down lists. Use the **Pressures** button to include or exclude available pressures in the report.
7. Click **OK** to close the window.
8. Click **Preview** on the *Report Options* tab to view all reports selected in the previous window.

EDIT A SCRIPT



When a script is added, the code is stored within the application. If the script changes outside of the application, the script file will have to be re-added to the Advanced Report Options window for the changes to take affect.

Field or Button	Description
Add	Adds one or more scripts to the <i>Available Scripts</i> box. The added scripts then become available as options in the <i>Selected Reports</i> section.
Edit	Edits the script stored within the application but does not affect the original .py text file.
Remove	Removes the script from the <i>Available Scripts</i> box but does not affect original .py text file
Replace	Replaces the contents of the selected script however, the script name remains the same.

REMOVE A SCRIPT

Select the script in the *Available Scripts* box then click **Remove**. The script is removed from the application however, the original .py text file is not affected.

GRAPHIC REPORTS

Available Mic Python calls for graphic reports:

- Add a curve
- Add a curve using the second Y-axis
- Create a new graphic report

SUMMARY REPORT

This script produces a summary report with two summaries:

```
import mic
mic.summary( "My Summaries" )

mic.summary.add( "Summary A",
                 ["label 1:", "label 2:", "label 3:"],
                 ["val1", "val2", "val3"] )

mic.summary.add( "Summary B",
                 ["label 4:", "label 5:", "label 6:"],
                 ["val4", "val5", "val6"] )
```

The result is:

<p>Summary A label 1: val1 label 2: val2 label 3: val3</p> <p>Summary B label 4: val4 label 5: val5 label 6: val6</p>

TABULAR REPORT

If more than one column is required, the call *mic.table* is employed. This script produces a tabular report consisting of two tables. **NOTE:** This script uses the Python package "numpy" and c-style formatting of the numerical values.

```
import mic
import numpy as np

mic.table("My Tables")

mic.table.addtable( "My set A" )
mic.table.addcolumn( "x", ["1.0", "2.0", "3.0"] )
mic.table.addcolumn( "y", ["0.5", "1.0", "1.5"] )

x1 = 0.2
x2 = 0.5
x3 = 3.14159/2
mic.table.addtable( "My set B" )
mic.table.addcolumn( "x", ["%8.3f" % x1,
                           "%8.3f" % x2,
                           "%8.3f" % x3 ] )

mic.table.addcolumn( "sin(x)", ["%8.3f" % np.sin(x1),
                                "%8.3f" % np.sin(x2),
                                "%8.3f" % np.sin(x3)] )

mic.table.addcolumn( "cos(x)", ["%8.3f" % np.cos(x1),
                                "%8.3f" % np.cos(x2),
                                "%8.3f" % np.cos(x3)] )
```

The result is:

My set A		
x	y	
1.0	0.5	
2.0	1.0	
3.0	1.5	

My set B		
x	sin(x)	cos(x)
0.200	0.199	0.980
0.500	0.479	0.878
1.571	1.000	0.000

ACQUIRE BASIC INFORMATION

To acquire the adsorption isotherm and other basic information about the sample being edited, the calls *mic.isotherm*, *mic.sample_information* and *mic.adsorptive_data* are applied.

This script produces a graph of the adsorption and desorption isotherms for both relative and absolute pressure, and prints summaries of the sample information and the adsorptive properties.

```
import mic

prel, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('rel')
mic.graph( 'Graphical Report 1', 'Rel. Press', 'Quantity Adsorbed' )
mic.graph.add( 'Sample isotherm', prel, qads )

pabs, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('abs')

mic.graph( 'Graphical Report 2' 'Abs. Press', 'Quantity Adsorbed')
mic.graph.add('Sample Isotherm', pabs, qads)

mass = mic.sample_information('sample mass' )
Tanl = mic.sample_information('analysis temperature' )
dens = mic.sample_information('sample density')

mic.summary( "Sample Information" )
mic.summary.add( "Sample Information:",
    [ "Number of adsorption points:",
      "Warm Free space:",
      "Cold Free space:" ,
      "Sample mass (g):",
      "Description:",
      "Analysis Temp:",
      "Sample Density (g/cm^3):" ],
    [ "%8.3f" % n_ads,
      "%8.3f" % warm_fs,
      "%8.3f" % cold_fs,
      "%8.3f" % mass,
      desc,
      "%8.3f" % Tanl,
      "%8.3f" % dens ] )

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()

mic.summary.add( "Adsorptive Data",
    [ "Cross Sectional Area",
      "Hard Sphere Diameter",
      "Density Conversion Factor",
```

```
        "Molecular Weight",  
        "Analysis gas"],  
    [ "%8.3f" % csa,  
      "%8.3f" % hsd,  
      "%8.3f" % dcf,  
      "%8.3f" % mol_weight,  
      analysis_gas  ]  )
```

Note the calls to *mic.isotherm* and *mic.adsorptive_data* above are each returning results as a list with elements of varying return type.

ACQUIRE REPORT RESULTS

Sample file report results may be accessed using the *mic.report* call. This script prints a summary of the results of the *t*-plot and BET reports.

```
import mic

sa  = mic.report("bet",  "surface area")
c   = mic.report("bet",  "bet constant")
vm  = mic.report("bet",  "monolayer capacity")
esa = mic.report("tplot", "external surface area")
vol = mic.report("tplot", "micropore volume")

mic.summary( "BET and T-plot Results" )

mic.summary.add( "Report Results",
                [ "bet surface area",
                  "bet constant",
                  "bety 6" ,
                  "tplot external surface area",
                  "tplot micropore volume"],
                [ "%10.5f" % sa,
                  "%10.5f" % c,
                  "%10.5f" % vm,
                  "%10.5f" % esa,
                  "%10.5f" % vol ] )
```

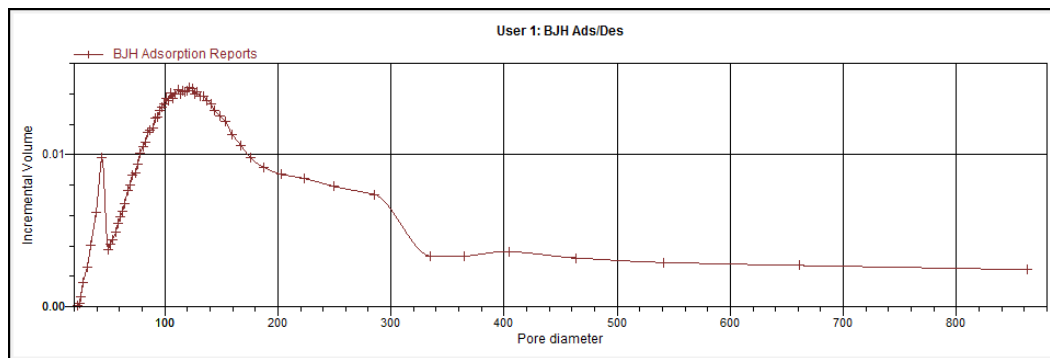
The result is:

Report Results	
bet surface area	796.36286
bet constant	137786.85871
bet monolayer capacity	182.96348
tplot external surface area	416.38843
tplot micropore volume	0.17931

Acquiring the results from a pore-distribution report such as the BJH method is done in a similar way as in the previous script except the return values from the *mic.report* call are slightly different since they involve lists of data. For example,

```
import mic
xdat, ydat, desc = mic.report('bjhads', 'incremental distribution' )
mic.graph( 'BJH Ads/Des', 'Pore diameter', 'Incremental Volume' )
mic.graph.add( desc, xdat, ydat )
```

The result is:



See the *Mic Module Python Calls* section for a more complete description of the usage and scope of the *mic.report* call.

ACQUIRE OVERLAY SAMPLE DATA

The call to obtain overlay sample data is similar to the calls for the primary sample. This script involves two overlay sample files.

The calls to obtain adsorptive data and report results for an overlay sample file using *mic.report* and *mic.adsorptive_data* have a very similar interface as the *mic.overlay* call, and a summary of their usage is shown in the example in this topic.

```
import mic

p, q, n, fsw, fsc, mass, desc = mic.isotherm('rel')
p1, q1, n1, fsw1, fsc1, mass1, desc1 = mic.overlay( 1, 'rel')
p2, q2, n2, fsw2, fsc2, mass2, desc2 = mic.overlay( 2, 'rel')

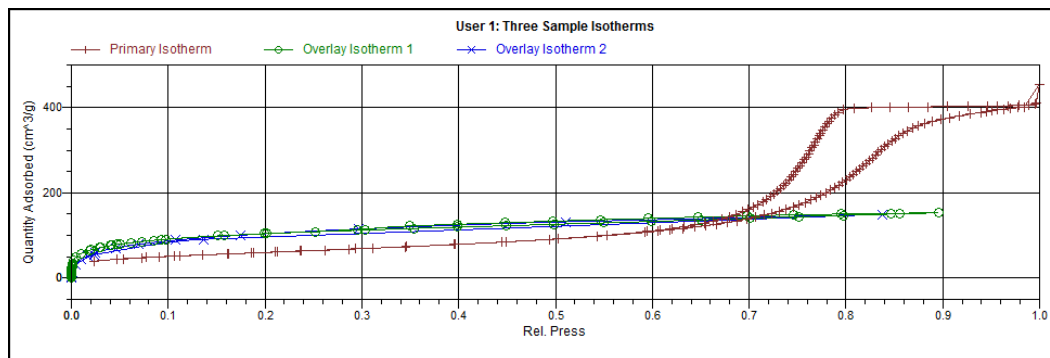
mic.graph( 'Three Sample Isotherms',
           'Rel. Press',
           'Quantity Adsorbed (cm^3/g)' )

mic.graph.add( 'Primary Isotherm ', p, q )
mic.graph.add( 'Overlay Isotherm 1', p1, q1 )
mic.graph.add( 'Overlay Isotherm 2', p2, q2 )

mic.summary( "A summary report" )

mic.summary.add( "Two samples",
                [ "Primary Sample:",
                  "Overlay Sample 1:",
                  "Overlay Sample 2:" ],
                [ desc,
                  desc1,
                  desc2] )
```

The results are:



Two samples

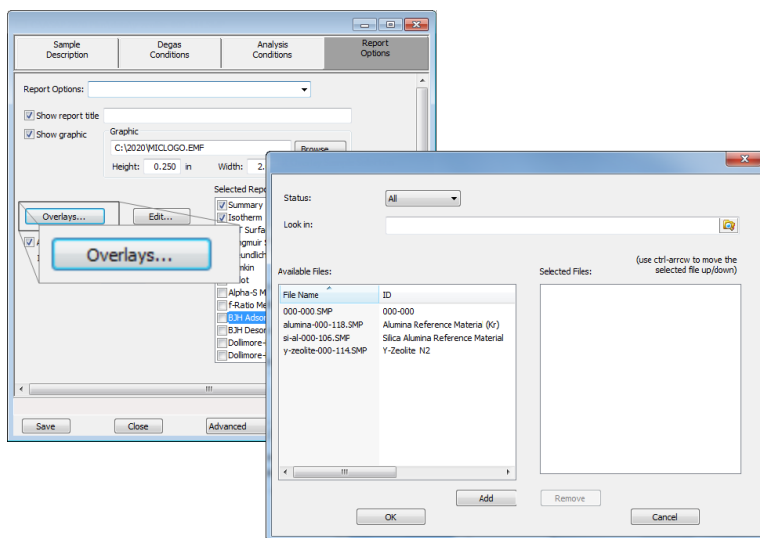
Primary Sample: 12 mm Tube N2 Silica-Alumina ADS-DES with FS
 Overlay Sample 1: Activated Carbon Hexane Dosed from Port 3 - 2
 Overlay Sample 2: Activated Carbon Tube C4 Butane Port 3

To enable the use of overlay data in the Advanced reports, the following two actions must be taken prior to running the script.

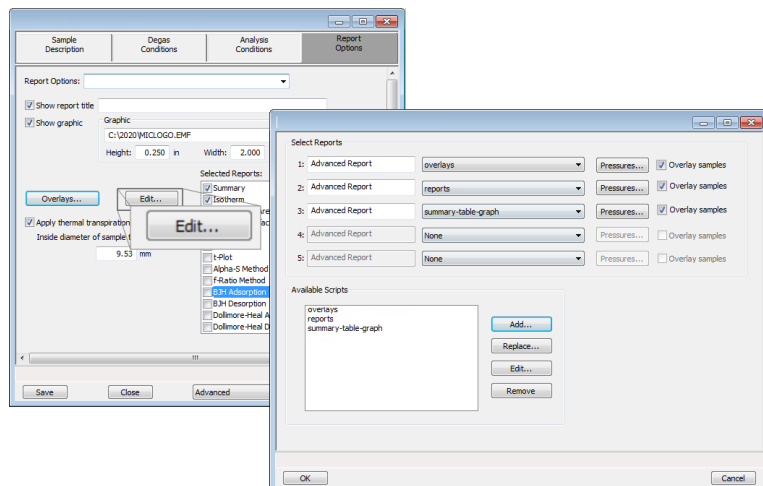
- Sample files to overlay must be selected, and
- The *Overlay samples* checkbox on the *Advanced Report Options* window must be selected

ENABLE THE USE OF OVERLAY DATA

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



- Double click a file name in the *Available Files* box to move the file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, double click the file name in the *Selected Files* box, or
 - Select a file name in the *Available Files* box. Click **Add** to move the selected file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, select a file name in the *Selected Files* box, then click **Remove**. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
3. Click **OK**.
 4. On the *Report Options* tab, highlight *Advanced* in the *Selected Reports* list box.
 5. Click **Edit** to the left of the *Selected Reports* list box.
 6. Select the *Overlay samples* checkbox to the right of the selected report.
 7. Click **OK**.
 8. Run the script using the instructions found in [Run a Script on page E - 1](#).



MIC MODULE PYTHON CALLS

TABLES

Available Mic Python calls for tables:

- Create a new tabular report
- Add a column
- Add a table

Create a New Tabular Report

```
mic.table( title='User Table' )
```

Keyword arguments:

```
title --- the tabular report title (default = 'User Table')
```

Add a Table

This script adds a table to the last created tabular report:

```
mic.table.addtable( name )
```

Keyword arguments:

```
name --- the table name
```

Add a Column

This script adds a column to the last created table:

```
mic.table.addcolumn( header, values )
```

Keyword arguments:

```
header --- column header; must be a string (or convertible)
```

```
values --- column values; must be a list of strings (or convertible)
```

SUMMARY REPORTS

Available Mic Python calls for summary reports:

- Add a summary section to the last created summary report
- Create a new summary report

Create a New Summary Report

```
mic.summary( title='User Summary' )
```

Keyword arguments:

```
    title --- the summary title
```

Add a Summary Section

This script adds a summary section to the last created summary report:

```
mic.summary.add( name, labels, values )
```

Keyword arguments:

```
    name    --- summary section name
    labels  --- column of labels; must be a list of strings
               (or convertible) and the same length as values
    values  --- column of values; must be a list of strings
               (or convertible) and the same length as labels
```

GRAPHIC REPORTS

Available Mic Python calls for graphic reports:

- Add a curve
- Add a curve using the second Y-axis
- Create a new graphic report

Create a New Graphical Report

```
mic.graph( title='User Graph', xlabel='X axis', ylabel='Y axis', ylabel='YY axis', xlinear=True, ylinear=True, yylinear=True )
```

Keyword arguments:

```
title      --- the graphical report title (default = 'User Graph')
xlabel     --- x-axis label (default = 'X axis')
ylabel     --- y-axis label (default = 'Y axis')
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)
```

Add a Curve

This script adds a curve to the last created graphical report:

```
mic.graph.add( name, x, y, yyaxis=False, color=None, linestyle='-', marker='a', graphtype='both' )
```

Keyword arguments:

```
name       --- the curve name
x          --- list of x values; must be a list of floats
            (or convertible) and the same length as y
y          --- list of y values; must be a list of floats
            (or convertible) and the same length as x
yyaxis     --- place this curve on the yy-axis if True
            otherwise place on the y-axis (default = False)
color      --- RGB color as an HTML hex string (e.g., '#4169e1')
            or a three-element list or tuple (e.g., [65,105,225]);
            if None, color is automatically selected (default = None)
linestyle  --- line style; (default = '-')
            '-' : solid
```



```

        '--'      : dash
        '.'       : dot
        '-.'      : dash dot
        '-..'     : dash dot dot
marker    --- marker shape; (default = 'a')
        '+'      : plus
        'o' or '0' : circle
        'x'      : cross
        '^'      : up triangle
        'v'      : down triangle
        's'      : square
        'd'      : diamond
        '8'      : hourglass
        '~'      : horizontal hourglass
        '' or None : no marker
        'a'      : automatically selected
graphtype --- graph type; (default = 'both')
        'curve' or 'c' : curve
        'points' or 'p' : points
        'both' or 'b' : curve-and-points
        'hist' or 'h' : histogram

```

Add a Curve Using the Second Y-Axis

This script adds a curve to the last created graphical report using the second y-axis:

```
mic.graph.addyy( name, xx, yy )
```

Add a curve to the last created graphical report using the second y-axis. The arguments to this call are the same as to mic.graph.add with the argument

GET PRIMARY ISOTHERM DATA

```
mic.overlay( overlay_number = 1, press_type='rel' )
```

Keyword arguments:

```

overlay_number --- the overlay number (1 through 8; default = 1)
press_type     --- the pressure basis; use 'rel' for relative pressure,
                  'abs' for absolute (default = 'rel')

```

Usage:

```

p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.overlay(1, 'rel')

p      --- array of pressure (relative or absolute);

```

```

                                empty-array if overlay is unavailable
qads      --- array of cumulative quantity adsorbed;
                                empty-array if overlay is unavailable
num_ads   --- number of points in the adsorption curve;
                                0 if overlay is unavailable
warm_fs   --- warm free-space; 0.0 if overlay is unavailable
cold_fs   --- cold free-space; 0.0 if overlay is unavailable
mass      --- sample mass; 0.0 if overlay is unavailable
desc      --- sample description; empty-string if
                                overlay is unavailable

```

GET OVERLAY ISOTHERM DATA

```
mic.overlay( overlay_number = 1, press_type='rel' )
```

Keyword arguments:

```

overlay_number --- the overlay number (1 through 8; default = 1)
press_type      --- the pressure basis; use 'rel' for relative pressure,
                    'abs' for absolute (default = 'rel')

```

Usage:

```
p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.overlay(1, 'rel')
```

```

p      --- array of pressure (relative or absolute);
                                empty-array if overlay is unavailable
qads   --- array of cumulative quantity adsorbed;
                                empty-array if overlay is unavailable
num_ads --- number of points in the adsorption curve;
                                0 if overlay is unavailable
warm_fs --- warm free-space; 0.0 if overlay is unavailable
cold_fs --- cold free-space; 0.0 if overlay is unavailable
mass    --- sample mass; 0.0 if overlay is unavailable
desc    --- sample description; empty-string if
                                overlay is unavailable

```

GET ADSORPTIVE DATA FOR EACH SAMPLE

```
mic.adsorptive_data( sample_number = 0 )
```

Keyword arguments:

```

sample_number --- Identifier for the adsorptive data to retrieve
                0           : the current sample file
                1 through 8 : the corresponding overlay sample file

```

Usage:

```
csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()
csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data(0)
```

```
csa          --- cross sectional area (nm^2)
hsd          --- hard sphere diameter (angstroms)
dcf          --- density conversion factor (dimensionless)
mol_weight   --- molecular weight
analysis_gas --- mnemonic for the analysis gas species
               (e.g., 'CO', 'H2')
```

GET SAMPLE INFORMATION ITEM

```
mic.sample_information( item, sample_number = 0 )
```

Keyword arguments:

```
item          --- string identifying the item to be returned.
                  Accepted identifiers are

                  'sample mass'
                  'sample description'
                  'analysis temperature' (degrees Kelvin)
                  'sample density'      ( g/cm^3 )

sample_number --- Sample to retrieve (default = 0).
0              : the current sample file
1 through 8    : the corresponding overlay sample file
```

Usage:

```
mass = sample_information('sample mass')
mass = sample_information('sample mass',0)
```

GET REPORT RESULTS

This script gets report results for the indicted report and sample.

```
mic.report( report_name, result, sample_number = 0 )
```

Keyword arguments:

```
sample_number --- Identifier for the sample data to retrieve
0              : the current sample file
1 through 8    : the corresponding overlay sample file
```

Usage:

```
sa = mic.report( 'bet' , 'surface area' )
porewidth, incvol, desc = mic.report( 'bjhads' ,
                                     'incremental distribution' )
```

The available report keywords, result keywords and a corresponding description of the result is listed in the table below:

Report keyword	Result keyword	Description
bet	surface area	Surface area (m ² /g)
bet	bet constant	BET constant (dimensionless)
bet	monolayer capacity	Monolayer capacity (cm ³ /g)
tplot	external surface area	External surface area (m ² /g)
tplot	micropore volume	Micropore volume (cm ³ /g)
bjhads	incremental distribution	Incremental Distribution
bjhdes	incremental distribution	Incremental Distribution
dhads	incremental distribution	Incremental Distribution
hk	incremental distribution	Incremental Distribution
dft	incremental distribution	Incremental Distribution
nldft	incremental distribution	Incremental Distribution

where the incremental pore distribution result above (for those reports which return this) is a list with three components being,

```
porewidth --- array of pore dimension boundaries (angstroms);
              empty-array if unavailable.
incvol      --- array of incremental pore volumes (cm3/g);
              empty-array if unavailable.
desc        --- Name of data set; empty-string if unavailable.
```

GET IMPORTED PORE DATA

```
mic.imported_pore_data( import_number = 1 )
```

Keyword arguments:

```
import_number --- the import number (1 through 8)
```

Usage:

```
xdat, ydat, desc = mic.imported_pore_data(1)

xdat --- array of pore dimension boundaries (angstroms);
        empty-array if unavailable.
ydat --- array of incremental pore volumes (cm3/g);
```

```
empty-array if unavailable.  
desc --- Name of data set; empty-string if unavailable.
```

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F WORKSHEETS

Worksheets in this section may be copied as needed.

- Input Gas Worksheet
- Sample Data Worksheet

INPUT GAS WORKSHEET

Physisorption

Port	Gas
P1	
P2	
P3	
P4	
P5	
P6	

SAMPLE DATA WORKSHEET

Use this form to assist in obtaining an accurate sample mass for report calculations.

Sample tube: _____ Sample: _____

Before Degas:

- | | | |
|---|-------|---|
| 1. Mass of empty sample tube | _____ | g |
| 2. Mass of sample tube plus sample | _____ | g |
| 3. Mass of sample (step 2 minus step 1) | _____ | g |

After Degas:

- | | | |
|---|-------|---|
| 4. Mass of sample tube plus sample | _____ | g |
| 5. Mass of sample (step 4 minus step 1) | _____ | g |

After Analysis:

- | | | |
|---|-------|---|
| 6. Mass of sample tube plus sample | _____ | g |
| 7. Mass of sample (step 6 minus step 1) | _____ | g |

Compare the sample mass obtained after analysis (Step 7) with the sample mass after degas (Step 5). These two values should be close in range. If a significant difference is noted, analysis problems may exist or the sample may have been improperly degassed.

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