MicroActive

Interactive Data Analysis Software

MICROMERITICS

Operator Manual

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ABOUT THIS MANUAL

The following formats are used throughout 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**
Field Labels	Field Labels are represented as italicized words — such as: *Sample, Automatically Collected,* and *Analysis Conditions*
Keyboard Commands	Keyboard commands are represented as bold font and black letters — such as: F2 and Alt+F4.
Menu Instructions	Menu instructions are represented as bold and italicized words — such as: ***File > New Sample*** and ***Reports > Start Report***.
Screen Tabs	Screen Tabs are represented as italicized words — such as: *Sample Description*, *Analysis Conditions*, and *Report Options*.
Screen Titles	Screen Titles are represented as italicized words — such as: *Analysis Adsorptive Properties*, *Free Space*, and *Sample Tube*.

Table of Contents

1 About the Software

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

The MicroActive feature offers a Windows interface with an easy way to collect, organize, archive, and reduce raw data and store sample information files for later use. Cut and paste graphics, scalable and editable graphs, and customized reports are easily generated. 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.

Computer Requirements

- Windows 7 Professional or higher operating system is recommended for the best user experience.
- CD-ROM

Menu Structure

All program functions use standard Windows menu functionality.

Main Menu Bar Options

Option	Description
File	Use to manage files.
Reports	Use to run reports and view the results.
Options	Use to edit the default method, specify system configuration, and data presentation formats.
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's manual, online instructional tutorials, the Micromeritics web page, the analyzer web page, and information about the analyzer.

ABOUT OPTION PRESENTATION

Options > Option Presentation

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

Presentation Display Table

Presentation Dislay	Description
Advanced	Displays all parts of sample information and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays the entire sample information file in a single window with no tabs across the top. This display option is used once the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Restricted	Displays the sample information file in a single window similar to the *Basic* display option with certain functions disabled. A password is set when the *Restricted* option is selected. That same password must be entered to change to the *Basic* or *Advanced* display option. This display type is typically used in laboratories where analysis conditions must remain constant — such as the pharmaceutical industry. The *Advanced* option is not available at the bottom of the window when using the *Restricted* display option.

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.

Physisorption and Chemisorption File Status and Description Table

File Status	Description
Complete	Sample information files used in an analysis that has been completed.
No Analysis	Sample information files which have not been used to perform an analysis.

Mercury Porosimetry File Status and Description Table

File Status	Description
HP Complete	A high pressure analysis has been performed using this sample file.
LP Complete	A low pressure analysis has been performed using this sample file.
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
Materials properties	.MTP	Param Directory
Methods	.MTH	Methods Directory
Penetrometer properties	.PEN	Param Directory
Report options	.RPO	Param Directory
Sample file	.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	

File Type, Extension, and Location Table (continued)

File Type	File Name Extension	Default Location
Spreadsheet	.XLS	
Unicode	.TXT	
Portable document format	.PDF	

Default File Location Table

File type	Default Location
Adsorptive Properties	Param directory
Analysis Conditions	Param directory
Degas Conditions	Param directory
Methods	Data directory
Penetrometer	Param directory
Report Options	Param directory
Sample Information	Data directory
Sample Tube	Param directory

APPLICATION SHORTCUTS

MENU SHORTCUTS

Shortcut menus are available for:

- 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 underscored letters do not display menus or windows, press the **Alt** key on the keyboard.

Keyboard Shortcut Table

Field or Button	Description
Alt + F	Opens a new sample file.
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Alt + H	Opens the *Help* menu.
Alt + R	Opens the *Reports* menu.
Alt + S	Opens the *Options* menu.
Alt + W	Opens the *Window* menu.
Ctrl + N	Opens a new sample file.
Ctrl + O	Opens the *File Selector*.
Ctrl + P	Closes all open reports.
Ctrl + S	Saves the open file.
F1	Opens the operator manual.
F2	Displays the *File Selector* window.
F3	When in the *File Selector* window, displays the file search box.
F4	When in the *File Selector* window, opens the address bar.

Keyboard Shortcut Table (continued)

Field or Button	Description
F6	Cascades open windows.
F7	Tiles open windows.
F8	Opens the *File Selector* to start a report from a selected .SMP file.
F9	Closes all open reports.
F10	Displays the *Heat of Adsorption* window.

COMMON FIELDS AND BUTTONS

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

Common Fields and Buttons Table

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

Common Fields and Buttons Table (continued)

Field or Button	Description
Destination group box	• **Preview.** Previews the predefined report on the screen. • **Print.** Sends the report to the default printer. • **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected. • **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
Edit	When working with report parameters, highlight the item in the *Selected Reports* list box and click **Edit** to modify the report details.
Exit	If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the application without saving the changes.
Export	Exports isotherm data in a sample information file as a .REP, .TXT or .XLS file. When saved to a file, the data can be imported into other applications.
File name text box	Select a file from either the *Name* column or from the library. The file name displays in the *File name* text box. Click **Open** or double click the file name to open the file. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
From / To text boxes	When working with report parameters windows, enter the *From* and *To* range for x- and/or y-axes.
List	Provides the option to create a list of sample or report options file information, for example, file name, date / time the file was created or last edited, file identification and file status.
Name column	A list of files in the selected directory or library.
Next	Click to move to the next window or next step.
OK	Saves and closes the active window.
Open	Opens the selected file. Alternatively, double click the file name in the *Name* column to open the file.
Prev	Click to move to the previous window.
Preview	Previews predefined reports. Click the tabs at the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled.
Print	Sends the report to the selected destination (screen, printer or file).
Remove	Click to remove an item from the list.

Common Fields and Buttons Table (continued)

Field or Button	Description
Replace	Click to select another file where the values will replace the current file's values.
Replace All	Click to select another .SMP file where the values will replace all values for the active Sample Information file. The original file will remain unchanged.
Report	Click to display a window to specify report output options. • **Start Date.** Displays a calendar to select the start date for the report. • **Preview.** Previews the predefined report on the screen. • **Print.** Sends the report to the default printer. • **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected. • **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
Save	Saves changes to the active window.
Save As	Saves a file in the active window under a different file name.
Table buttons	Use to modify the table contents. • **Insert.** Inserts one row above the selected row. • **Delete.** Deletes the selected row. • **Clear.** Clears all table entries and displays only one default value. • **Append.** Inserts one row at the end of the table.

OPTION PRESENTATION DISPLAY

Options > Option Presentation

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

Presentation Display Table

Presentation Dislay	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.
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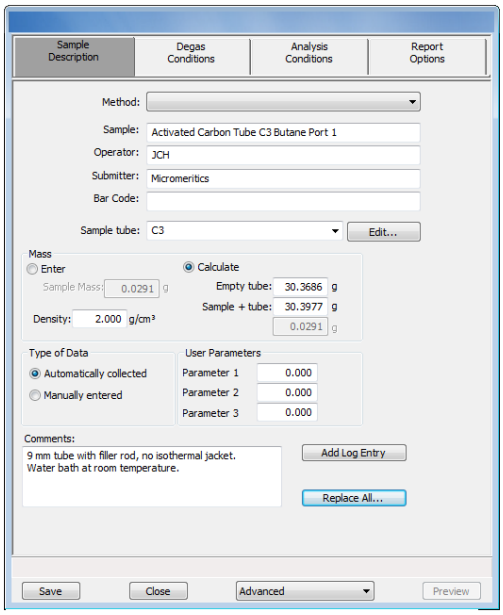
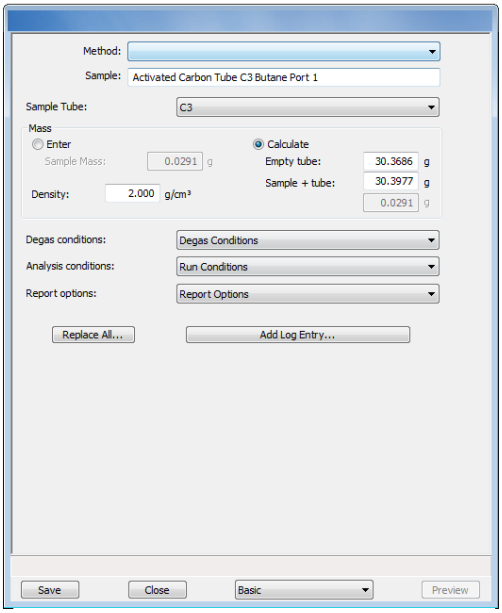
Restricted	Displays the sample information file in a single window similar to the *Basic* display option with certain functions disabled. A password is set when the *Restricted* option is selected. That same password must be entered to change to the *Basic* or *Advanced* display option. This display type is typically used in laboratories where analysis conditions must remain constant — such as the pharmaceutical industry. The *Advanced* option is not available at the bottom of the window when using the *Restricted* display option.

Specify or change the default presentation option 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 display

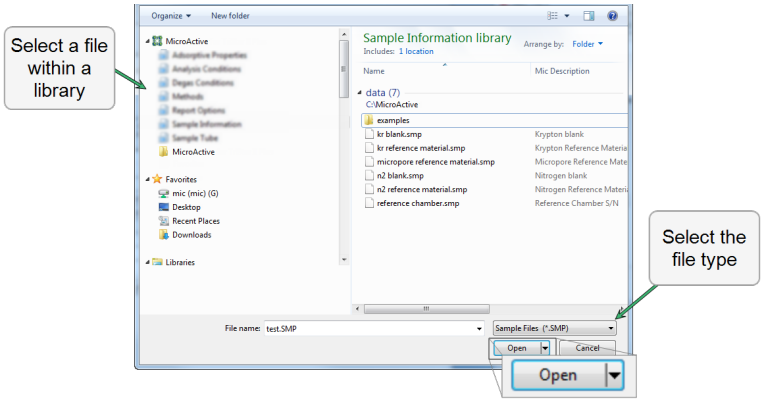


Basic / Restricted presentation display

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**.

MANAGE LIBRARIES

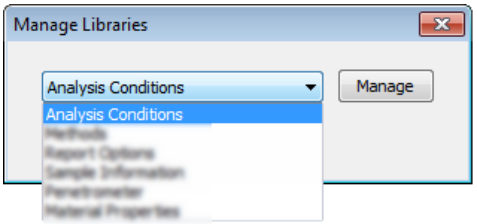
Options > Manage Libraries

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

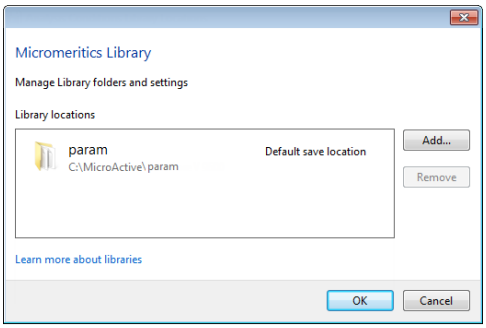
The library gathers sample and parameter files that are stored in multiple locations — such as folders on a computer's C drive, a network location, a connected external hard drive, or a connected USB flash drive — providing instant access at once to all of those files. Even though libraries do not store 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.**

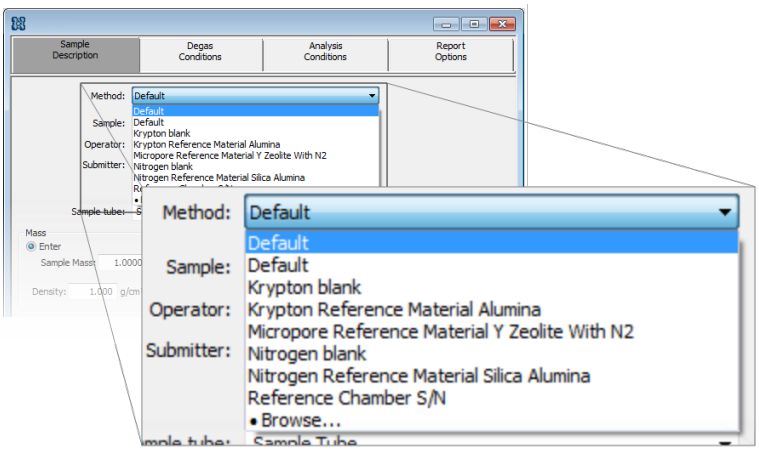
> When removing a folder from a library, the folder and its contents are not deleted from the original file storage location. However, when deleting files or folders from within a library, they are deleted from their original file storage location.

2. Click **OK** when done.

ABOUT 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.



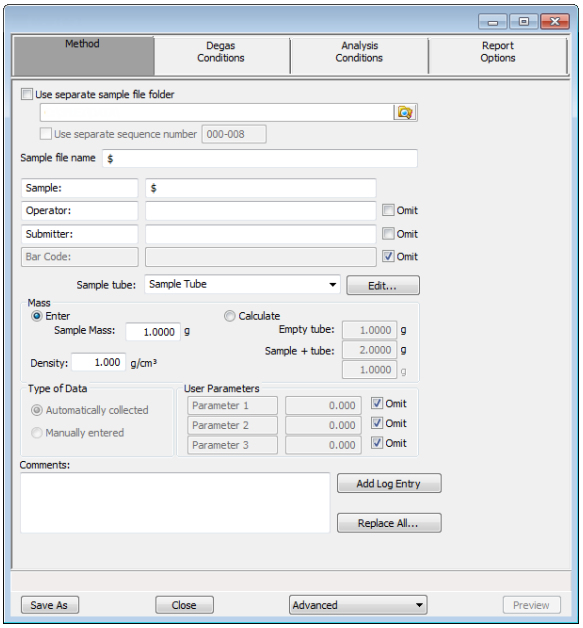
Method selections will vary

Default Method Files

Default Method Type Selected	Default File Modified
Physical Adsorption	Microactive.SMP
Chemical Adsorption	MicroactiveChemi.SMP
Mercury Porosimetry	MicroactiveHg.SMP

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*.
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**.

EDIT THE DEFAULT METHOD

Options > Default Method

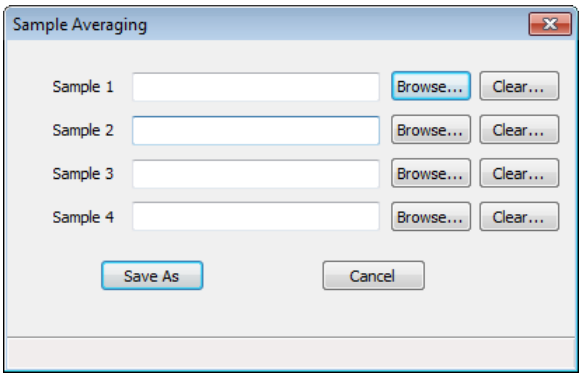
See *"Edit a Method" on the previous page* for detailed instructions on completing this window.

SAMPLE AVERAGING

File > Average

A sample file can be created in which the collected data are the average of up to four similar analyses. All information in the new sample file will be the same as in the first selected file except for the information entered in the *Sample Averaging* window. The collected data in the file will be the average of the data in the selected files.

Sample averaging can be used for sample files and blank correction files.



1. Go to ***File > Average.***
2. Click **Browse** to locate and select a sample file to be used for averaging. Click **Clear** to clear the selection or select another sample file.
3. Click **Save As** and enter a .SMP file name. Click **Save** to save the new file name or click **Cancel** to return to the previous window.
4. Click **Average** to display a graph combining all selections.

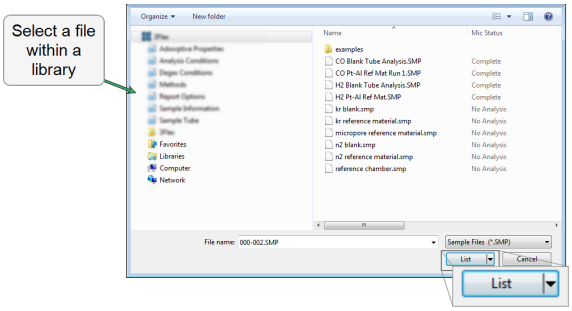
LIST FILES

File > List

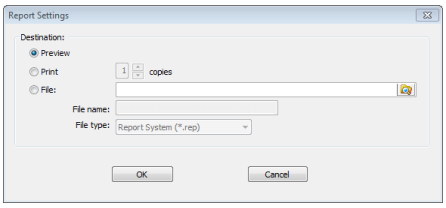
Provides the option to create a list of sample file information, for example, file name, date, time the file was created or last edited, file identification, and file status.

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

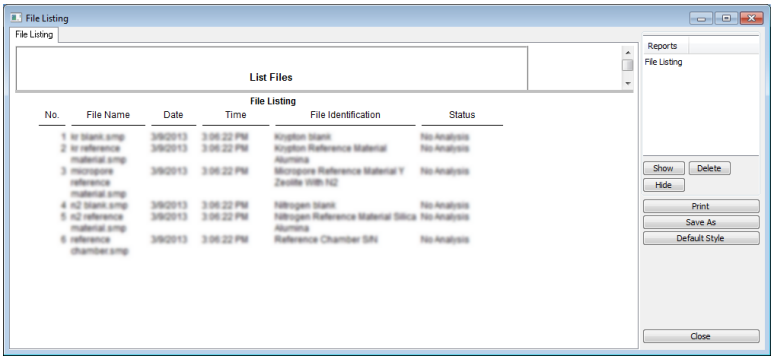
1. Select one or more files from the library. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.



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



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

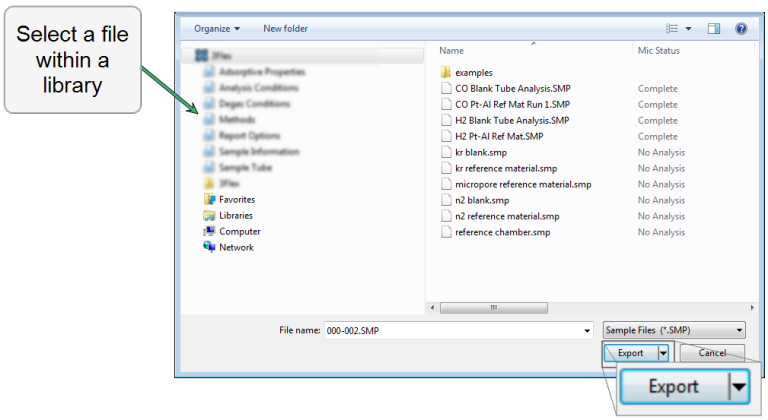


EXPORT FILES

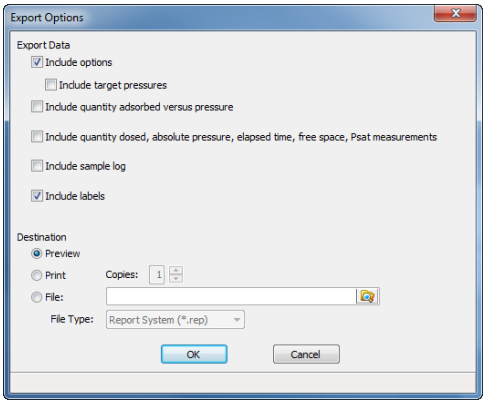
File > Export

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

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



2. Click **Export**.
3. In the *Export Options* window, select the type of data to include in the export file.



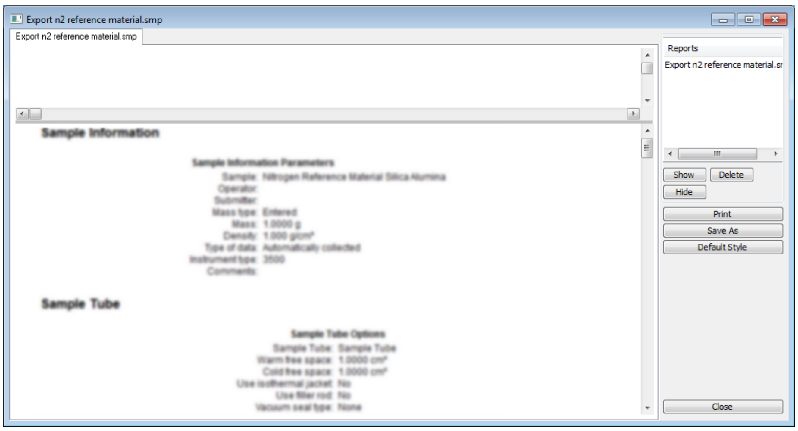
Types of data that can be included:

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

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

   - **Preview.** Previews the predefined report on the screen.
   - **Print.** Sends the report to the default printer.
   - **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected.
   - **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.

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



CONVERT FILES

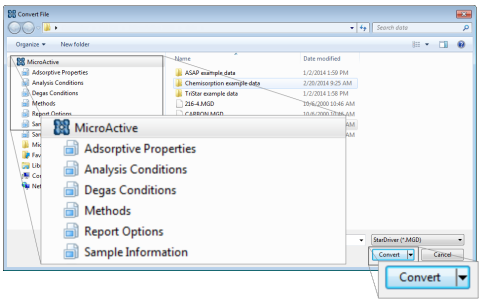
File > Convert

Converts the following samples file types to be compatible with the MicroActive application:

File Types for Conversion

File Type	File Name Convention
ASAP 2000 / ASAP 2400	SI*carbon*.DAT
ASAP 2000 Micropore	SM*carbon*.DAT
ASAP 2405	SK*carbon*.DAT
StarDriver	*carbon*.MGD
NOTE: Substitute the word carbon (shown above) for the actual file name.	

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 **Convert**. the file is automatically converted to a .SMP file.

SOFTWARE UPDATES

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

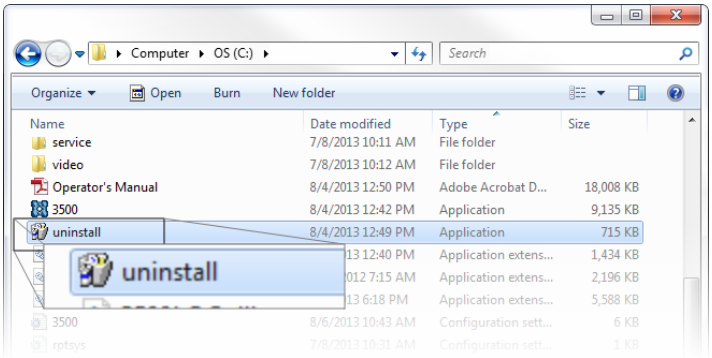
- a later version than the current installation
- the same version as the current installation
- an earlier version than the current installation

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

UNINSTALL THE SOFTWARE

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

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



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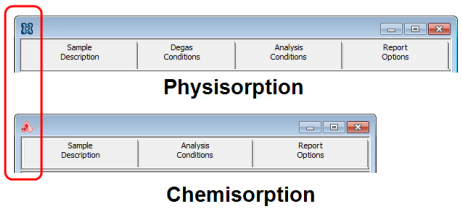
2 About Sample Files

Files created in the MicroActive application cannot be used in an analyzer for future analyses.

When viewing files from an analyzer, the screens are the same as the original application and may differ slightly from the screens in this operator manual.

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

A sample information file can consist of parameter sets; however, parameter sets can also stand alone. The icon on the left side of the title bar indicates the file type being edited. A sample information file may be created either prior to or at the time of analysis.



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

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

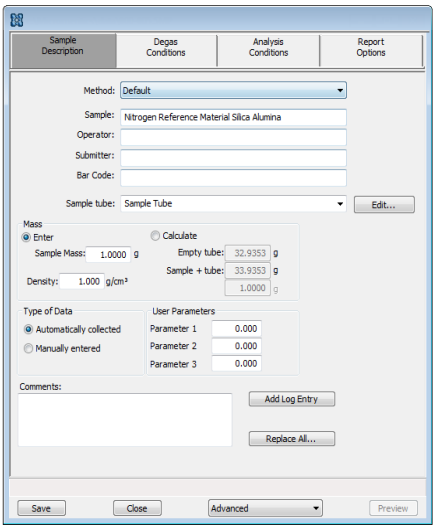
Specify or change the default presentation option by selecting ***Options > Option Presentation,*** or select *Basic* (or *Advanced*) from the drop-down list at the bottom of the window.

OPEN A SAMPLE FILE

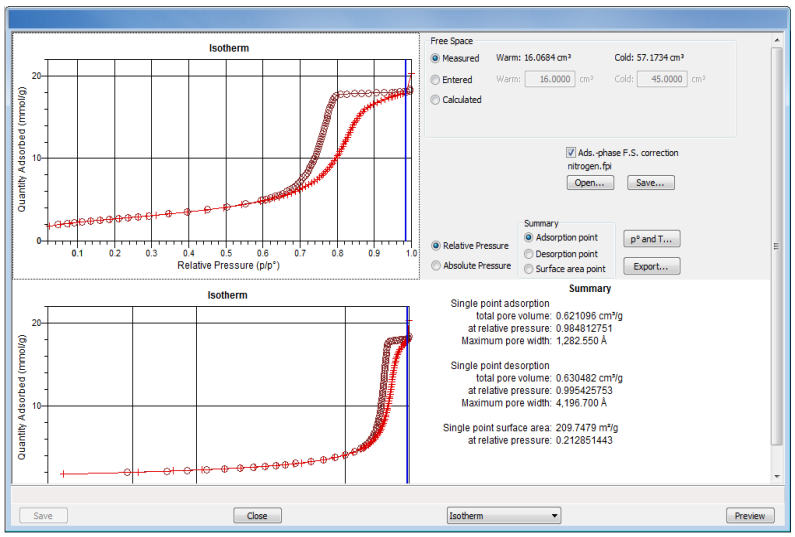
- When working with an existing file, it is recommended that a copy of the file be used rather than the original.
- Columns on the *File Selector* window can be sorted by clicking the column header. To sort the file list by status, click the *Mic Status* column header.

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

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



Example of tabbed file editor



Example of MicroActive report window

MANUALLY ENTER DATA

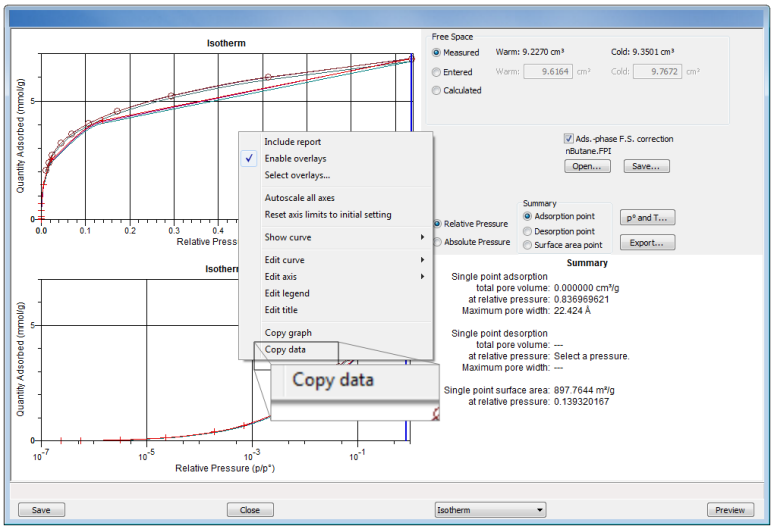
The images shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

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:

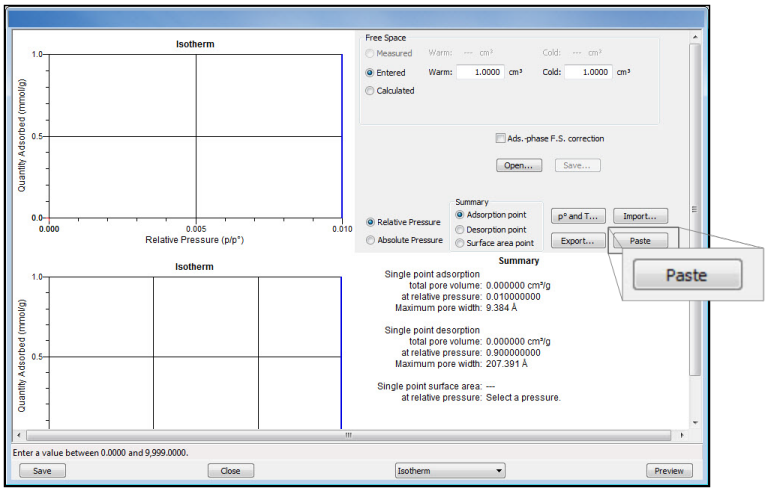
- Copy and paste onto the graph area of the interactive window
- Import data into the interactive window

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 either *Isotherm* or *Intrusion* depending on the type of sample.
7. Resize the interactive window to display the **Paste** button.



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

Import Manually Entered Data

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

ASCII text file format rules

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

For Physisorption or Chemisorption:

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

For Mercury Porosimetry:

- Pressure (psi)
- Pressure (MPa)
- Pressure (kPa)
- Pressure (Pa)
- Pressure (bar)
- Pressure (mbar)
- Pressure (Atm)\
- Intrusion (cm^3)
- Intrusion (cm^{263})
- Intrusion (ml)
- Intrusion (mm^3)
- Intrusion (mm^{263})

```
Silica Alumina : Adsorption
Relative Pressure        Quantity Adsorbed (cm3/g STP)
0.108629                 50.6657
0.22288                  60.7813
0.339909                 71.3095
0.459512                 84.4172
```

```
0.577447                102.672
0.654583                121.707
0.760074                179.096
0.855713                334.565
0.958511                394.675
0.996251                403.793


Silica Alumina : Desorption
Relative Pressure       Quantity Adsorbed (cm3/g STP)
0.996251                403.793
0.86016                 389.626
0.753567                256.264
0.664418                133.099
0.542416                96.7366
0.422295                79.7351
0.346371                71.5994
0.2519                  62.8256
0.152718                54.2336
0.103389                49.5803
```

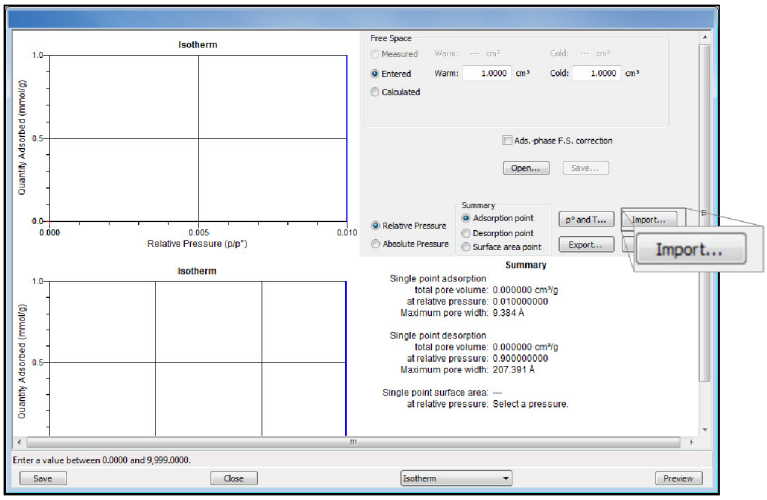
Sample Mercury Porosimetry ASCII text file

```
Cumulative Intrusion for Cycle 1
Pressure (MPa) Cumulative Intrusion (cm³/g)
0.138151               0.0
0.155414               0.00637965
0.310025               0.0327685
0.458529               0.0377315
0.816881               0.0411021
1.46145                0.0427142
2.60941                0.0444728
4.00728                0.0460848
5.35594                0.0474771
7.24049                0.0495286
9.63008                0.0519466
13.7208                0.0561233
24.4012                0.0808897
46.6833                0.800216
83.4181                1.15068
159.718                1.1586
286.294                1.16834
412.525                1.17574
```

To import the ASCII text file:

1. Go to ***File > New Sample***, then open a new sample information file.
2. On the *Sample Description* tab, select *Manually entered* in the *Type of Data* group box.

3. Click the *Advanced* down arrow at the bottom of the window, then select either *Isotherm* or *Intrusion* depending on the type of sample.
4. Resize the interactive window until the **Import** button displays.



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

3 Create Sample Files for the 3Flex

Create Sample Files using Advanced Presentation Option

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

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

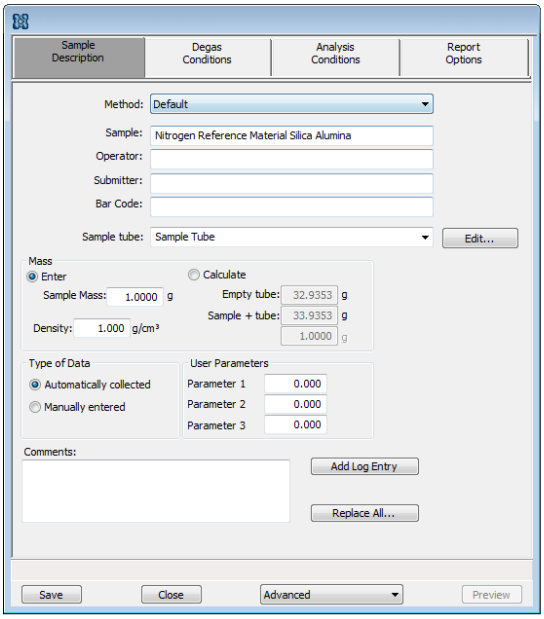
Specify or change the default display option by selecting ***Options > Option Presentation*** or select *Basic / Advanced* from the drop-down list at the bottom of the window.

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

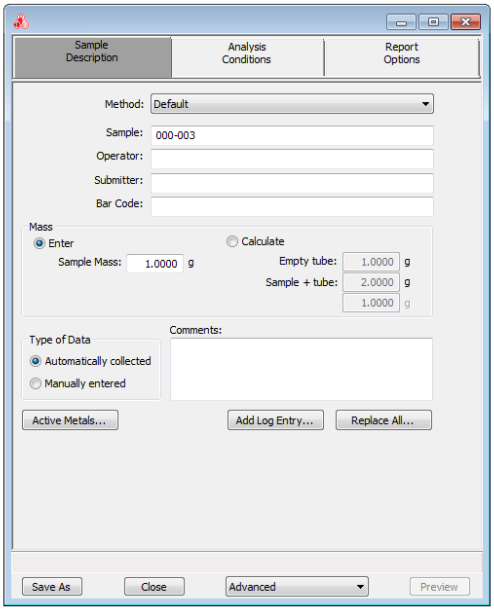
- *Sample Tube* files are edited on the *Sample Description* tab.
- *Adsorptive Properties* files are edited on the *Analysis Conditions* tab.

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

1. Go to ***Options > Option Presentation > Advanced*** and ensure *Advanced* has a checkmark.
2. Go to ***File > Open***.
3. Enter a new file name in the *File name* text box. Click **Open**.
4. Select the type of sample file.
5. Select a method from the *Method* drop-down list.

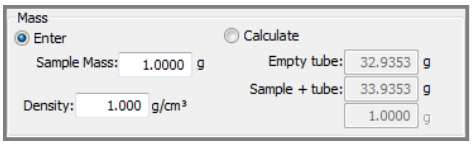


Physisorption

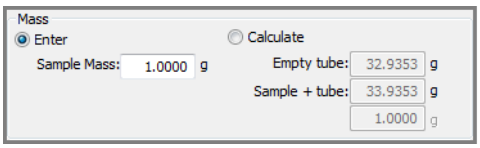


Chemisorption

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



Physisorption



Chemisorption

If mass = 1, the reported surface area equals the total surface area but it is always shown as m^2/g. If the actual mass is entered, the surface area is reported as m^2/g. Choose whether to enter mass manually or have the system automatically calculate mass. Enter a

value for sample mass. Mass can be changed any time before, during, or after analysis.

- **Enter.** Enables the *Sample Mass* field. Enter a value for the sample mass.
- **Calculate.** Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass:

$$Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$$

- **Density**. (for physisorption.) Value is used for the calculated free space method only. Use 0.000 for a blank analysis.

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.
    - **Automatically collected.** Select for all sample runs where the data are collected.
    - **Manually entered**. Select when another sample has been run on a different analyzer or different model analyzer so that data can be analyzed or used for comparison. If *Manually entered* is selected, the data are entered in the Isotherm interactive report. See *"Manually Enter Data" on page 2 - 3*.
11. For physisorption: The optional user-defined fields in the *User Parameters* group box may be used to enter and track information from another analyzer or source, along with other statistical process control (SPC) data.
12. For chemisorption: Click **Active Metals** to display a window of active metals. *"Active Metals" on page 3 - 8*.
13. Enter any pertinent information about the sample information file in the *Comments* text box. Entered comments are displayed in the report header.
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 **Replace**.
16. After completing the *Sample Description* tab options, click the other parameter tabs to edit more sample information file parameters.
17. Click **Save**, then click **Close**. To save as a different file name, go to ***File > Save As*** and enter a new file name. The file can also be saved as a different file type to save part of the sample file as a new parameter set such as Analysis Conditions, Report Options, etc.

Sample File Fields and Buttons Table

Field or Button	Description
Active Metals (for chemisorption)	Displays a list of active metals. See *"Active Metals" on page 3 - 8*.
Comments	Enter comments about the sample or analysis. Comments display in the report header.

Sample File Fields and Buttons Table (continued)

Field or Button	Description
Mass group box	If mass = 1, the reported surface area equals the total surface area but it is always shown as m^2/g. If the actual mass is entered, the surface area is reported as m^2/g. Choose whether to enter mass manually or have the system automatically calculate mass. Enter a value for sample mass. Mass can be changed any time before, during, or after analysis. • **Enter.** Enables the *Sample Mass* field. Enter a value for the sample mass. • **Calculate.** Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass: $Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$ • **Density**. (for physisorption.) Value is used for the calculated free space method only. Use 0.000 for a blank analysis.
Method	Select a method from the drop-down list. See *"About Methods" on page 1 - 13*.
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 5 - 1*.
Submitter	Enter submitter identification information. This text box may have been renamed or may not display if modified in ***Options > Default Methods.***
Type of Data group box	• **Automatically collected.** Select if the type of data will be automatically collected by the system while an analysis is running. • **Manually entered.** Use to enter data manually that was collected from another source. If *Manually entered* is selected, the Isotherm Report becomes available in the *Basic/Advanced* drop-down list for pasting or importing data into the file. See *"Manually Enter Data" on page 2 - 3*.

Sample File Fields and Buttons Table (continued)

Field or Button	Description
User Parameters group box **(for physisorption)**	These fields are primarily used for the SPC (Statistical Process Control) reporting to specify sample characteristics or its manufacturing process but may be used for other data by entering specific analysis conditions or sample criteria. The entered parameters display on the *Summary Report*. This option may not display (or may have a different field label) if modified in the method from which the sample file was created, either through ***Options > Default.Method*** or ***File > Open > Method***.

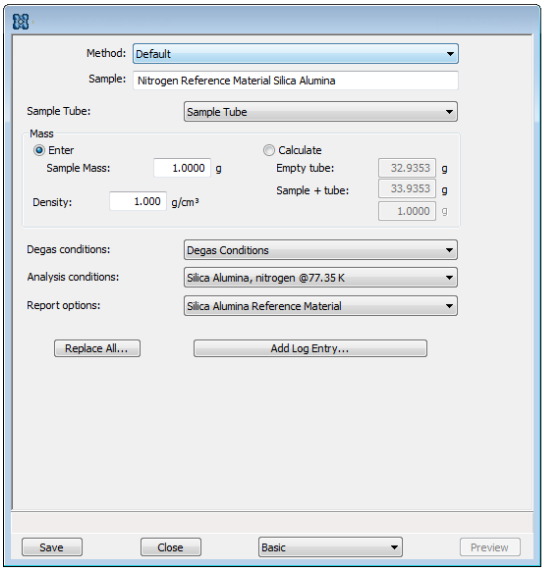
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

CREATE SAMPLE FILES USING BASIC PRESENTATION OPTION

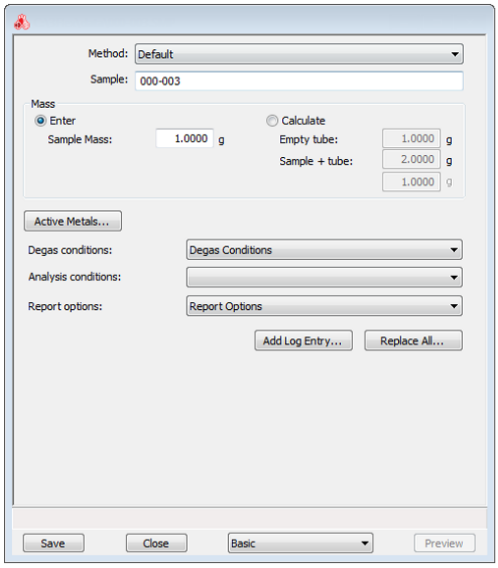
The *Basic* and *Restricted* formats use predefined parameter files to create a sample information file.

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

1. Go to ***Options > Option Presentation > Basic*** (or ***Restricted***).
2. Go to ***File > Open***.
3. Enter a new file name in the *File name* text box. Click **Open**.
4. Select the type of sample file.
5. Select a method from the *Method* drop-down list. See *"About Methods" on page 1 - 13*.

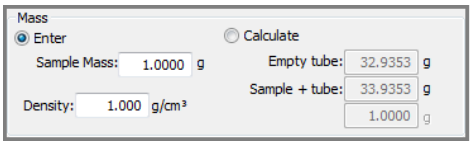


Physisorption

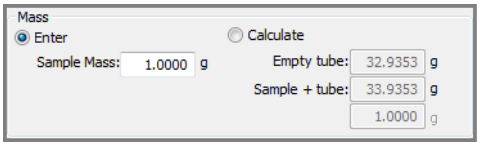


Chemisorption

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



Physisorption



Chemisorption

If mass = 1, the reported surface area equals the total surface area but it is always shown as m^2/g. If the actual mass is entered, the surface area is reported as m^2/g. Choose whether to enter mass manually or have the system automatically calculate mass. Enter a value for sample mass. Mass can be changed any time before, during, or after analysis.

- **Enter.** Enables the *Sample Mass* field. Enter a value for the sample mass.
- **Calculate.** Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass:

$$Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$$

- **Density**. (for physisorption.) Value is used for the calculated free space method only. Use 0.000 for a blank analysis.

9. For chemisorption: Click **Active Metals** to display and modify the characteristics of up to 15 elements used for analysis. Click **OK** when done. See *"Active Metals" on the next page*
10. 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***.
11. 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**.
12. 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.
13. Click **Save**, then click **Close**.The file can be retrieved later from the *Sample Information* folder in the library.

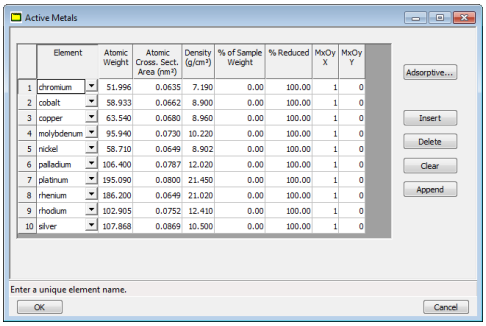
CREATE SAMPLE FILES USING THE RESTRICTED PRESENTATION OPTION

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

ACTIVE METALS

Chemisorption

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



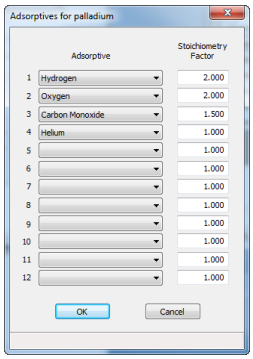
To enter a metal not shown in the default list:

1. Highlight an existing *Element* field. Alternatively, click either **Insert** or **Append**, then enter the new metal.
2. Click the fields to the right of the element and make the necessary modifications.
3. Click **OK** when done.

Active Metals Fields and Buttons Table

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

Active Metals Fields and Buttons Table (continued)

Field or Button	Description
Adsorptive	Click to display and modify both the adsorptive and Stoichiometry for the selected element.. .  • **Stoichiometry Factor.**A factor which expresses the ratio between the number of active metal molecules and the number of adsorbate molecules.
Atomic Cross Sect. Area (mn^2)	Atomic cross-sectional area of the element.
Atomic Weight	Atomic weight of the element.
Density	Density of the element.
Element	Select or enter the active metal.
MxOy, X	Number of metal atoms in the oxide.
MxOy, Y	Number of oxygen atoms in the oxide.

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

Atomic Weights and Cross-sectional Areas for Selected Metals

Metal	Symbol	Atomic Weight (g/mole)	Cross-sectional Area (sq nm)	Density (g/mL)
chromium	Cr	51.996	0.0635	7.19
cobalt	Co	58.933	0.0662	8.9
copper	Cu	63.54	0.0680	8.96
gold	Au	196.967	0.08696	18.9
hafnium	Hf	178.490	0.0862	13.3
iridium	Ir	192.220	0.0769	22.4

Atomic Weights and Cross-sectional Areas for Selected Metals (continued)

Metal	Symbol	Atomic Weight (g/mole)	Cross-sectional Area (sq nm)	Density (g/mL)
iron	Fe	55.847	0.0613	7.89
manganese	Mn	54.938	0.0714	7.43
molybdnum	Mo	95.940	0.0730	10.22
nickel	Ni	58.710	0.0649	8.9
niobium	Nb	92.906	0.0806	8.57
osmium	Os	190.220	0.0629	22.6
palladium	Pd	106.400	0.0787	12.02
platinum	Pt	195.090	0.0800	21.45
rhenium	Re	186.2	0.0649	21.02
rhodium	Rh	102.905	0.0752	12.1
tuthenium	Ru	101.070	0.0613	12.4
silver	Ag	107.868	0.0869	10.5
tantalum	Ta	180.947	0.0800	16.6
thorium	Th	232.038	0.1350	11.7
tin	Sn	118.710	0.1082	4.54
tungsten	W	183.850	0.0741	19.3
vanadium	V	50.942	0.0680	6.11
zirconium	Zr	91.220	0.0877	6.51

4 Create Sample Files for the AutoPore

Create Sample Files using Advanced Presentation Option

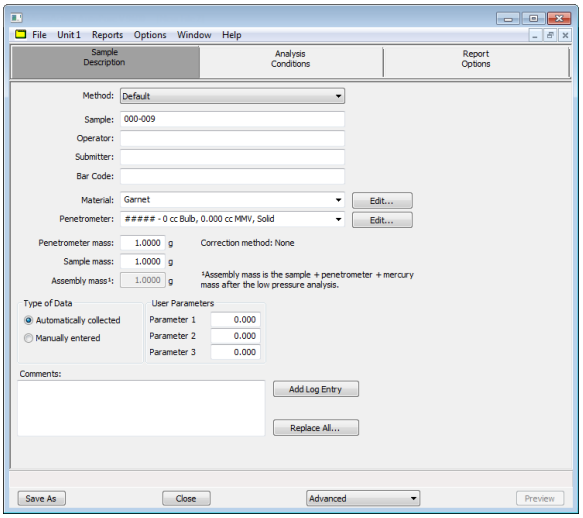
Each analysis must be linked with a sample information file before the analysis can proceed. A sample information file can consist of parameter files; however, parameter files can also stand alone.

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

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

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

1. Go to ***Options > Option Presentation > Advanced*** and ensure *Advanced* has a checkmark.
2. Go to ***File > Open***.
3. Enter a new file name in the *File name* text box. Click **Open**.
4. Select the type of sample file.
5. Select a method from the *Method* drop-down list.



6. Select a method from the *Method* drop-down list.

7. Enter a sample description in the *Sample* text box.
8. Enter *Operator*, *Submitter*, and *Bar Code* identification information in the respective text boxes. This information will display on the *Sample Description* tab of new sample information files. This option may not display (or may have a different field label) if modified in the method from which the sample file was created, either through ***Options > Default.Method*** or ***File > Open > Method***.
9. To select the sample material, click the *Material* drop-down arrow. Alternatively, click **Browse** and locate the file. See *"Material Properties" on page 6 - 10*.
10. In the *Penetrometer* drop-down list, select a penetrometer. If the required penetrometer does not appear in the list, click **Edit** and enter the description and other parameters for this tube. Then go to ***File > Save As > Penetrometer File*** to save these values for the next time this penetrometer is used.
11. The *Penetrometer Mass* field auto-populates with the value from the selected *Penetrometer Properties* file. If the value shown is not correct, enter the correct value. See .*"Penetrometer Properties" on page 6 - 12*
12. Enter the *Sample Mass*. The *Assembly Mass* entry is disabled until a low pressure analysis has been completed on the sample.
13. 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.
    - **Automatically collected.** Select for all sample runs where the data are collected.
    - **Manually entered**. Select when another sample has been run on a different analyzer or different model analyzer so that data can be analyzed or used for comparison. If *Manually entered* is selected, the data are entered in the Isotherm interactive report. See *"Manually Enter Data" on page 2 - 3*.
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. Enter any pertinent information about the sample information file in the *Comments* text box. Entered comments are displayed in the report header.
13. Click **Add Log Entry** to enter notes for the analyzer log report. Create entries that cannot be recorded automatically through the software.
14. 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 **Replace**.
15. After completing the *Sample Description* tab options, click the other parameter tabs to edit more sample information file parameters.
16. Click **Save As** to save as a different file name.The file can also be saved as a different file type to save part of the sample file as a new parameter set such as Analysis Conditions, Report Options, etc.

Sample File Fields and Buttons Table

Field or Button	Description
Assembly mass	This field is ntered after the low pressure analysis is completed.. Sample + penetrometer + mercury mass after the low pressure analysis.
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Material	Select the material to be analyzed from the drop-down list. See *"Material Properties" on page 6 - 10*.
Method	Select a method from the drop-down list. See *"About Methods" on page 1 - 13*.
Operator	Enter operator identification information. This field label may have been renamed or may not display if modified in ***Options > Default Methods.***
Penetrometer	Select a penetrometer file from the drop-down list, or click **Edit** to modify or create a new Penetrometer file. See *"Penetrometer Properties" on page 6 - 12*.
Sample	Enter a sample description.
Submitter	Enter submitter identification information. This text box may have been renamed or may not display if modified in ***Options > Default Methods.***
Type of Data group box	**Automatically collected.** Select if the type of data will be automatically collected by the system while an analysis is running.**Manually entered.** Use to enter data manually that was collected from another source. If *Manually entered* is selected, the Isotherm Report becomes available in the *Basic/Advanced* drop-down list for pasting or importing data into the file.See *"Manually Enter Data" on page 2 - 3*.
User Parameters group box	These fields are primarily used for the SPC (Statistical Process Control) reporting to specify sample characteristics or its manufacturing process but may be used for other data by entering specific analysis conditions or sample criteria. The entered parameters display on the *Summary Report*. This option may not display (or may have a different field label) if modified in the method from which the sample file was created, either through ***Options > Default.Method*** or ***File > Open > Method***.

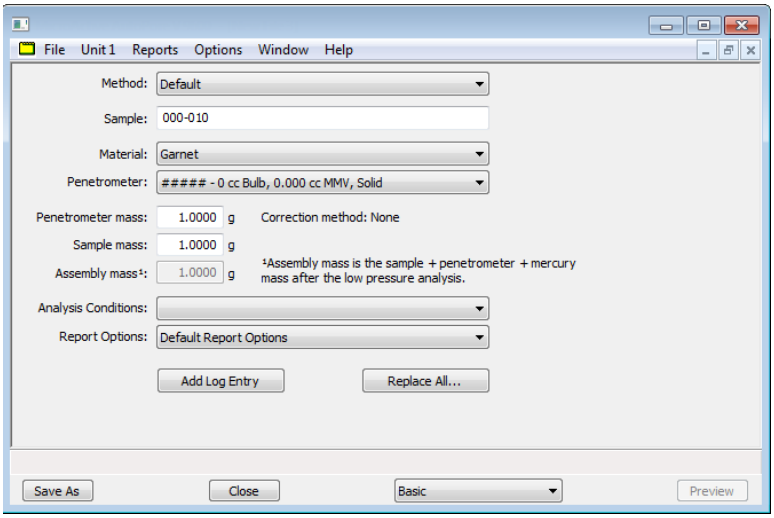
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

CREATE SAMPLE FILES USING BASIC PRESENTATION OPTION

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

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

1. Go to ***Options > Option Presentation > Basic*** (or ***Restricted***).
2. Go to ***File > Open***.
3. Enter a new file name in the *File name* text box. Click **Open**.
4. Select the type of sample file.



5. Select a method from the *Method* drop-down list. See *"About Methods" on page 1 - 13*.
6. In the *Sample* field, enter a sample description.
7. Select a sample material from the *Material* drop-down list. Alternatively, click **Browse** and locate the file. See *"Material Properties" on page 6 - 10*.
8. Select a penetrometer from the *Penetrometer* drop-down list. Alternatively, click **Browse** and locate the file. See *"Penetrometer Properties" on page 6 - 12*.
9. The *Penetrometer Mass* field auto-populates with the value from the selected *Penetrometer Properties* file. If the value shown is not correct, enter the correct value. See .*"Penetrometer Properties" on page 6 - 12*
10. Enter the *Sample Mass*. The *Assembly Mass* entry is disabled until a low pressure analysis has been completed on the sample.

11. Click the down arrows to select default parameter files for *Analysis conditions*, and *Report options*.
12. 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**.
13. 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.
14. Click **Save**, then click **Close**.The file can be retrieved later from the *Sample Information* folder in the library.

Sample File Fields and Buttons Table

Field or Button	Description
Assembly Mass	This field is calculated by the application: *Sample + penetrometer + mercury mass* after the low pressure analysis.
Material	Select the material to be analyzed from the drop-down list. See *"Material Properties" on page 6 - 10*.
Method	Select a method from the drop-down list. See *"About Methods" on page 1 - 13*.
Penetrometer	Select a penetrometer file from the drop-down list. See *"Penetrometer Properties" on page 6 - 12*.
Penetrometer Mass	The mass of the empty penetrometer.
Sample	Enter a sample description.
Sample mass	The mass of the sample.

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

CREATE SAMPLE FILES USING THE RESTRICTED PRESENTATION OPTION

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

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5 About Parameter Files for the 3Flex

Files created in the MicroActive application cannot be used in an analyzer for future analyses.

When viewing files from an analyzer, the screens are the same as the original application and may differ slightly from the screens in this operator manual.

Sample Tube

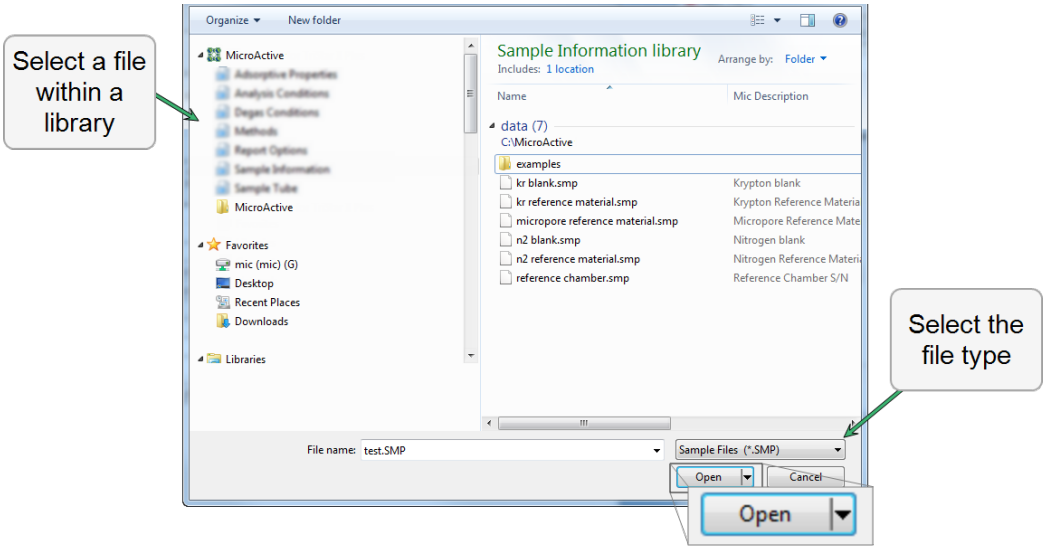
Physisorption

File > Open > [Sample Tube file]

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

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 3Flex. Filler rods help to ensure accuracy in samples with lower total surface areas by reducing the free-space volume. It is generally a good practice to use filler rods for samples having less than 100 square meters of total surface area. Filler rods are unnecessary for samples with total surface areas greater than 100 square meters.
5. Select the vacuum seal type to be used.
6. Click **Save**, then click **Close**.

Sample Tube Fields and Buttons Table

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

DEGAS CONDITIONS

Physisorption

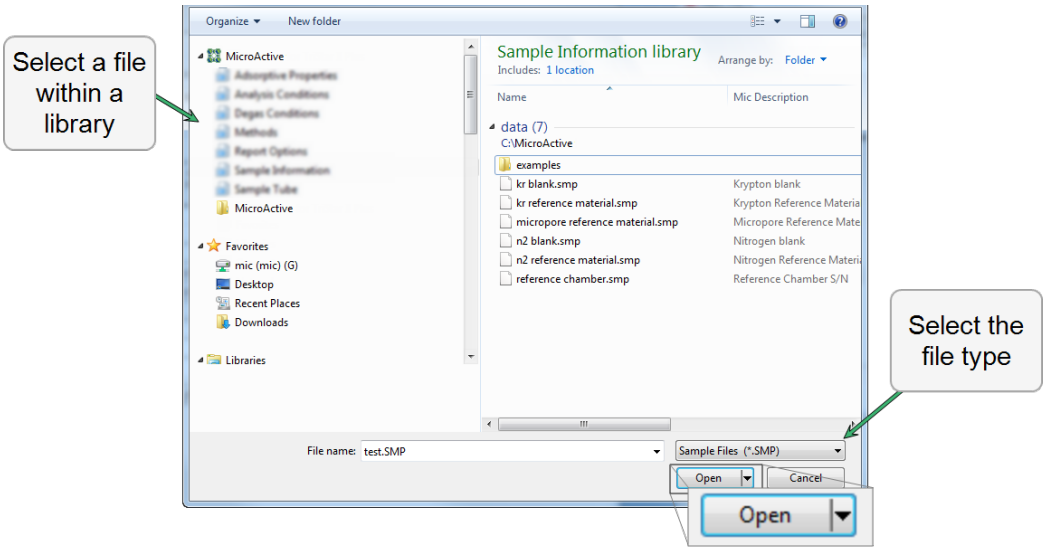
File > Open > [Degas Conditions file]

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

If using the Smart VacPrep, reference the Smart VacPrep Operator Manual (part number 067-42801-01) for additional information. A PDF link to the Smart VacPrep Operator Manual is also available in the Online Help page of this operator manual.

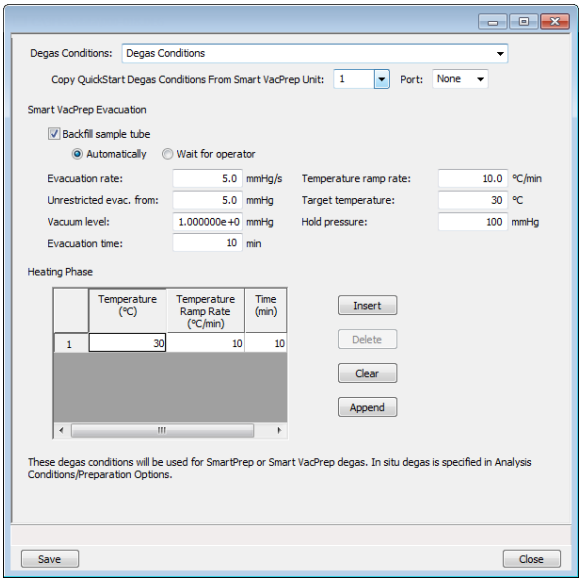
Degassing is a required step in preparation for an analysis. The *Degas Conditions* tab provides settings that will be automatically applied during the degassing procedure when using the Smart VacPrep.

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



2. To overwrite degas conditions with parameters from another *Degas Conditions* file, click the *Degas Conditions* down arrow, then select a file from the list. Alternatively, click **Browse** and locate the file.

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



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

Degas Conditions Fields and Buttons Table

Field or Button	Description
Degas Conditions	Use to browse for a .DEG file that contains degas condition parameters to be used in the analysis.
Heating Phase	This option is applicable when degassing with either a Smart VacPrep or a SmartPrep. Enter up to five stages of degas conditions. • **Temperature.** Degas temperature. • **Temperature Ramp Rate.** Rate at which the temperature is to change. • **Time.** Amount of time to heat the sample.

Degas Conditions Fields and Buttons Table (continued)

Field or Button	Description
	Use to modify the table contents. • **Insert.** Inserts one row above the selected row. • **Delete.** Deletes the selected row. • **Clear.** Clears all table entries and displays only one default value. • **Append.** Inserts one row at the end of the table.
Smart VacPrep Evacuation	This option is applicable only when degassing with a Smart VacPrep. • **Backfill sample tube.** Indicate if the sample tube should be backfilled automatically or wait for operator response. • **Evacuation Rate.** Rate used for evacuation. • **Unrestricted evac. from.** Pressure at which the unrestricted evacuation is to begin. • **Vacuum level.** Pressure for unrestricted evacuation. • **Evacuation time.** Length of time for preliminary evacuation before proceeding with the *Heating Phase* temperature schedule. The timer starts when the vacuum level is reached. • **Temperature ramp rate.** Rate at which the temperature is to change when advancing to the target pressure. • **Target temperature.** Targeted pressure for evacuation. • **Hold pressure.** Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold* pressure. This prevents damage to the sample structure due to 'steaming' and /or elutriation due to excessive escaping gas velocity.

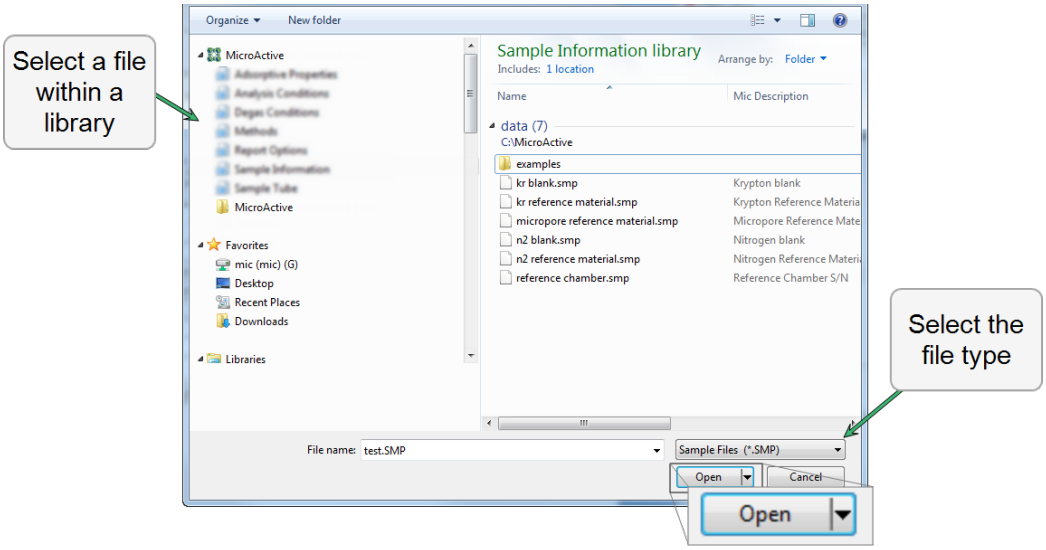
ANALYSIS CONDITIONS

File > Open > [Analysis Conditions file]

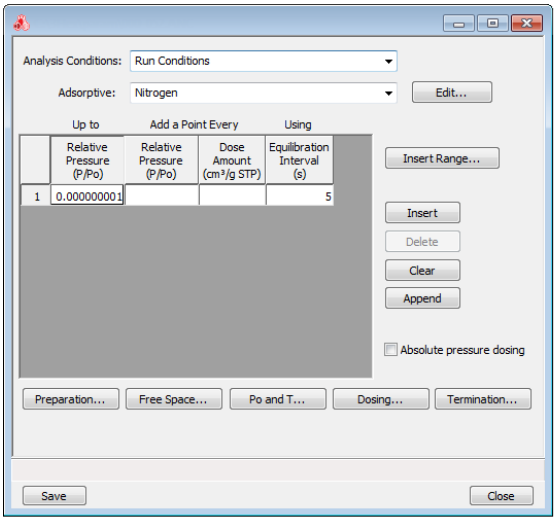
(or click the *Analysis Conditions* tab when in *Advanced* presentation display)

Analysis conditions specify the data used to guide an analysis.

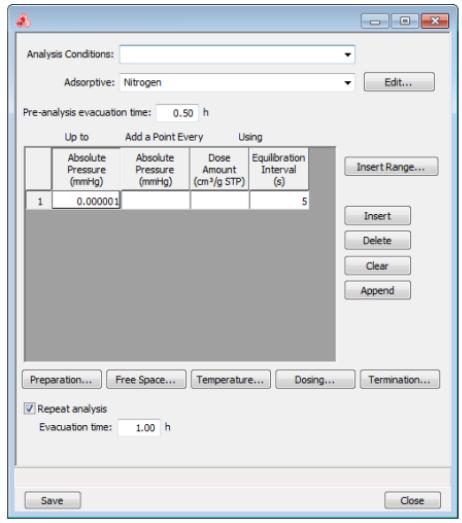
1. Go to ***File > Open***.
   - Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
   - Select the appropriate file type from the drop-down list on the lower right portion of the window,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



Physisorption



Chemisorption

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 5 - 14* to create a new *Adsorptive Properties* parameter file.
4. Click **Insert Range** to enter starting and ending relative pressure points.
5. Enable *Absolute pressure dosing* to specify pressure targets in mmHg, mbar, or kPa instead of relative pressure. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, H_2 adsorption on carbon at liquid nitrogen temperature.
6. Use the following buttons to specify:

Analysis Conditions Buttons Table

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

Analysis Conditions Buttons Table (continued)

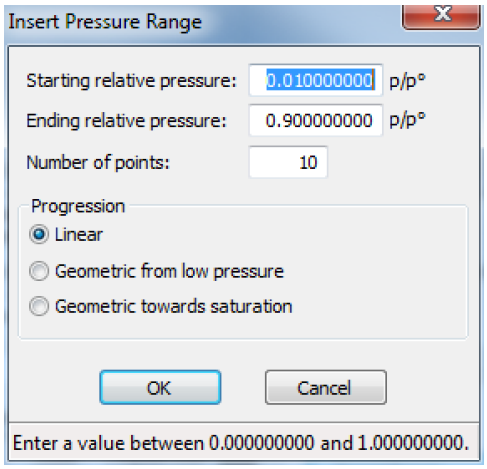
Button	Use to Specify...
Termination (for chemisorption)	Backfill o ptions after analysis.

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

Analysis Conditions Fields and Buttons Table

Field or Button	Description
Absolute pressure dosing (for physisorption)	Select to specify pressure targets in mmHg, mbar, or kPa instead of relative pressure. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, H_2 adsorption on carbon at liquid nitrogen temperature. If this option is selected, the *Relative Pressure* labels and entries change to *Absolute Pressure* in the selected pressure units.
Adsorptive	Select an *Adsorptive Properties* file from the drop-down list. See *"Adsorptive Properties" on page 5 - 14*.
Analysis Conditions	Use to browse for a .ANC file that contains analysis condition parameters to be used in the analysis.

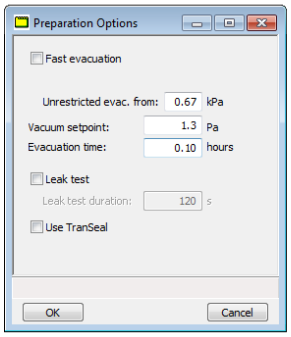
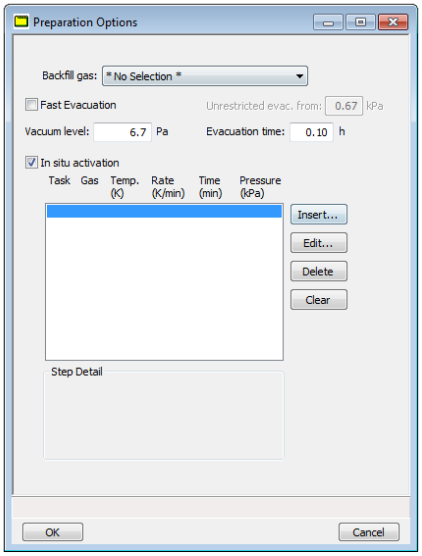
Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
Insert Range	Click to display the *Insert Pressure Range* window for entering parameters for the system to autofill the *Up to* column with starting pressure, ending pressure, the number of points to insert within the specified range and whether to have linear or geometric progression.  • **Starting relative pressure.** Enter the relative pressure at which data points will start to be taken. • **Ending relative pressure.** Enter the relative pressure at which data points will no longer be taken. • **Number of points.** Enter the number of points to be taken between the specified starting and ending relative pressures. *Progression* group box - • **Linear.** Use to insert evenly spaced points into the table. • **Geometric from low pressure.** The added pressure points will be spaced farther apart as the pressures get higher. • **Geometric towards saturation.** The added pressure points will be spaced closer together as the pressures get higher (closer to saturation).

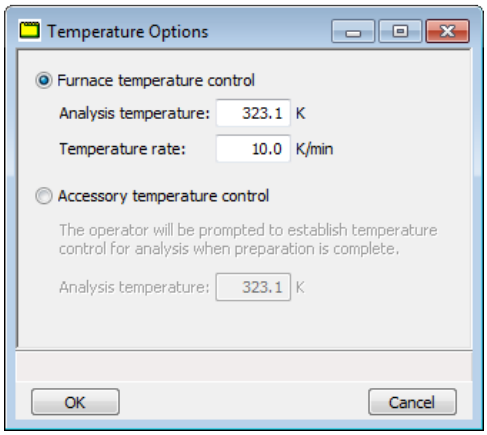
Analysis Conditions Fields and Buttons Table (continued)

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

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
Preparation (for 2020 ASAP Plus)	Use to enter analysis preparation details.  **Physisorption**  **Chemisorption** ***Physisorption*** • **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. • **Vacuum setpoint.** Specify the vacuum level to be achieved before time evacuation begins. • **Evacuation time.** Enter the length of time for preliminary evacuation, which takes place prior to the free-space measurement. • **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 using the TranSeal to transfer the sample from the preparation port to the analysis port under vacuum.

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	Chemisorption • **Backfill gas.** Lists the available backfill gases. • **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. • **Vacuum level.** The pressure for unrestricted evacuation. • **Evacuation time.** The length of time for preliminary evacuation. • **In situ activation.** When selected, preparation steps will be done. If not selected, the task table is disabled and analysis starts after the preliminary evacuation.
Repeat analysis (for chemisorption)	Select to repeat the analysis. The initial analysis measures chemisorption and physisorption activity. The repeat analysis measures only physisorption activity. The difference between the initial and repeat analysis is the strong chemisorption activity of the sample. The evacuation rate, unrestricted pressure, and setpoint are set using the **Preparation** button.
Temperature (for chemisorption)	 • **Furnace temperature control.** Enter the analysis temperature and temperature rate. • **Accessory temperature control.** *For user supplied temperature control only*. Enter the intended analysis temperature. The operator will be prompted to establish the analysis temperature before analysis begins.

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
Termination	Select if backfill is to be done after the analysis. Click the drop-down list to select the backfill gas to be used. **Cool to less than 50 °C**. Select to enable the cool down option.

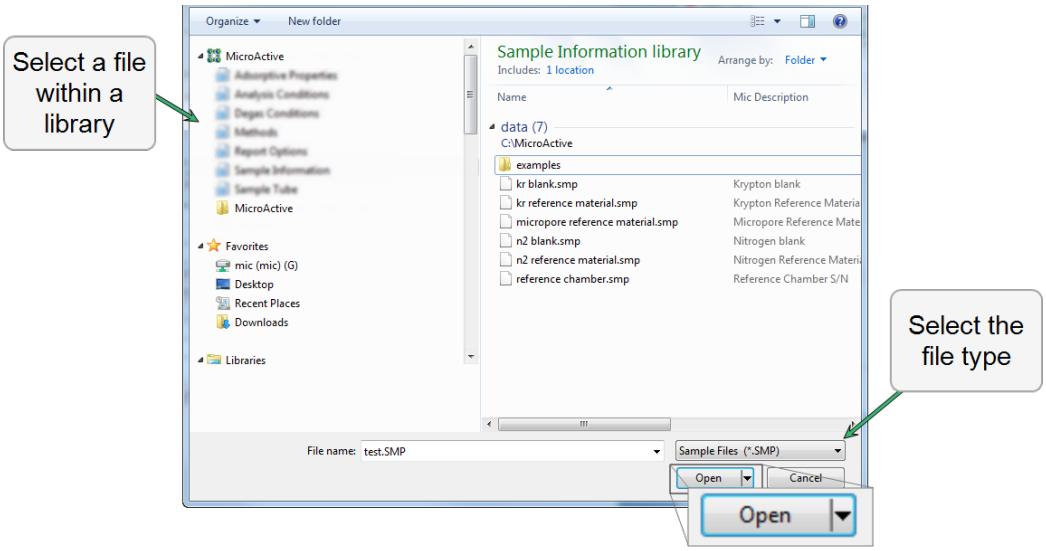
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

ADSORPTIVE PROPERTIES

File > Open > [Adsorptive Properties file]

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

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



2. Enter a description of the adsorptive in the *Adsorptive* text box (for example, the gas and the temperature). When saved, this description will display in the *Adsorptive* drop-down list of the *Analysis Conditions* window.
3. Enter the mnemonic for the adsorptive gas (for example, N2) in the *Mnemonic* text box. If this gas is connected to a gas inlet port, this mnemonic must be entered in the *Unit Configuration Gas Selection* for the inlet port.
4. Enter pressure, mass and diameter information in the text boxes.
5. For physisorption: Adsorbed molecules occupy volume in the sample tube, reducing the cold free space. Select the *Adsorbed-phase free-space correction* checkbox to adjust the reported quantity adsorbed to correct for this effect. This option is appropriate for all sample analyses that use the real gas equation of state.
6. To import parameters from a *Fluid Properties* file, click **Open**, locate and select the .FPI file containing the new parameters, then click **Open**. Click **Save** to save the changes. Changing fluid

properties should only be necessary if an adsorptive is to be used for which no adsorptive properties are provided or at an analysis temperature not covered by the standard properties. Contact Micromeritics Scientific Services if new fluid properties are required (http://tech-support.micromeritics.com/portal).

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

Adsorptive Properties Fields and Buttons Table

Field or Button	Description
Adsorbate molecular weight	The molecular mass is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot.
Adsorbed phase free-space correction	Adsorbed molecules occupy volume in the sample tube, reducing the cold free space. Select the *Adsorbed-phase free-space correction* checkbox to adjust the reported quantity adsorbed to correct for this effect. This option is appropriate for all sample analyses that use the real gas equation of state.
Adsorptive	Name of the adsorptive gas whose properties are being defined.

Adsorptive Properties Fields and Buttons Table (continued)

Field or Button	Description
Dosing Method	• **Normal.** Dose from a pressurized tank of gas attached to a gas inlet port. • **From Psat tube.** (for physisorption). Select if the Psat tube is to be filled with condensed adsorptive and dosed from the Psat tube. Select this option if using Krypton. • **From sample port 3** (for physisorption) Select if the tube attached to sample port 3 is to be filled with condensed adsorptive and dosed from port 3. • **Vapor source.** Select if a container of condensed vapor is to be attached to the Psat port in place of the Psat tube and is dosed from the Psat port. • **Charge from inlet.** (for physisorption) Use to have the tube automatically charged with condensate from a gas inlet port after the dewar is raised. • **Purify adsorptive.** (for physisorption) Use to have the condensate in the tube purified after charging by evacuating the gas over the condensate. If *Charge from inlet* is selected, select *Purify adsorptive* to have noncondensing contaminants automatically removed from the dosing tube prior to analysis. After the adsorptive has condensed in the selected Psat tube or port 3, the remaining gas in the tube will be evacuated to remove noncondensing contaminants. A small amount of the purified adsorptive condensate will then return to gas phase to restore equilibrium pressure in the tube.
Fluid properties	Use to import parameters from a *Fluid Properties* file. Click **Open** to browse and select a .FPI file. Locate and select the file, then click **Open** on the file selector window. Click **Save** to save the changes made from the importing selected the .FPI file. Changing fluid properties should only be necessary if an adsorptive is to be used for which no adsorptive properties are provided.
Mass flow constant	Scaling factor for the Mass Flow Controller measured flow rate. Applicable only for the gas used in the flow prep tasks. The default is preset for gases provided with the application. .
Maximum manifold pressure	The highest pressure to which the manifold will be dosed. To avoid damage to the analyzer, this number is limited to 925 mmHg. Low pressure sources, such as vapors, will require lower numbers. For gases to be used for dosing after changing a tube from the gas inlet, enter the maximum pressure for dosing from the inlet, not from the tube of condensate.

Adsorptive Properties Fields and Buttons Table (continued)

Field or Button	Description
Mnemonic	Enter the mnemonic name for the adsorptive. If this gas is connected to a gas inlet port, this mnemonic must be entered in the *Unit Configuration Gas Selection* for the inlet port. .
Molecular cross-sectional area	The area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations.
Therm. tran. hard-sphere diameter	An estimate of molecular size used in calculating the thermal transpiration correction.
Vapor Source Temperature	Select if the vapor source temperature is to be controlled by the analyzer. This field is enabled only if *Vapor Source* is selected.

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

REPORT OPTIONS

File > Open > [Report Options file]

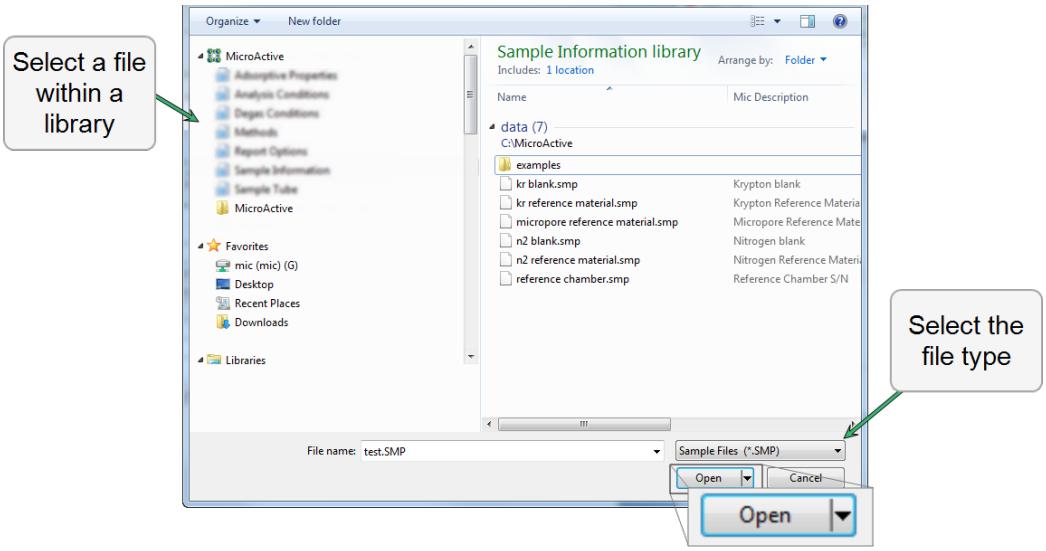
(or click the *Report Options* tab when in *Advanced* presentation)

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 and easy 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 *"Generate Multiple Graph 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	Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Always use thermal transpiration when performing micropore analysis. See *"Thermal Transpiration Correction" on page 12 - 47*. • **Inside diameter of sample tube.** Enabled when *Apply thermal transpiration correction* is selected. Enter the inside diameter of the sample tube used in the analysis. If filler rods are used, enter the filler rod capillary diameter of 1 mm instead.
Import (for physisorption)	Import up to 25 pore distribution data files. These datasets are shown only in BJH and Dollimore-Heal reports.
Name column	Displays a list of files in the selected directory.
Overlays	See *"Generate Multiple Graph 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. For BJH reports, BJH pore dimension can be calculated in pore width (w), pore radius (R) or pore diameter (D). Go to ***Options > Units*** to specify default calculations.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.

Report Options Fields and Buttons Table (continued)

Field or Button	Description
Show report title	Select and enter a report title to appear on the report header.

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

6 About Parameter Files for the AutoPore

Files created in the MicroActive application cannot be used in an analyzer for future analyses.

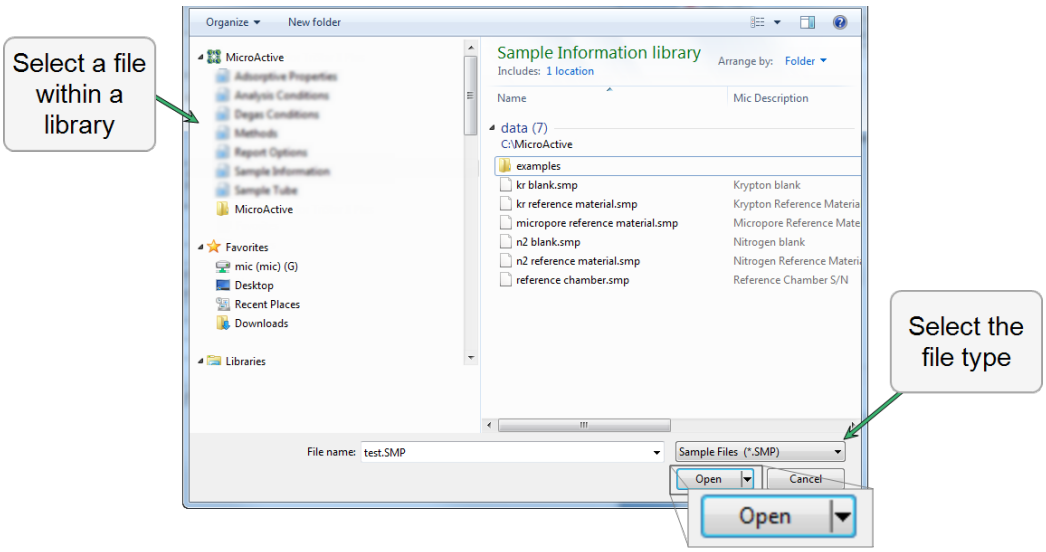
When viewing files from an analyzer, the screens are the same as the original application and may differ slightly from the screens in this operator manual.

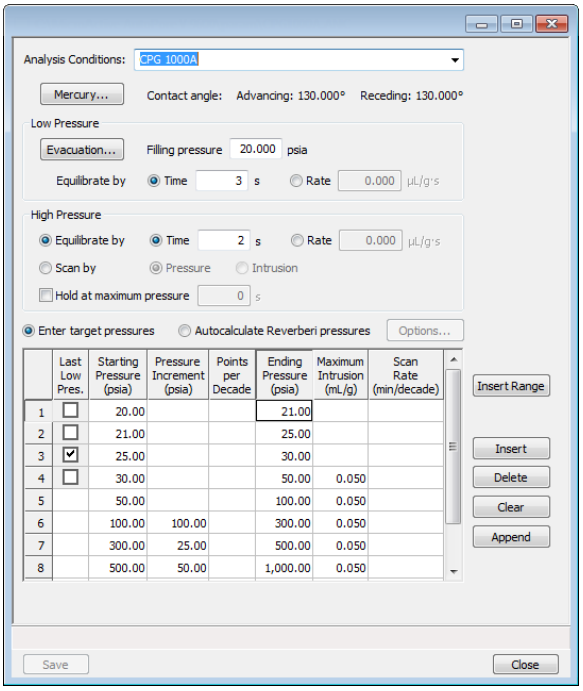
Analysis Conditions

File > Open > [Analysis Conditions file]

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.**





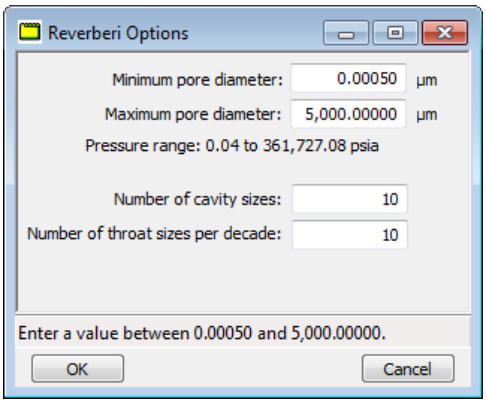
2. To overwrite analysis conditions with parameters from another *Analysis Conditions* file, click the *Analysis Conditions* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file.
3. To edit the *Contact angle* parameters, click **Mercury** to enter mercury properties.
4. In the *Low Pressure* group box, click **Evacuation** to set the set the low pressure evacuation options.
5. Indicate how the low pressure is to be equilibrated. Select and enter either *Time* or *Rate*.
6. In the *High Pressure* group box, indicate if the high pressure is to be equilibrated or scanned. If *Equilibrate by* is selected, enter the *Time* and *Rate*.
7. Select *Enter target pressures* to manually enter pressures into the table or select *Autocalculate Reverberi pressures* to have the application auto-calculate the pressures for Reverberi.
8. To manually add a sequence of pressures, enter the pressures in the *Ending Pressure* column.
9. Click **Insert Range** to enter starting and ending relative pressure points if entering target pressures manually.

   This consolidated pressure table allows specification of a series of linearly spaced or log spaced points to be specified on a single line.

If the *Last low Pressure* checkbox is selected, the *Ending Pressure* for this row is the *Last Low Pressure Point*. The *Last Low Pressure* column must have only one checkbox selected. Only rows before the first row with an *Ending Pressure* greater than 50 psia are eligible and will have a checkbox (meaning that the first *Ending Pressure* cannot be greater than 50 psia). *Ending Pressure* entries up to and including the row currently designated *Last low Pressure* are limited to a maximum pressure of 50 psia. If the *Last Low Pressure* row is deleted, the previous row is designated.

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

Analysis Conditions Fields and Buttons Table

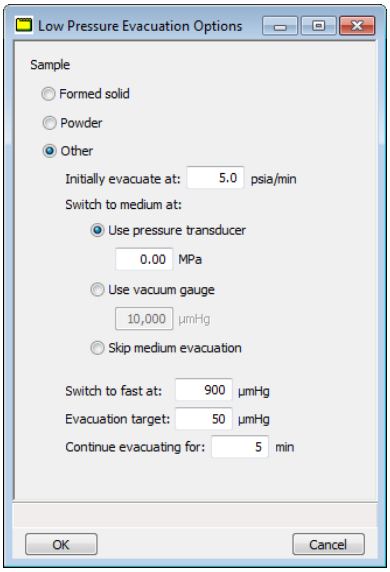
Button	Use to Specify...
Autocalculate Reverberi pressures	Select to have the application automatically use the Reverberi pressures. Selecting this option disables the pressures table. Click **Options** to enter additional Reverberi options. 
Enter target pressures	Select to manually enter pressures into the table.

Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
High Pressure group box	**Equilibrate by.** Select the option for equilibration based on elapsed *Time* (in seconds) or decrease in *Rate* of intrusion (or extension) in mL/g per second. **Scan by:** • **Pressure**. The instrument goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment ends at the specified pressure. The pressure is programmed to increase or decrease at a rate to give a constant time per decade of pressure. Along the way, the instrument takes intrusion points at the specified number of points per decade, ending at the specified ending pressure. Also, any points in the pressure table, as well as points separated by the maximum intrusion volume, are collected. • **Intrusion**. The instrument goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment ends at the specified pressure The pressure rate is the maximum achievable safe rate (up to 0.5 min/decade) and is programmed to increase or decrease at a rate to give a constant intrusion/extrusion rate. The instrument takes intrusion points at the specified number of points per decade, ending at the specified ending pressure. Also collected are points in the pressure table, as well as points separated by the maximum intrusion volume. If both high pressure ports are in use, they may have different intrusion rates. In this case, the left port (port 1) determines when data are collected. The right port collects data at the same times as the left. This allows a differential analysis to be performed in this mode. • **Hold at maximum pressure.** Enter additional amount of time to remain at the maximum pressure in a pressure table intrusion segment before beginning extrusion..

Analysis Conditions Fields and Buttons Table (continued)

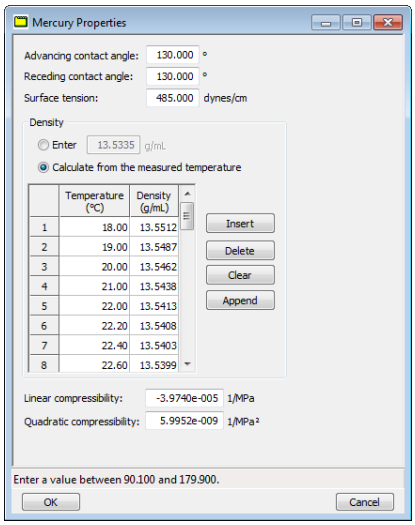
Button	Use to Specify...

Low Pressure group box	**Evacuation**. Select if the sample is a *Formed solid*, *Powder*, or *Other*. If *Other* is selected, the remaining fields are enabled.
	
	This window is also available from ***Unit [n] > Evacuate Low Pressure***.
	
	 
	
	• **Initially evacuate at**. Enter the initial maximum evacuation rate.
	• **Switch to medium at**. Enter the method and pressure the system must reach before medium evacuation begins.
	  ○ Use pressure transducer
	  ○ User vacuum gauge
	  ○ Skip medium evacuation
	• **Switch to fast at.** Enter the pressure the system must reach before fast evacuation begins.
	• **Evacuation target.** Enter the evacuation pressure.
	• **Continue evacuating for**. Enter the evacuation duration.
	
	Filling pressure. The penetrometer is filled with this pressure prior to data collection. It is recommended to set the filling pressure slightly lower than the first low pressure point on the pressure table.
	
	A filling pressure of at least 0.5 psia is recommended.

Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	Because mercury generates pressure and because fill pressures less than 0.5 psia can fail to fill the corner radii and gaps between the glass and sample in the penetrometer, using a lower pressures may reduce the accuracy of data. If the filling pressure is higher than any point in the table, an error message occurs. Delete the pressures lower than the filling pressure or change the filling pressure. **Equilibrate by.** Select the option for equilibration based on elapsed *Time* (in seconds) or decrease in *Rate* of intrusion (or extension) in mL/g per second.

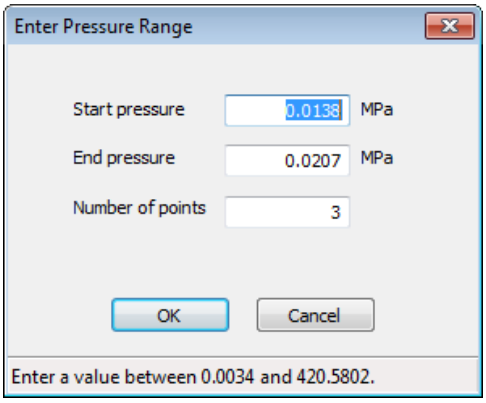
Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
Mercury	Enter the mercury properties. Mercury properties may change with variations in temperature.  **Advancing contact angle.** Enter the advancing (intrusion) contact angle. **Receding contact angle**. Enter the receding (extrusion) contact angle. **Surface tension**. Enter the surface tension of mercury. **Density.** • **Enter**. Select to manually enter a density. • **Calculate from the measured temperature**. Select to use the entries in the table. • **Linear compressibility**. Enter the linear compressibility coefficient. • **Quadratic compressibility**. Enter the quadratic compressibility coefficient.

Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
Table Options	**Last low pressure**. Select the rows to indicate the last low pressure. **Starting Pressure.** This column is not editable. The data are taken from the *Ending Pressure* of the preceding row and the *Filling Pressure* for the first row. **Pressure Increment**. Enter the pressure increment for this segment if a sequence of linearly spaced pressures is preferred. Either the *Pressure Increment* or *Points Per Decade* can be specified. If one is entered, the other is automatically set to zero and displayed as blank. By default, both columns are blank (zero) when a new row is inserted or appended. **Points per Decade.** Enter the number of points per decade for this segment if a sequence of logarithmically spaced pressures is preferred. Either the *Pressure Increment* or *Points Per Decade* (but not both) can be specified. If one is entered, the other is automatically set to zero and displayed as blank. By default, both columns are blank (zero) when a new row is inserted or appended. **Ending Pressure**. Enter the ending pressure for this segment. **Maximum Intrusion.** The instrument automatically takes additional readings between points on the pressure table when this volume of additional intrusion is detected. Enter the intrusion volume per gram of sample that must be reached in order for additional data pair readings to be recorded. Use 0 to prevent readings between pressure points. See *"Use of the Maximum Intrusion Volume Option" on page 16 - 1*. The *Maximum Intrusion* entry is set to the same value as the preceding row by default when a new row is inserted or appended. **Scan Rate**. If scanning by pressure, enter the minutes per decade for this segment. If scanning by intrusion, enter the intrusion rate for this segment. The *Scan Rate* column is set to blank (zero) and disabled for all rows up to and including the *Last Low Pressure*. If *Scan By Pressure* or *Intrusion* is selected, the column title will display the appropriate units (min/decade) or (mL/g-sec) and each high pressure row must contain a value. The value for the previous row is set by default when a new row is inserted or appended, or the default value (5 min/decade or 0.001 mL/g-sec) for the first high pressure row. **Insert Range**. Click to display the *Enter Pressure Range* window for entering parameters for the system to autofill the table with starting pressure, ending pressure, and the number of points to insert within the

Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	specified range. 

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

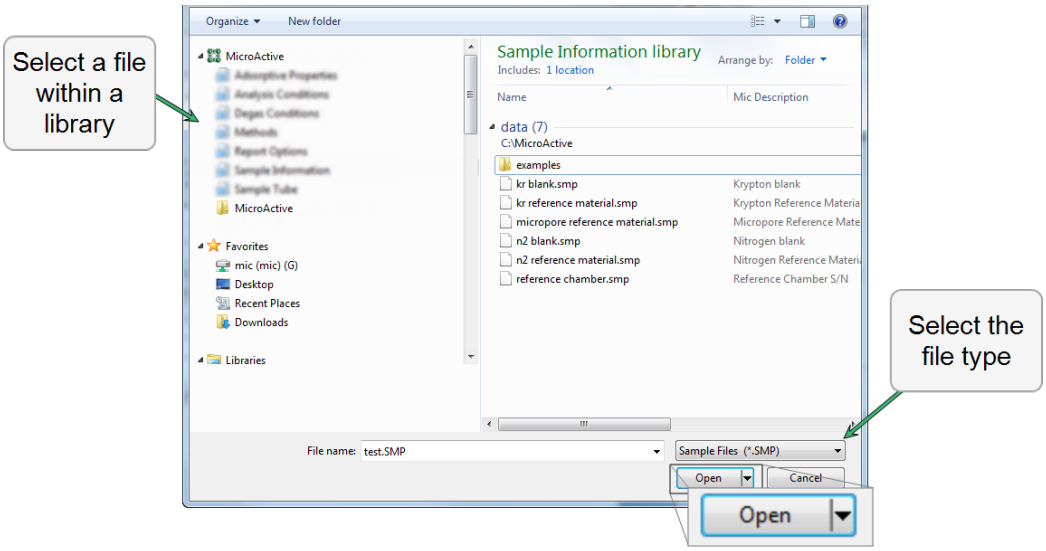
MATERIAL PROPERTIES

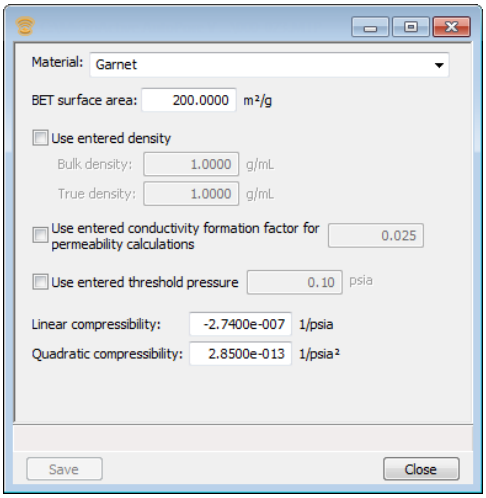
File > Open > [Material Properties file]

Material properties specify the properties of the material to be used in 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. In the *Material* drop down, select the material being analyzed. To overwrite material properties with parameters from another *Material Properties* file, click the *Material Properties* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file.
3. Enter the *BET surface area*.
4. If using the entered density, select the *Use entered density* checkbox and enter the bulk and / or true density in the appropriate text box.
5. If using the entered conductivity formation factor for permeability calculations, select the *Use entered conductivity formation factor for permeability calculations* check box and enter the factor in the text box.
6. If using the entered threshold pressure, select the *Use entered threshold pressure* checkbox and enter the pressure in the text box.
7. Enter the *Linear compressibility* and *Quadratic compressibility* pressures in the appropriate text boxes. These values can be copied from the *Compressibility Report* after a sample of the material has been analyzed.
8. Click **OK** to save the changes or click **Cancel** to close the window.

Material Properties Fields and Buttons Table

Field or Button	Description
BET Surface Area	Enter the BET surface area.
Linear compressibility	Enter the linear compressibility coefficient.
Quadratic compressibility	Enter the quadratic compressibility coefficient.
Receding contact angle	Enter the receding (extrusion) contact angle.
Surface tension	Enter the density of mercury being used.
Use entered conductivity formation factor...	Select if using the entered conductivity formation factor for permeability calculations and enter the factor in the text box.
Use entered density	Select if using the entered density and enter the bulk and /or true density in the appropriate text boxes.
Use entered threshold pressure	Select if using the entered threshold pressure and enter the threshold pressure in the text box.

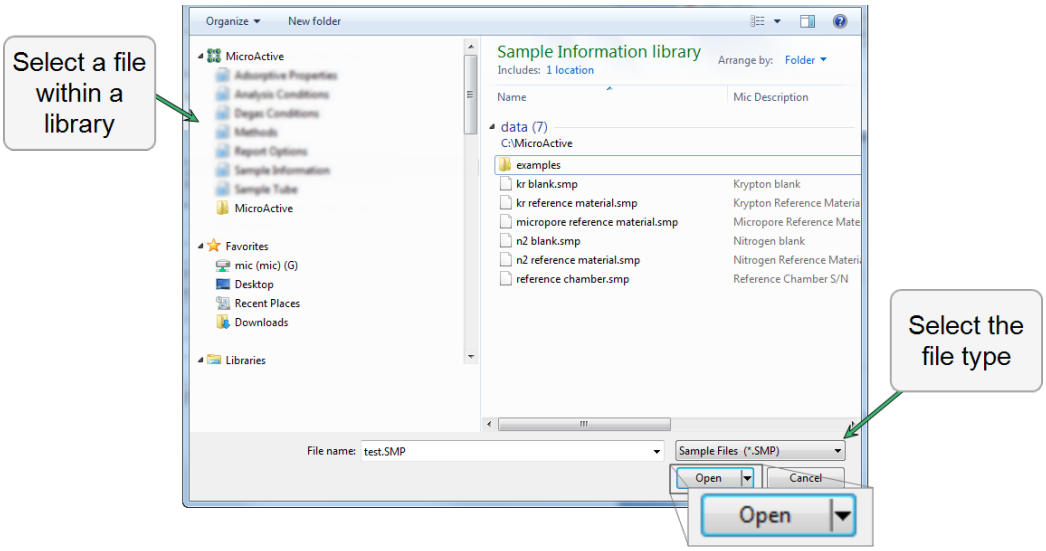
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

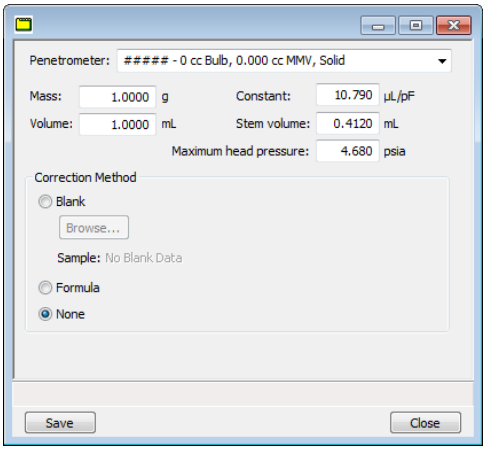
PENETROMETER PROPERTIES

File > Open > [Penetrometer file]

Penetrometer properties specify the properties of the penetrometer to be used in 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 penetrometer properties with parameters from another *Penetrometer Properties* file, click the *Penetrometer* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file. If this is a new penetrometer, enter the penetrometer description in the *Penetrometer* text box. It is recommended to use an identifier similar to:
   ##### *- 0 cc Buld, 0.000 cc MMV, solid* where ##### is the serial number etched on the penetrometer.
3. The *Mass*, *Volume*, *Constant*, and *Stem Volume* fields are filled if an existing penetrometers file is selected. If a new penetrometer information is being entered, complete these fields as they pertain to the new penetrometer.
4. In the *Correction Method* group box, select the appropriate correction method. If *Blank* if selected, click **Browse** and select a *Blank Correction* sample file.
5. Click **Save**, then click **Close**.

Penetrometer Properties Fields and Buttons Table

Field or Button	Description
Maximum head pressure	Enter the maximum head pressure.
Constant	Enter the penetrometer constant provided with the penetrometer. Verify the field contents if the **Replace** option has been used.
Correction Method	**Blank**. If using the blank correction method, click **Browse** to select a sample file. **Formula**. **None**.
Mass	Mass of the empty, assembled penetrometer (excluding the spacer).
Penetrometer	Enter identifying information for this file.
Stem volume	Enter the penetrometer stem volume provided with the penetrometer.
Volume	Enter the volume of the penetrometer. This is required to calculate density or when using the blank correction formula.

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

REPORT OPTIONS

File > Open > [Report Options file]

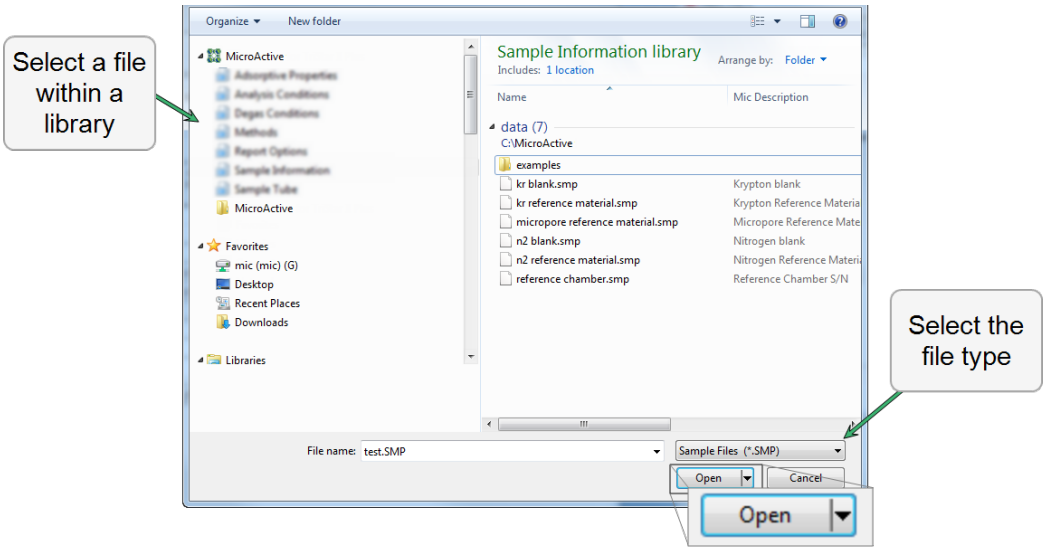
(or click the *Report Options* tab when in *Advanced* format)

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 user-defined report tables.

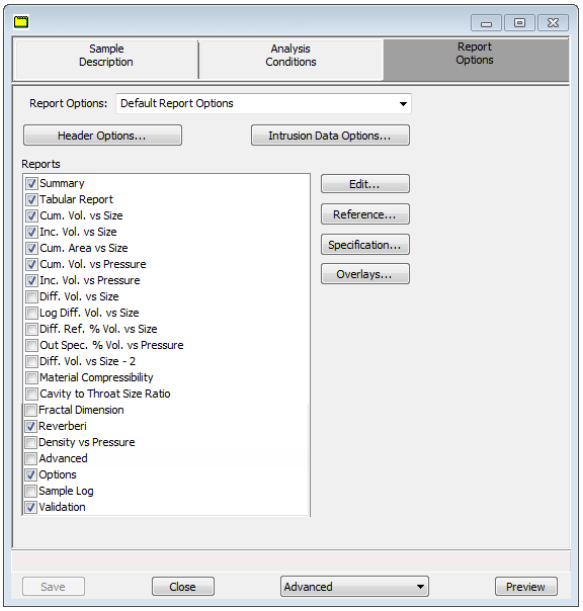
Customized report options files can be created then loaded into a sample file, allowing quick and easy generation of reports.

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 *"Generate Multiple Graph 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.



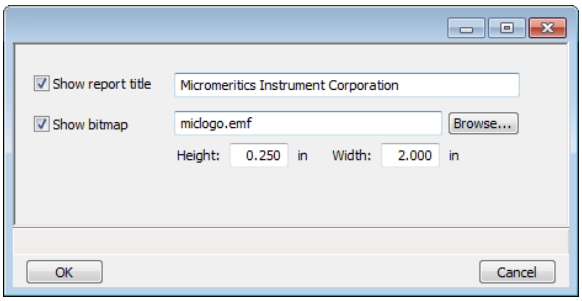
3. To have a report title display on the report header, select **Header Options**, then enter a title to appear on the report header.
4. To have a graphic display on the report header, select *Show graphic* to insert a graphic in the report header. Click **Browse** to locate a .BMP or a .EMF file. Specify 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**.

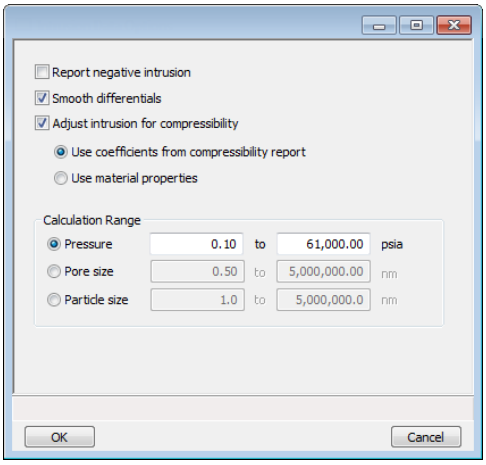
Report Options Fields and Buttons Table

Field or Button	Description
Edit	Click **Edit** to edit graph options. • **Plot Points.** Select to plot points on the graph.. • **Plot Curve.** Select to plot curves on the graph.. • **Show Histogram.** Select to show the graph as a histogram. When selected, the *Plot Points* and *Plot Curve* selections are disabled.

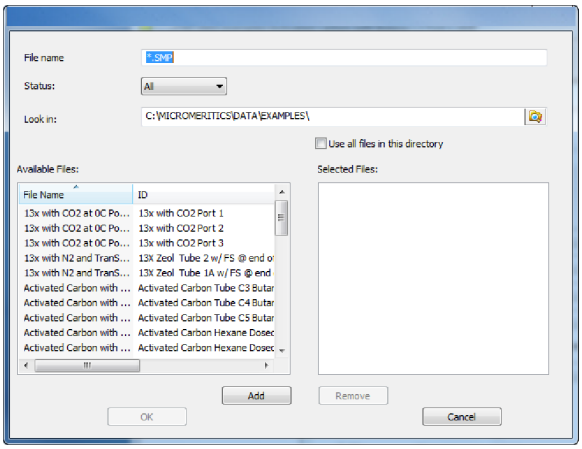
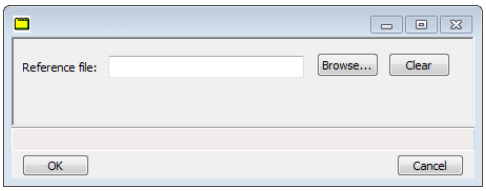
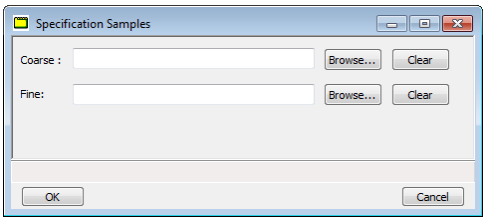
Report Options Fields and Buttons Table (continued)

Field or Button	Description
Header Options	 **Show report title**. Enter a report title to appear on the report header. **Show bitmap.** Displays the selected graphic on the report header. Click **Browse** to locate the graphic in either .BMP or .EMF format. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.

Report Options Fields and Buttons Table (continued)

Field or Button	Description
Intrusion Data Options	 **Report negative intrusion.** Select to report small incorrect polarities (negative intrusions or positive extrusions) which may indicate the presence of noise, improper blank correction, or instrument malfunction. **Smooth differentials.** Select to apply smoothing to any differentials reported in tables or graphs. **Use coefficients from compressibility report.** Select to have the application use the coefficients from the *Material Compressibility* report rather than from the *Material Properties*. **Use materials properties.** Select to have the application use the parameters from *Material Properties* rather than from the *Material Compressibility* report. **Calculation Range.** Select to indicate if reports should be limited in range by pore size, pressure, or particle size; then enter the range(s). The *from* value must be less than the *to* value.

Report Options Fields and Buttons Table (continued)

Field or Button	Description
Overlays	See *"Generate Multiple Graph Overlays" on page 7 - 29* 
Reference	Click **Reference** to select a sample file to compare analysis results with the current sample. 
Reports list box	Select the report names to include in the report.
Specification	Click **Specification** to select the sample files to be used for the boundaries of the coarse and fine specifications. This helps in determining if the results of the current sample are within the specified boundaries. 

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

7 About Reports

Review this section for information on the *Reports* menu options as well as customizing and running reports.

Reports can be generated for data:

- collected on a sample that has completed analysis
- manually entered

Open and Close Reports

Reports > Open Report... > [Report file]

Opens saved reports.

Reports > Close Reports

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

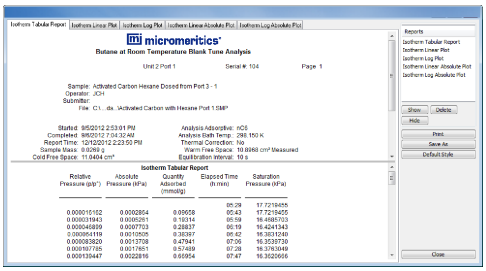
Start Reports

Reports > Start Report

1. Select one or more a .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 file was selected in Step 1, select *Preview* and click **OK** to display the *Select Reports* window. Verify the reports to generate and select additional reports if necessary, then click **OK**. If multiple files were selected, this window does not display.

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

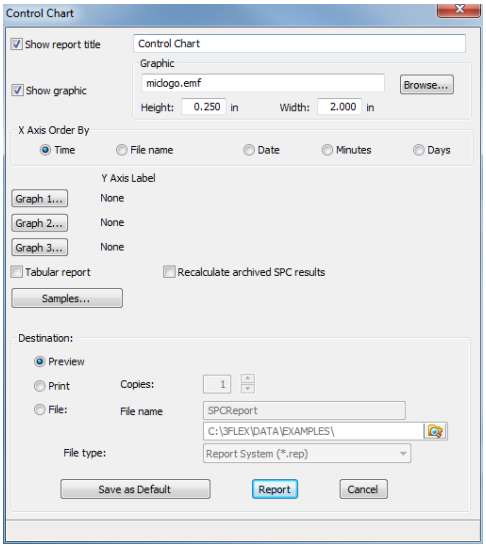


CONTROL CHART REPORT

Physisorption

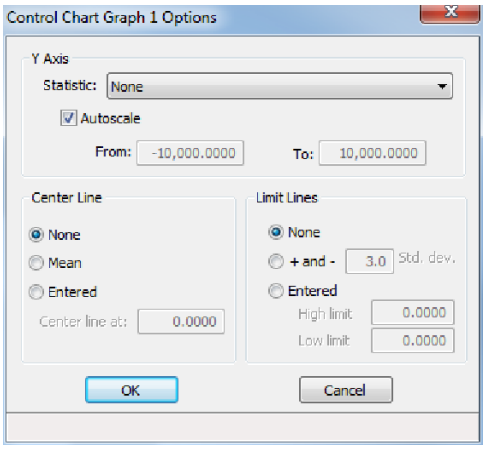
Reports > Gas Adsorption Control Chart

Reports > Mercury Intrusion Control Chart



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

Control Chart Fields and Buttons Table

Field or Button	Description
Graph [*n*]	Click to define the y-axis of each graph.  • **Statistic.** Displays the SPC variables selected on the ***Reports > SPC Report Options*** window. The selected variable will be plotted against time. This selection also becomes the y-axis label. • **Autoscale.** Allows the y-axis to be scaled automatically. To specify a range, deselect this option and enter a range in the *From* and *To* fields. • **Center Line.** Displays placement options for the center line in the graph. Choose *Entered* to specify placement of the line. • **Limit Lines group box.** Displays limiting lines options. Lines can be placed at some multiple of the standard deviation or at specified positions (*Entered*). When *Entered* is selected, enter the *High limit* and *Low limit* fields with appropriate values.

Control Chart Fields and Buttons Table (continued)

Field or Button	Description
Recalculate archived SPC results	Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report. If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application. When this option is selected, the following message displays: **Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.** **Do not show me this message again.** If *Do not show me this message again* is selected, the message cannot be redisplayed without Micromeritics assistance. The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Report	Generates the report.

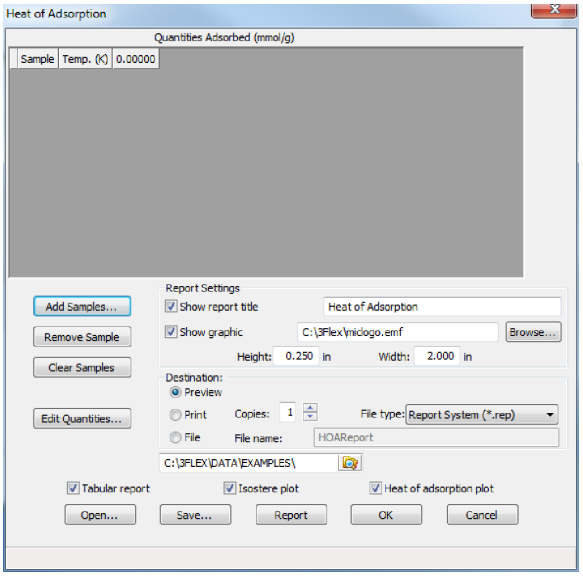
Control Chart Fields and Buttons Table (continued)

Field or Button	Description
Samples	To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Available Files.** Contains files located in the directory specified in the *Look In* text box. • **Selected Files.** Files added from the *Available Files* list box. • **Add / Remove.** Select a file in the *Available Files* list box, then click **Add** to move the file to the *Selected Files* list box. Or select a file in the *Selected Files* list box, then click **Remove** to move the file back to the *Available Files* list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X Axis Order by	Select the order in which x-axis statistics are placed. Sort by: • **Time.** Time the files were analyzed. • **File name.** Alphanumeric order. • **Date.** Date the files were analyzed. • **Minutes.** Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. • **Days.** Number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.

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

HEAT OF ADSORPTION REPORT

Reports > Heat of Adsorption

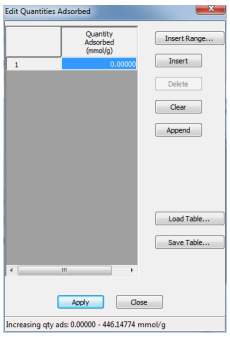


Use to select sample files, define quantities, and generate a *Heat of Adsorption* report. The isosteric heat of adsorption is an important parameter for characterizing the surface heterogeneity and for providing information about the adsorbent and the adsorption capacity. Multiple adsorption isotherms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

Heat of Adsorption Fields and Buttons Table

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

Heat of Adsorption Fields and Buttons Table (continued)

Field or Button	Description
Edit Quantities	Use to specify the range of surface coverage to include in the report.  **Insert Range.** Click to specify the starting and ending quantities adsorbed and number of points to insert. Use to modify the table contents. • **Insert.** Inserts one row above the selected row. • **Delete.** Deletes the selected row. • **Clear.** Clears all table entries and displays only one default value. • **Append.** Inserts one row at the end of the table. • **Load Table.** Imports values from another file. • **Save Table.** Saves the current table as a .QNT file. • **Apply.** Applies all table changes. • **Close.** Closes the table without saving changes.
Heat of adsorption plot	Generates the *Heat of Adsorption* data in a graphical format.
Isostere plot	Generates a graph showing quantities of gas adsorbed versus the temperature.
Remove Sample	Removes the selected sample from the list.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.

Heat of Adsorption Fields and Buttons Table (continued)

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

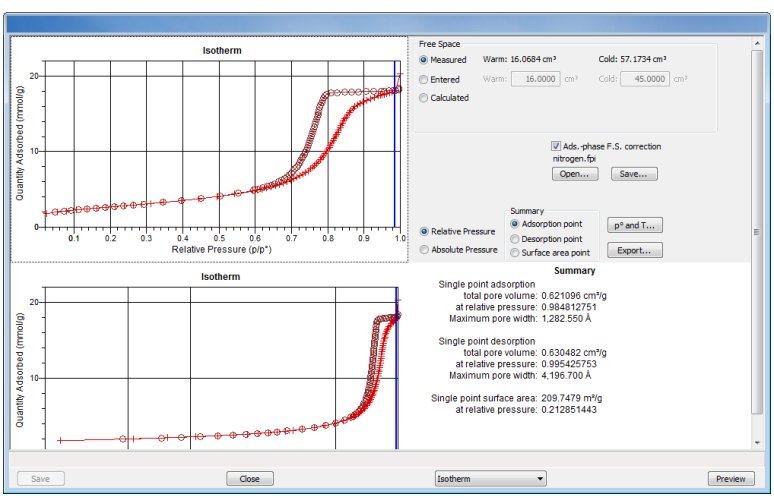
INTERACTIVE REPORTS

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

> When manipulating sample information and parameter file data in the MicroActive application, it is recommended that the data be manipulated in a file copy rather than the original file. Files that have been modified in the MicroActive program are not usable in the originating application.

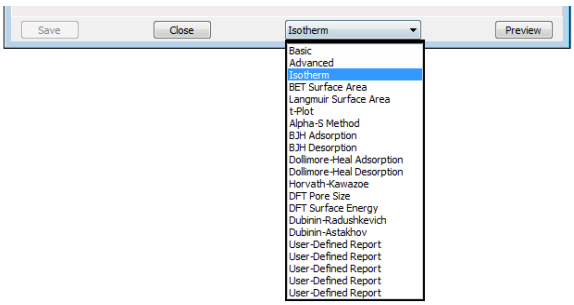
When importing a sample file that contains data from an analysis performed on a Micromeritics gas adsorption instrument, the interactive reporting feature is enabled. For a tutorial on using the interactive reports, go to ***Report Tutorials*** in online help.

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 presentation display 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.
   - Press **CTRL**, then left click on a data point in the Isotherm Linear Plot to include or omit the data point from the BET plot.
   - Right click to display a popup menu to include reports; enable or select overlays; edit curves, axes, legends, titles; and copy and paste the data in a graph or in tabular format.
6. After editing the report, click **Save** to save the changes in the sample information file.

MICROACTIVE REPORTS

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

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

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

Physisorption

Chemisorption

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.

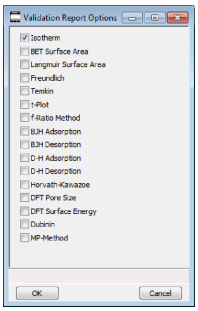
Regardless of the precautions exercised before the analysis, problems still may occur during the analysis, or as a result of using inappropriate parameters or even inappropriate methods. The analysis data should be inspected for evidence of experimental error. The traditional method of confirming the 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

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

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.

Evaluate the Isotherm Tabular Data Set

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

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

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

Review Reduced Data

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

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.

Physical Parameters

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

The area occupied by a single adsorbed molecule is a required parameter in the calculation of surface area by the BET and Langmuir methods. The software provides a default value, but other values are found in the literature. McClellan and Harnsberger[1)] provide a comprehensive review of such values.

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

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

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

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

using which, the Kelvin equation[1)] reduces to

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

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.

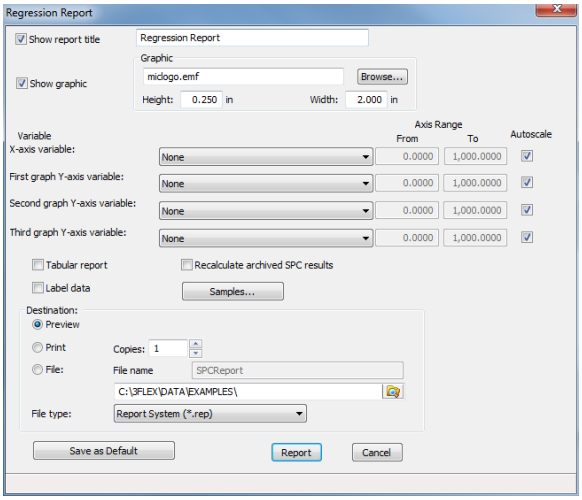
1) Thomson, W., Phil. Mag. S., 42, 448 (1871).

REGRESSION REPORT

Reports > Gas Adsorption Regression Report

Reports > Mercury Intrusion Regression Report

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



Regression Report Fields and Buttons Table

Field or Button	Description
Autoscale	When enabled, allows the x- and y-axes to be scaled automatically.
Axis Range	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if *Autoscale* is selected.
Label data	Use to label the points on the plot to correspond with the values in the sample files.

Regression Report Fields and Buttons Table (continued)

Field or Button	Description
Recalculate archived SPC results	Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report. If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application. When this option is selected, the following message displays: **Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.** **Do not show me this message again.** If *Do not show me this message again* is selected, the message cannot be redisplayed without Micromeritics assistance. The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.

Regression Report Fields and Buttons Table (continued)

Field or Button	Description
Samples	To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Available Files.** Contains files located in the directory specified in the *Look In* text box. • **Selected Files.** Files added from the *Available Files* list box. • **Add / Remove.** Select a file in the *Available Files* list box, then click **Add** to move the file to the *Selected Files* list box. Or select a file in the *Selected Files* list box, then click **Remove** to move the file back to the *Available Files* list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X- and Y-Axis Variable	Use to designate the x- and y-axes variables. The variables in the drop-down lists are those selected in the ***Reports > SPC Report Options*** window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.

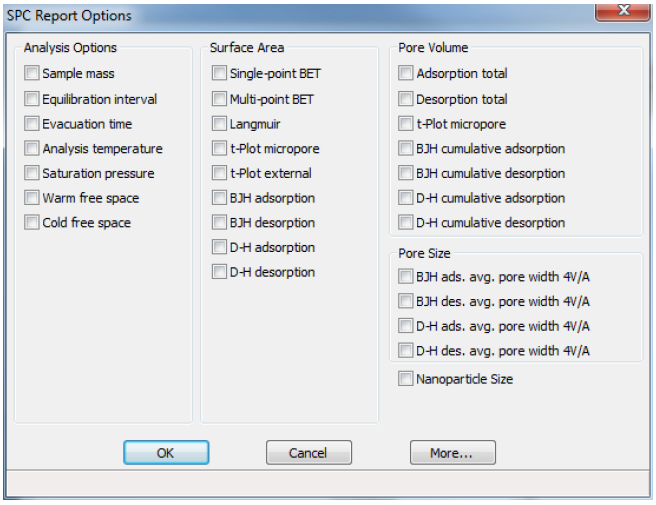
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

SPC REPORT

Physisorption

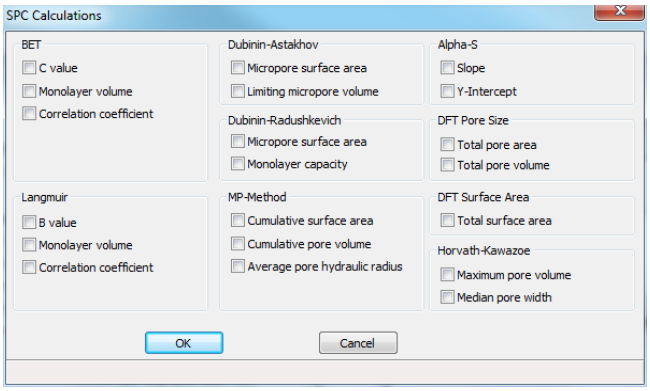
Use to generate reports with various *SPC* (Statistical Process Control) options. All selected variables must be computed for each sample file used in an SPC report; therefore, it is more efficient to select only the necessary variables.

Reports > SPC Report Options

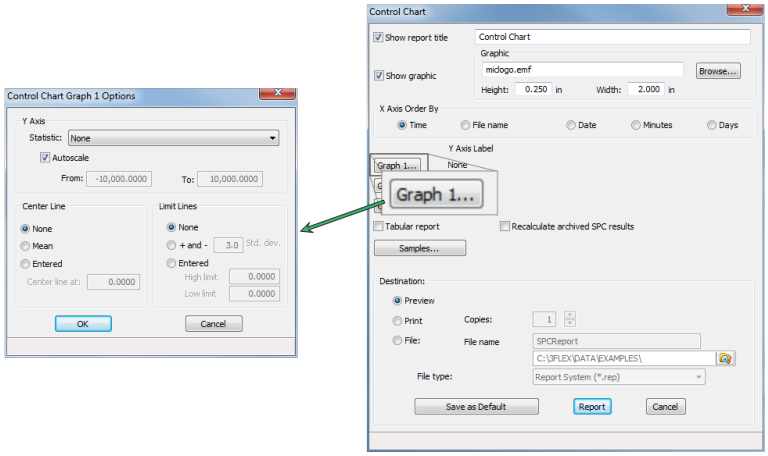


The selected items display as options on the ***Reports > Regression Report*** window as selections in the drop-down boxes and are used in graph selection in ***Reports > Control Chart***.

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



The selected items also display as options on the ***Reports > Control Chart*** window.

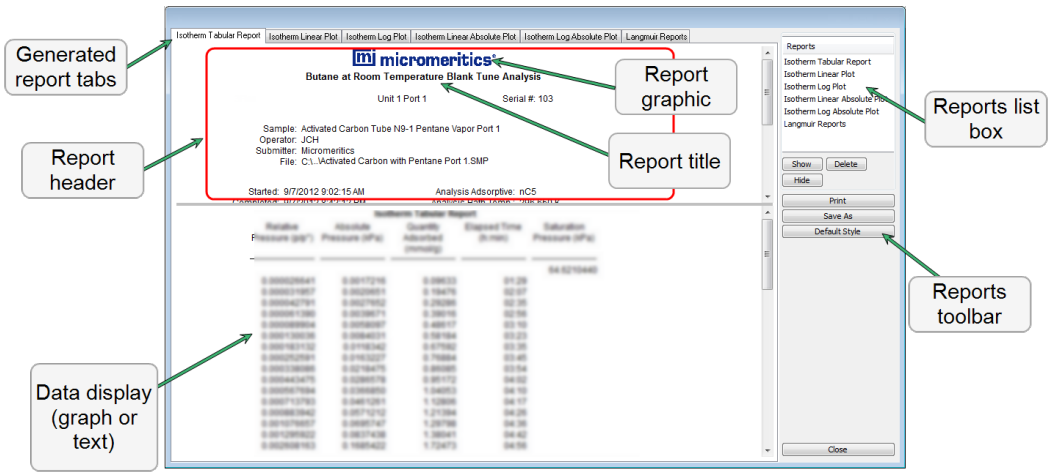


Click **Graph [*n*]**, then click the *Statistic* down arrow.

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.

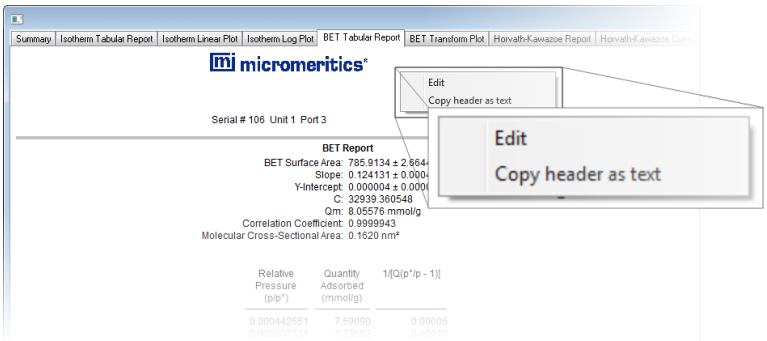


If configured, the report header can also contain a graphic and / or a title.

- 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 select the report in the *Reports* list box, then click **Show**.

Report Toolbar Fields and Buttons Table

Field or Button	Description
Default Style	Click to specify default report parameters for fonts and curve properties. • **Font Type.** Use to edit the font type and attributes for the selected item. Select an item in the list, click **Edit**, and select from various font options. • **Thickness**. Enter a thickness number for the curve. • **Histogram Fill Style.** Select a histogram fill option. • **Graph border line thickness.** Enter a thickness number for the graph border. • **OK**. Saves and closes the active window. • **Cancel**. Discards any changes or cancels the current process.
Delete	Deletes the selected report in the *Reports* list box. Deleted reports will have to be regenerated if deleted in error.
Hide	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the *Reports* list box. To redisplay the tab, select the report in the *Reports* list box and click **Show**.
Print	Displays the *Print* window for report output. • **Name** drop-down list and **Properties**. Select the printer from the drop-down list and click **Properties** to change printer setup, etc. • **Copies.** Select the number of copies and collate option. • **Current.** Selects the active report (or selected tab). • **All.** Selects all reports in the *Reports* list box. • **Shown.** Selects only the reports not hidden. • **Clear.** Clears all selections. • **OK.** Prints the selected report to the printer indicated. • **Cancel.** Closes the *Print* window.
Reports	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.

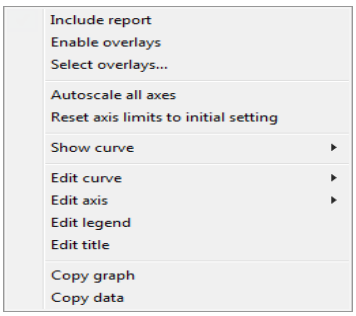
Report Toolbar Fields and Buttons Table (continued)

Field or Button	Description
Show	Displays the selected report in the *Reports* list box. If the report tab has been hidden using the **Hide** button, click **Show** to display the report and tab.

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

GRAPH FEATURES AND SHORTCUTS

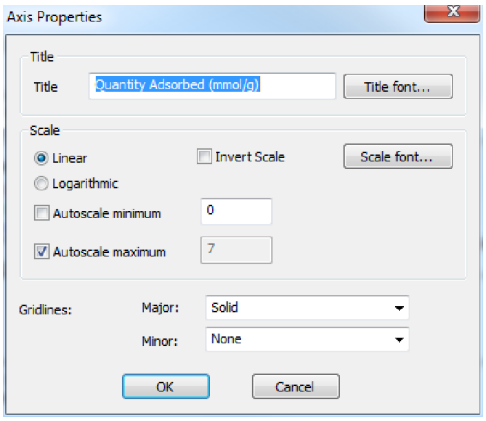
Display graph report shortcuts by right clicking in the body of the graph report.



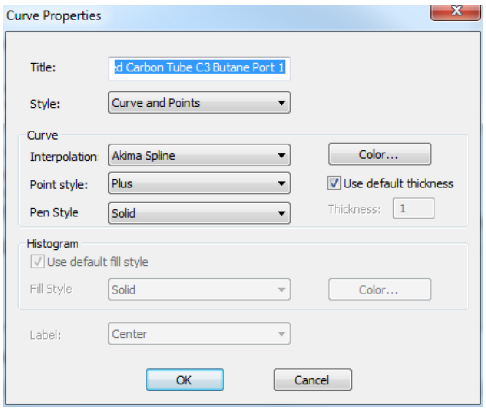
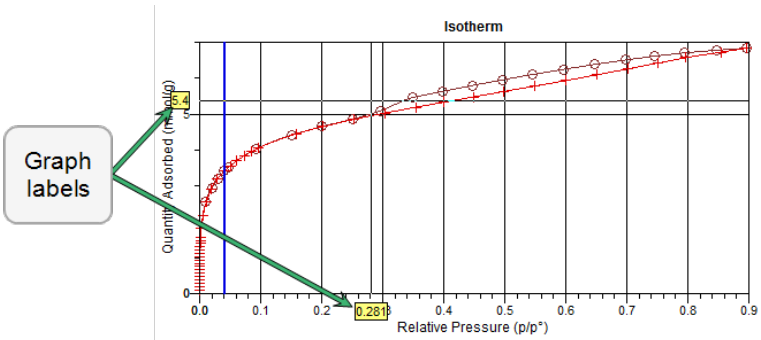
Graph Shortcuts Options and Description Table

Field or Button	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Copy Data	Copies the report data to the clipboard. It can then be pasted into other software programs as tab-delimited columns of text.
Copy Graph	Copies the graph to the clipboard. It can then be pasted into other software programs.

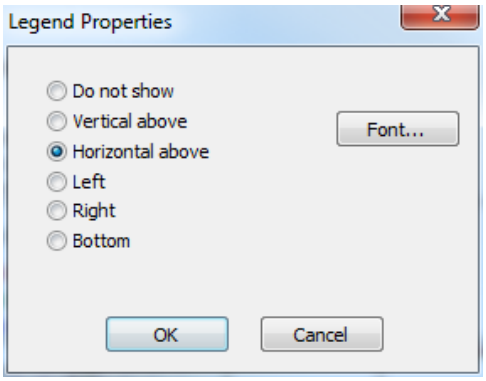
Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
Edit axis	Use to edit the selected axis properties.  • **Title.** Use to edit the selected axis label. • **Title font.** Use to modify the font for the selected axis label. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*. • **Linear / Logarithmic.** Select the option to scale the graph as linear or logarithmic. • **Autoscale minimum / maximum.** To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box. • **Invert scale.** Use to invert the scale. • **Scale font.** Use to modify the font for the scale label. Deselect *Use default font* to enable font options. • **Grid lines.** Use to change how to display major / minor grid lines.

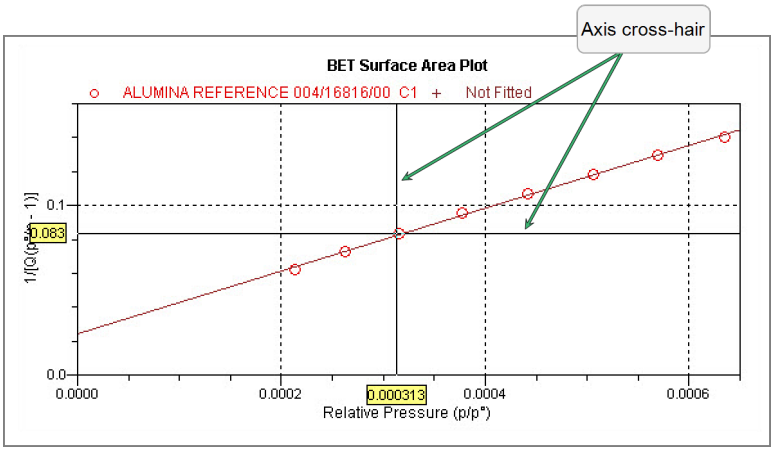
Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
Edit curve	Use to edit selected curve properties.  • **Title.** Use to change the title of the selected curve. • **Style.** Use to select another style for the collected data curve. • **Curve group box.** Use to change the interpolation, point style and pen style for the selected curve. These options are disabled if *Use default fill style* is selected in the *Histogram* group box. **Color.** Click to change the curve color. **Use default thickness.** Uses the default curve thickness. Deselect to enter a new thickness number in the *Thickness* text box. • **Histogram group box.** Enabled only if *Histogram* is selected in the *Style* drop-down list. Use to specify the type of fill, fill color and label position for the selected curve. • **Label.** Select where the graph point labels will display (left, right, center, etc.) on the SPC report.
 	

Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
Edit legend	Use to change the legend location and font. Click **Font** to modify font attributes. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*. 
Edit title	Use to change the graph title and font. Click **Font** to font attributes. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
Select overlays...	See. *"Generate Multiple Graph Overlays" on page 7 - 33*
Show curve	Displays a list of all curves. Select the curve(s) to display.

Axis Cross-Hair

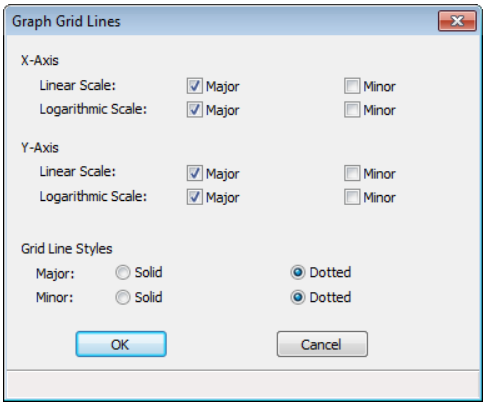


The cross-hair feature displays axis coordinates.

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* presentation option.

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.

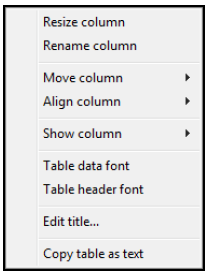
Zoom Feature

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

GENERATE MULTIPLE GRAPH OVERLAYS

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

- **Multiple Graph Overlays.** Overlay two different types of graphs from one sample.
- **Multiple Sample Overlays.** Overlay up to 20 graphs of the same type with that of the current plot.

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

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

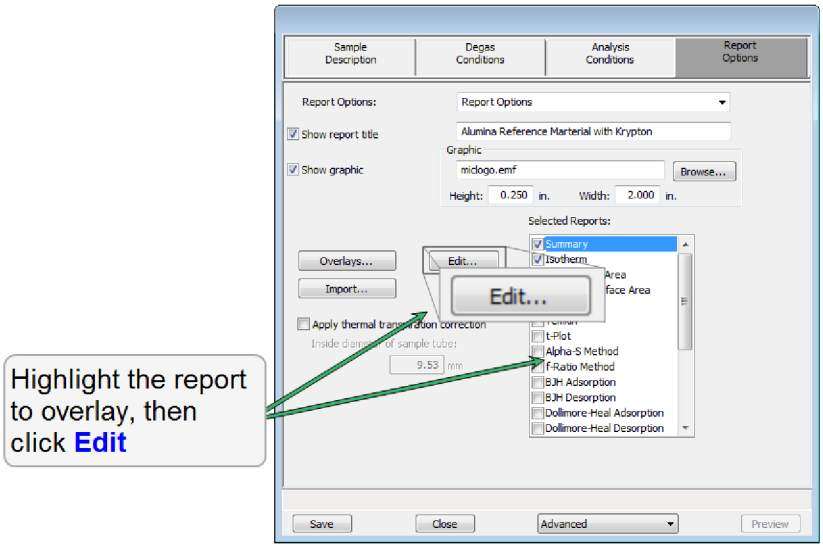
GENERATE MULTIPLE SAMPLE OVERLAYS

Physisorption ***Chemisorption***

To overlay the same type of graph on multiple samples:

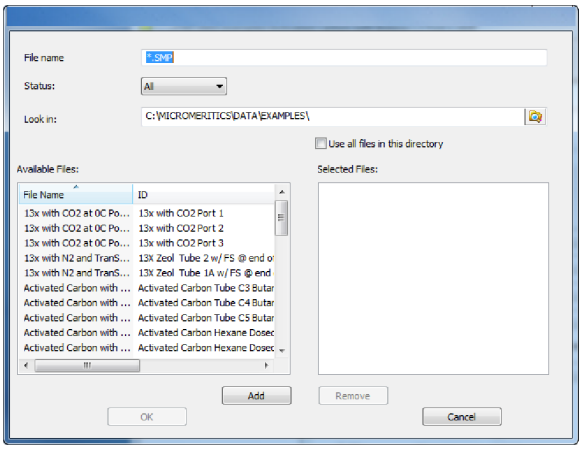
1. Go to ***File > Open***.
2. Select the .SMP file, then click **Open**.

   If a file with a status other than *Preparing*, *Prepared*, or *No Analysis* is selected, the Isotherm plot displays. Select *Advanced* from the drop-down list at the bottom of the window.
3. Click the *Report Options* tab at the top of the window. Refer to the following table for instructions for the selected report.



If overlaying this type of report...	Then...
• **Isotherm** (for physisorption)	a. In the *Selected Reports* list box, highlight *Isotherm*, then click **Edit**. Alternatively, double click the report. b. On the *Isotherm Report Options* window, select a plot in the *Selected Reports* group box, then click **Options** to the right of the selected plot. c. On the *Plot Options* window, select *Plot curve* and / or *Plot points* if they are to be included in the overlay. If the x- and / or y-axes are to be autoscaled, enable *Autoscale*; otherwise, enter the *From* and *To* points for the axes. Click **OK** to save and close the window. d. On the *Report Options* window, in the *Plot Options* group box, select *Plot overlays*. Click **OK**. e. Continue with Step 4.
(for physisorption) • **Alpha-S** • **BET Surface Area** • **f-Ratio** • **Freundlich** • **Langmuir** • **t-plot** • **Temkin** (for chemisorption) • **Difference Method** • **Sinfelt Method**	a. In the *Selected Reports* list box, highlight one of the report options shown on the left, then click **Edit**. Alternatively, double click the report. b. On the pop-up window, select *Overlay samples*. Verify other fields. Click **OK** to return to the *Report Options* tab. c. Continue with Step 4.
(for physisorption) • **BJH Adsorption** • **BJH Desorption** • **Dollimore-Heal Adsorption** • **Dollimore-Heal Desorption** • **MP-Method**	a. In the *Selected Reports* list box, highlight a report option shown on the left, then click **Edit**. Alternatively, double click the report. b. Select the report variable from the *Selected Reports* group box, then click **Edit**. c. Click the down arrow on the *Overlay* field, then select the *Samples* option. Verify other fields. Click **OK** to return to the *Report Options* window. d. Click **OK** again to return to the *Report Options* tab. e. Continue with Step 4.

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



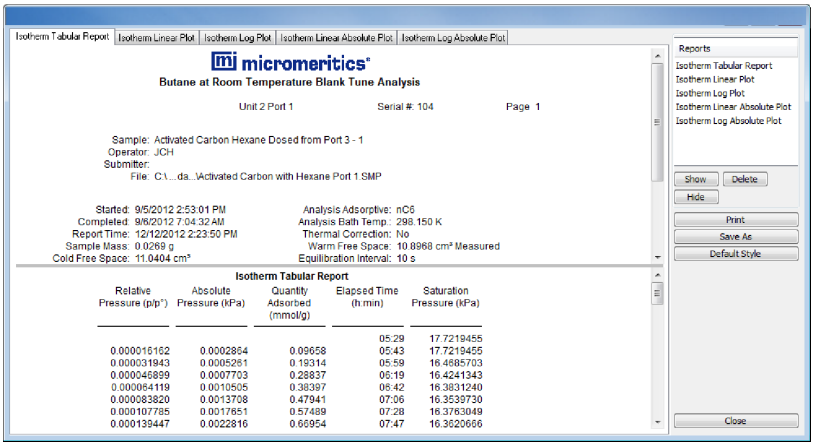
- Double click a file name in the *Available Files* box to move the file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, double click the file name in the *Selected Files* box, or
- Select a file name in the *Available Files* box. Click **Add** 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** on the sample file window. If the sample file has been closed, go to ***Reports > Start Report.*** Select the file used in the previous steps, then click **Report**. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.Choose the report destination on the *Report Settings* window, then click **OK**.

   If only one file was selected as an overlay, the *Select Reports* window displays. Verify the reports to generate and add or remove reports as necessary. Click **OK**.

   If multiple files were selected, the *Selected Reports* window will not display.

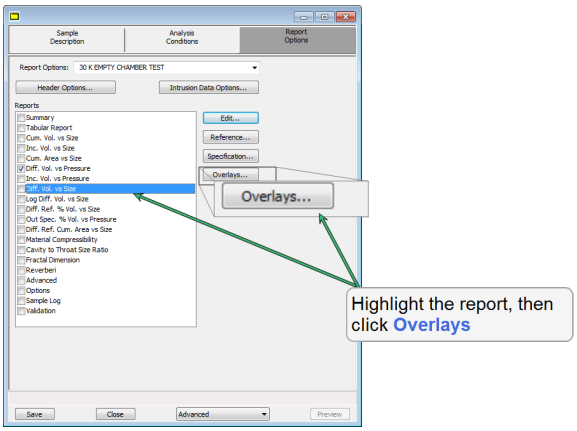
8. The report window displays with tabs across the top. Click each tab to view the reports.



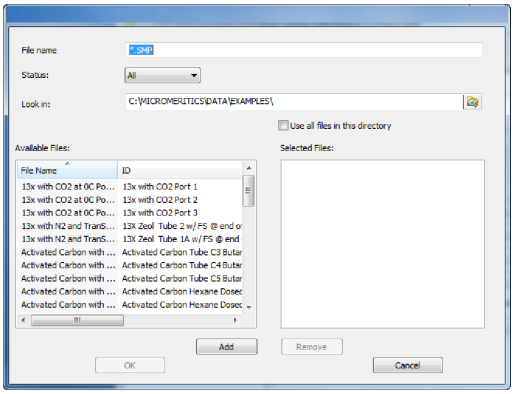
Mercury Porosimetry

This feature is applicable to overlaying samples from samples files with an *LP Complete*, *HP Complete* or *Entered* status.

1. Go to ***File > Open***.
2. Select the .SMP file, then click **Open**. Select *Advanced* from the drop-down list at the bottom of the window to display the *Report Options* tab.
3. Click the *Report Options* tab.
4. In the *Reports* list box, highlight a plot report, then click **Overlays**.



5. On the *Plot Overlay Sample Selection* window, use one of the following options to move files from the *Available Files* box to the *Selected Files* box. Once the files are in the *Selected Files*box, use **Ctrl** ↑ or **Ctrl** ↓ on the keyboard to change the position of the selected file.



- 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 **Save**, **Save As**, or **Preview**.
   - **Save.** Displays when the sample file has a status other than *No Analysis*. Click to save selected options.
   - **Preview.** Click to preview tabular reports and graph overlays. This button is disabled when the sample file has a status of *No Analysis*.
   - **Save As.** Displays if a new sample file was opened and has not been saved.

GENERATE MULTIPLE GRAPH OVERLAYS

Physisorption

Chemisorption

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 / DFT Surface Energy
- MP Method

ASCII text file format rules:

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

  Two examples of a header format:

  My Title (A, cm3/g, incremental)

  My Title (A, cm3/g, cumulative)

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

Sample ASCII text file

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

To import the ASCII text file to generate graph overlays:

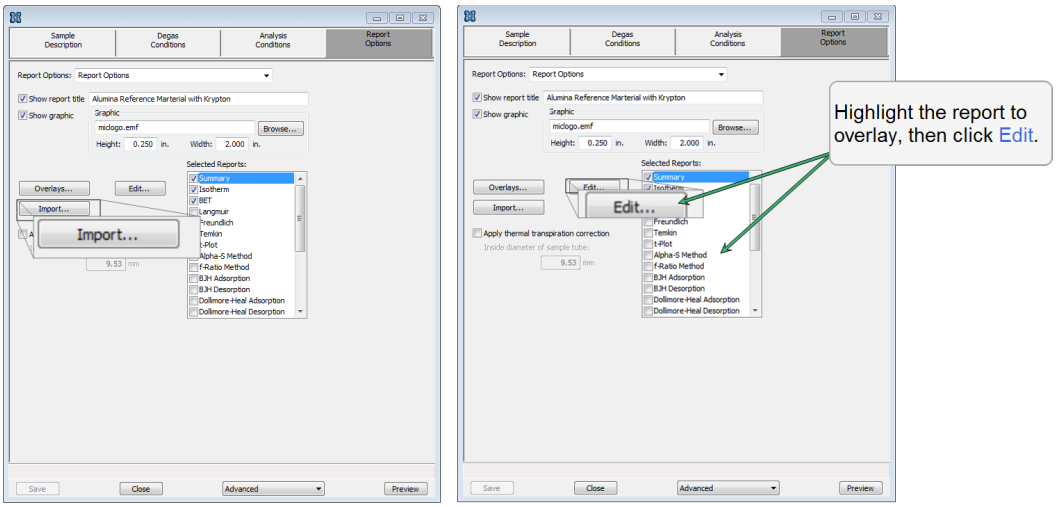
The following steps use BJH Adsorption as an example. Window appearance will vary depending on the selected report.

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**.

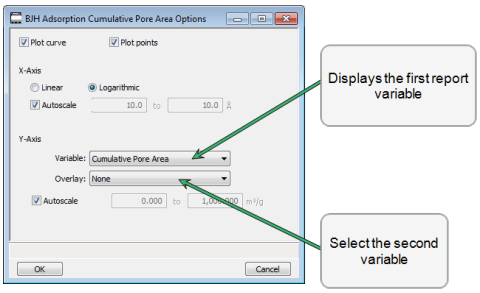
   If a file with a status other than *Preparing*, *Prepared*, or *No Analysis* is selected, the Isotherm plot displays. Select *Advanced* from the drop-down list at the bottom of the window to display the *Sample Description* tab.

2. Select the *Report Options* tab, then click **Import** to browse for the .TXT file. The *Select Imported Overlays* window displays.

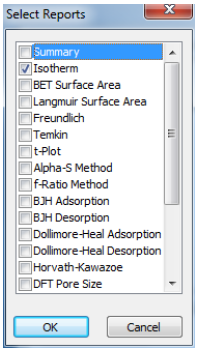
If the ASCII text file does not display on the *Select Imported Overlays* window, click **Import**. Locate the file, then click **Open**. Header information from the ASCII text file will then appear in the *Select Imported Overlays* window. Select the entry, then click **OK**. If an error message appears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



3. In the *Selected Reports* list box, highlight the type of report to overlay with a graph, then click **Edit.**
4. 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.

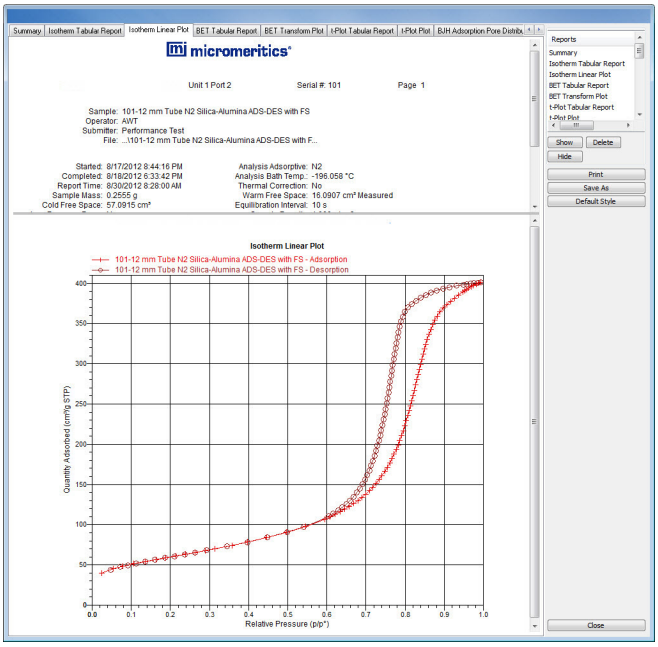


5. Click **OK** again to return to the *Report Options* tab.
6. Click **Save** to save the selections.
7. To view the report, click **Preview.**
8. If the sample file has been closed, go to ***Reports > Start Report***. Select the file used in the previous steps, then click **Report** .To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
9. Choose the report destination on the *Report Settings* window, then click **OK**.



If only one file was selected as an overlay, the *Select Reports* window displays. Verify the reports to generate, then add or remove reports as necessary. Click **OK**. If multiple files were selected, the *Selected Reports* window will not display.

10. The report window displays with tabs across the top. Click each tab to view the reports.



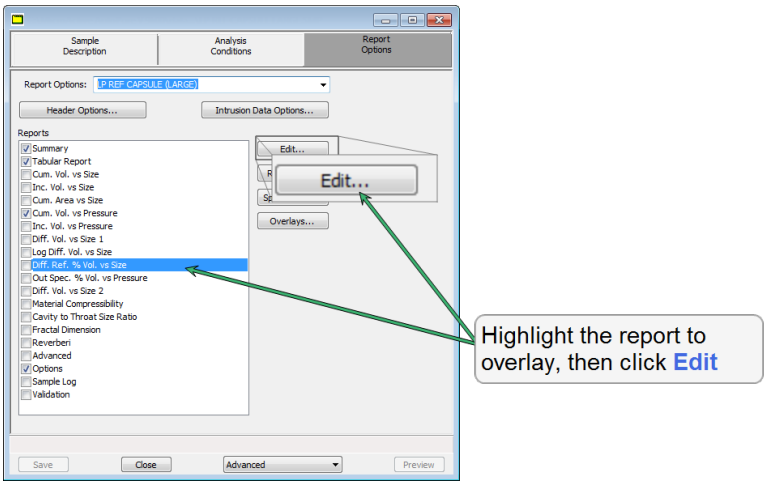
Mercury Porosimetry

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

   If a file with a status other than *No Analysis* is selected, the Intrusion plot displays. Select *Advanced* from the drop-down list at the bottom of the window to display the *Report Options* tab.

3. Click the *Report Options* tab.

4. In the *Reports* list box, highlight a plot report, then click **Edit**. Alternatively, double click the report.



5. On the *Plot Options* window, select *Options* to include in the overlay. If the x- and / or y-axes are to be autoscaled, select *Autoscale*; otherwise, enter the *From* and *To* points for the axes.
6. In the *Y-Axis* group box, select *Variable* and/or *Overlay* options.
7. Click **OK** to return to the *Report Options* tab.
8. Click **Save**, **Save As**, or **Preview**.
   - **Save.** Displays when the sample file has a status other than *No Analysis*. Click to save selected options from the drop-down lists.
   - **Preview.** Click to preview tabular reports and graph overlays. This button is disabled when the sample file has a status of *No Analysis*.
   - **Save As.** Displays if a new sample file was opened and has not been saved.

REPORT EXAMPLES FOR THE 3FLEX

T-PLOT REPORT EXAMPLE

micromeritics®

Unit 1 Port 2 Serial #: 101 Page 11

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

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

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

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

t-Plot Report

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

Thickness Curve

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

t-Plot Report - Data

Relative Pressure (p/p°)	Statistical Thickness (Å)	Quantity Adsorbed (cm³/g STP)	Fitted
0.053665461	3.2751	45.1706	
0.077824186	3.4987	48.2830	
0.106574940	3.7265	51.3824	*
0.135877231	3.9408	54.2113	*
0.163237219	4.1275	56.6908	*
0.188595088	4.2948	58.9330	*
0.213852389	4.4582	61.1303	*
0.238707954	4.6176	63.3032	*
0.263405375	4.7758	65.4875	*
0.288407930	4.9369	67.7418	*
0.313104034	5.0979	70.0202	
0.357549162	5.3950	74.3238	
0.397683828	5.6746	78.4880	
0.446861650	6.0373	84.0635	
0.496397055	6.4319	90.4326	
0.545717570	6.8629	97.8310	
0.594513636	7.3377	106.8632	
0.607888353	7.4780	109.7720	
0.620922246	7.6196	112.7842	
0.633541428	7.7617	115.9539	
0.645692079	7.9033	119.2176	
0.657391993	8.0446	122.6982	

BET SURFACE AREA

micromeritics®

Unit 1 Port 2 Serial #: 101 Page 9

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

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

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

BET Surface Area Report

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

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

BET Surface Area Plot

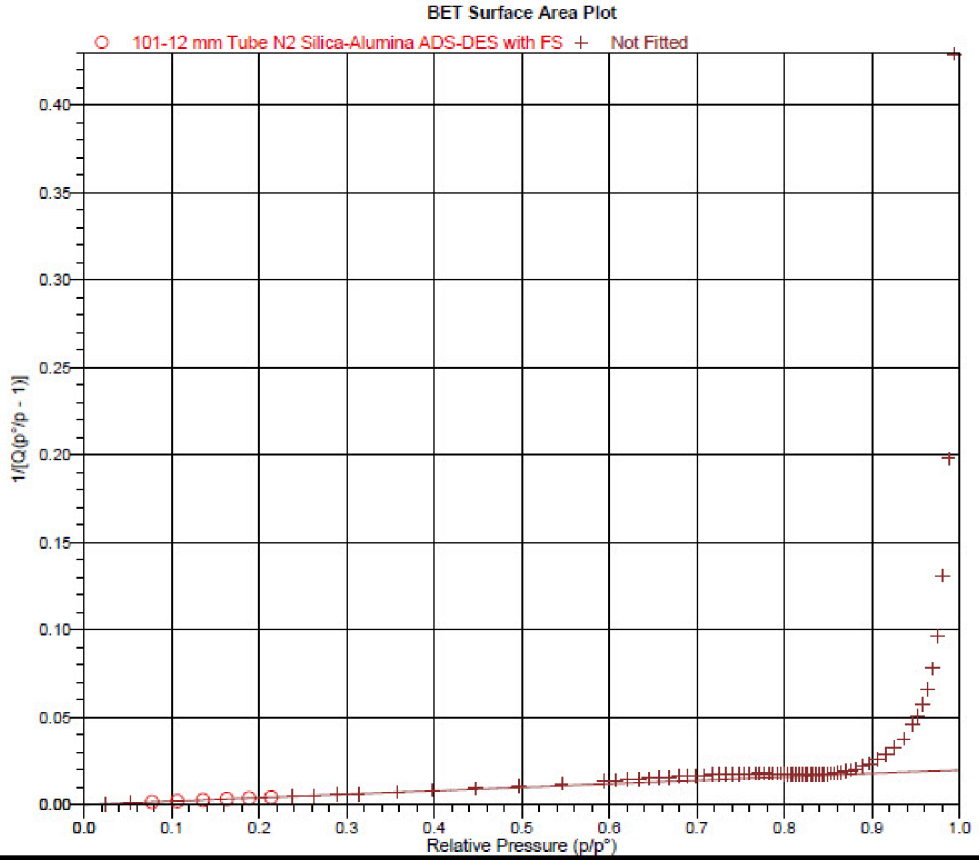
micromeritics®

Unit 1 Port 2 Serial #: 101 Page 10

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

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

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



ISOTHERM LINEAR PLOT

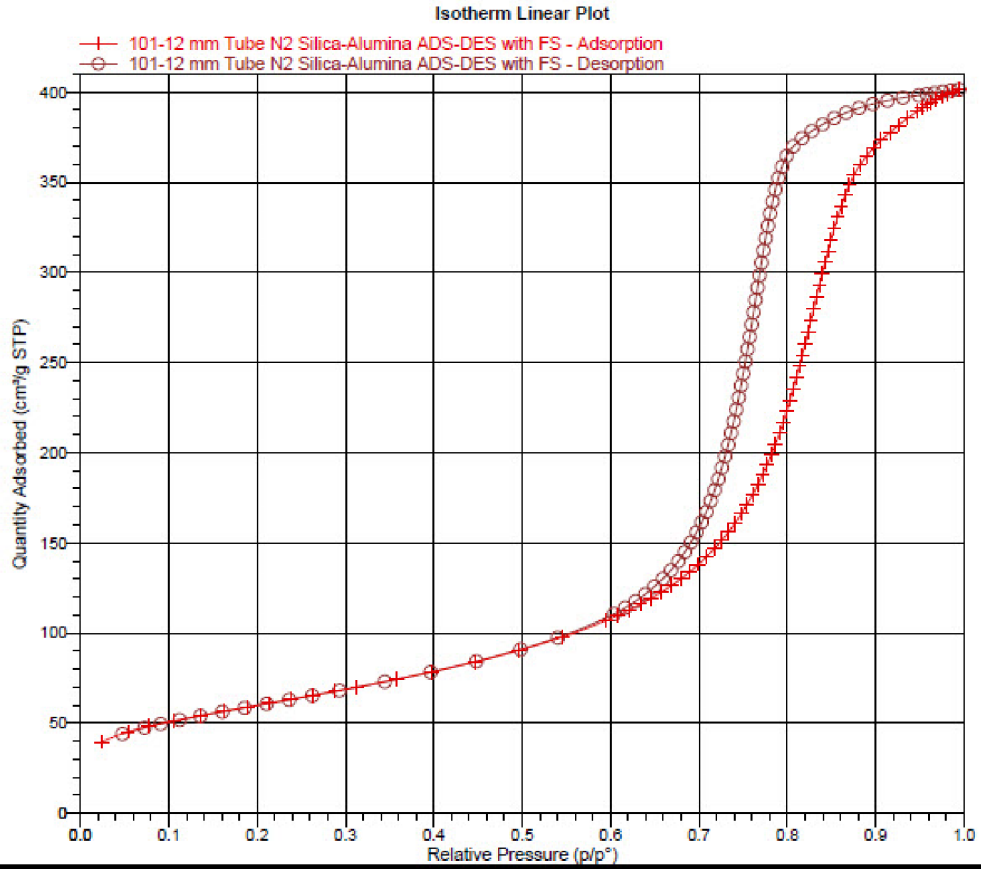
micromeritics®

Unit 1 Port 2 Serial #: 101 Page 8

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

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

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



BJH DESORPTION: CUMULATIVE PORE VOLUME

micromeritics®

Unit 1 Port 2 Serial #: 101 Page 24

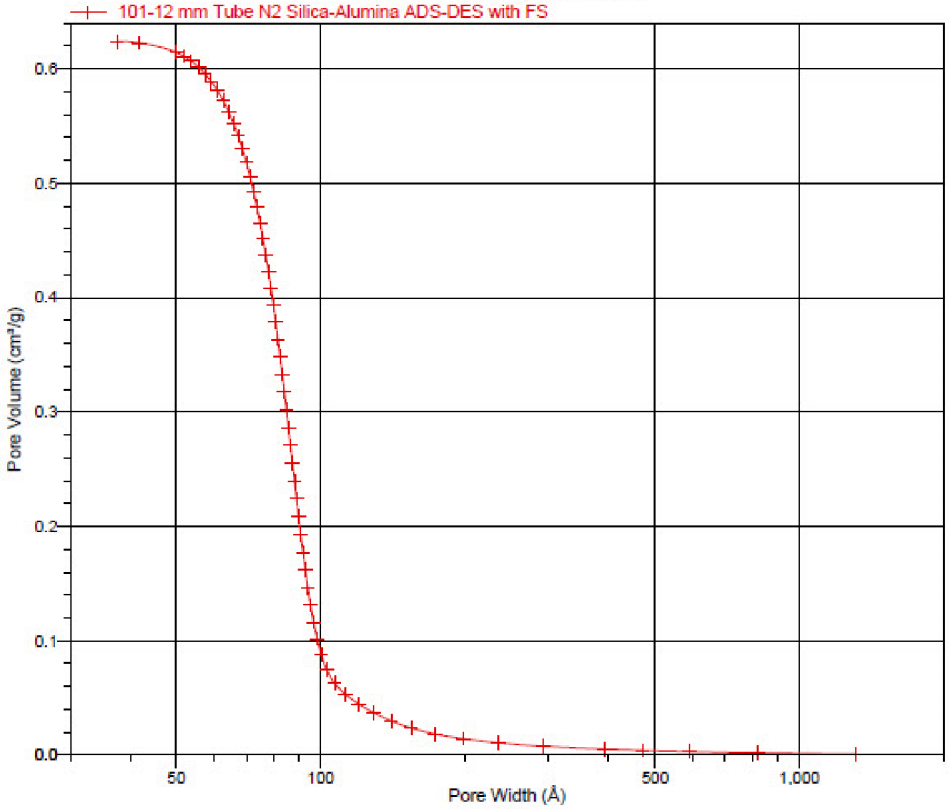
Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

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

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

BJH Desorption Cumulative Pore Volume (Larger)

Harkins and Jura : Faas Correction



BJH ADSORPTION: CUMULATIVE PORE VOLUME

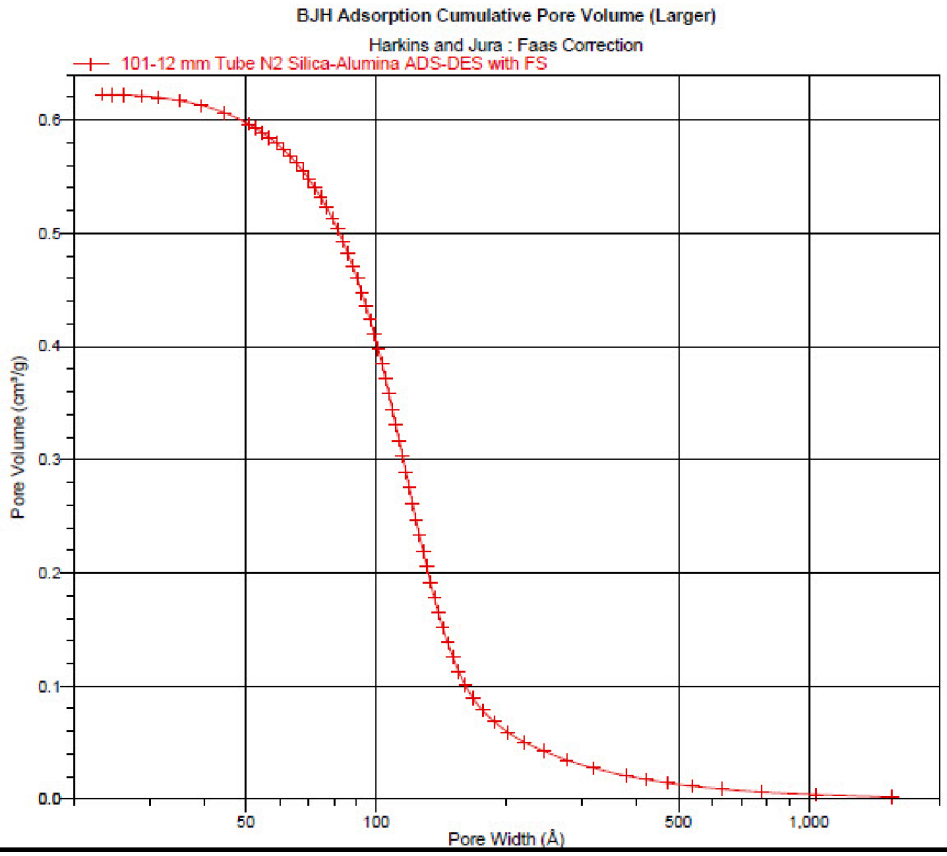
micromeritics®

Unit 1 Port 2 Serial #: 101 Page 16

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

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

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



REPORT EXAMPLES FOR THE AUTOPORE

CONTROLLED PORE GLASS PLOT

CONTROLLED PORE GLASS

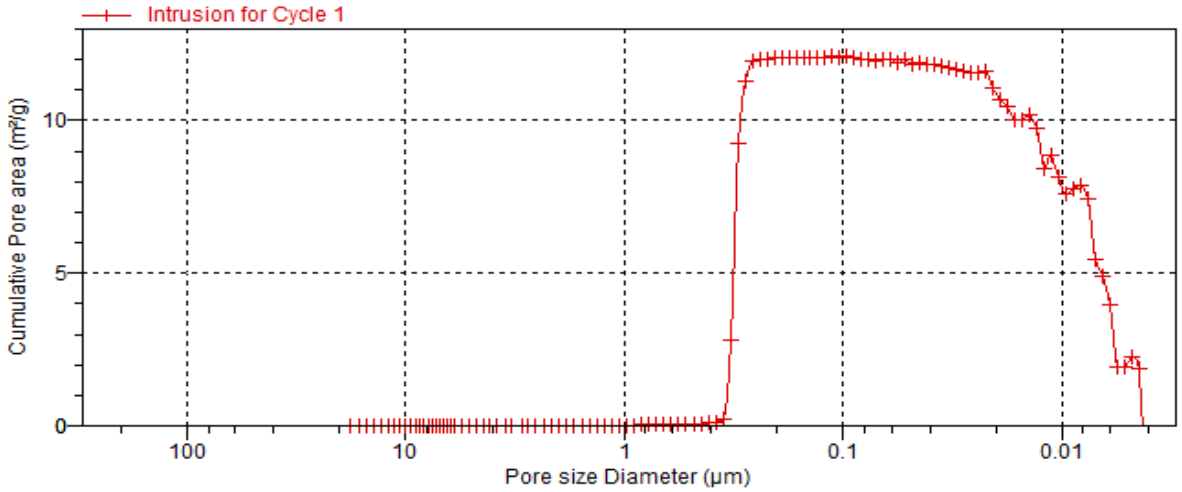
MicroActive AutoPore V 9600

MicroActive AutoPore V 9600
Serial # 102 Port 1/1

Sample: Controlled Pore Glass (3000 Angstrom)
Operator: BPM
Submitter: R. CAMP
File: C:\MicroActive AutoPore V 9600\...\CPG 3000 Port 1.SMP

LP Analysis Time:	4/11/2014 11:52:01 AM	Sample Mass:	0.2101 g
HP Analysis Time:	4/11/2014 1:12:06 PM	Stem Volume Used:	19 %
Report Time:	4/17/2014 7:47:16 AM	Show Neg. Int:	Yes
Report Range:	0.00050 to 5,000.00000 µm	Correction Type:	None
Adv. Contact Angle:	130.000 °	Mercury Temperature:	21.16 °C
Rec. Contact Angle:	130.000 °		

Cumulative Pore Area vs Pore size



GARNET TABULAR REPORT

Micromeritics Instrument Corporation

MicroActive AutoPore V 9600

MicroActive AutoPore V 9600
Serial # 102 Port 1/1

Sample: garnet ref mat 60k eqil (rate) 568
Operator: jch
Submitter: micromeritics performance test
File: C:\MicroActive AutoPore V ...\Garnet to 60K Port 1.SMP

LP Analysis Time: 4/10/2014 3:16:40 PM
HP Analysis Time: 4/10/2014 5:21:38 PM
Report Time: 4/17/2014 7:36:34 AM
Report Range: 0.10 to 61,000.00 psia
Adv. Contact Angle: 130.000 °
Rec. Contact Angle: 130.000 °

Sample Mass: 0.2899 g
Stem Volume Used: 28 %
Show Neg. Int: No
Correction Type: None
Mercury Temperature: 21.28 °C

Tabular Report

Pressure (psia)	Mean Diameter (µm)	Cumulative Pore Volume (mL/g)	Incremental Pore Volume (mL/g)	Cumulative Pore Area (m²/g)	Incremental Pore Area (m²/g)
1.02	176.49789	0.0000	0.0000	0.000	0.000
2.00	133.52629	0.0095	0.0095	0.000	0.000
3.00	75.44654	0.0206	0.0111	0.001	0.001
4.00	52.79904	0.0260	0.0053	0.001	0.000
5.00	40.72799	0.0314	0.0055	0.002	0.001
6.00	33.17779	0.0347	0.0032	0.002	0.000
6.99	28.00920	0.0379	0.0032	0.003	0.000
8.00	24.23739	0.0405	0.0026	0.003	0.000
8.99	21.36405	0.0429	0.0024	0.004	0.000
10.00	19.10306	0.0439	0.0011	0.004	0.000
10.99	17.27178	0.0460	0.0020	0.004	0.000
11.99	15.76445	0.0481	0.0021	0.005	0.001
13.00	14.49805	0.0499	0.0018	0.005	0.001
13.99	13.42028	0.0515	0.0016	0.006	0.000
14.99	12.49258	0.0531	0.0016	0.006	0.000
15.99	11.68533	0.0542	0.0011	0.007	0.000
16.99	10.97620	0.0555	0.0013	0.007	0.000
17.99	10.34770	0.0567	0.0012	0.008	0.000
18.99	9.78763	0.0577	0.0009	0.008	0.000
19.99	9.28496	0.0587	0.0010	0.008	0.000
20.99	8.83121	0.0598	0.0011	0.009	0.000
21.99	8.42027	0.0608	0.0010	0.009	0.000
22.99	8.04556	0.0616	0.0009	0.010	0.000
23.99	7.70264	0.0627	0.0010	0.010	0.001
24.99	7.38819	0.0635	0.0009	0.011	0.000
25.99	7.09860	0.0639	0.0004	0.011	0.000
26.99	6.83063	0.0653	0.0014	0.012	0.001
27.99	6.58155	0.0657	0.0004	0.012	0.000
28.99	6.35046	0.0671	0.0014	0.013	0.001
30.00	6.13444	0.0679	0.0009	0.014	0.001
30.45	5.98441	0.0683	0.0004	0.014	0.000
30.92	5.89437	0.0687	0.0004	0.014	0.000

REVERBERI REPORT PLOT

Micromeritics Instrument Corporation

oActive AutoPore V 9600

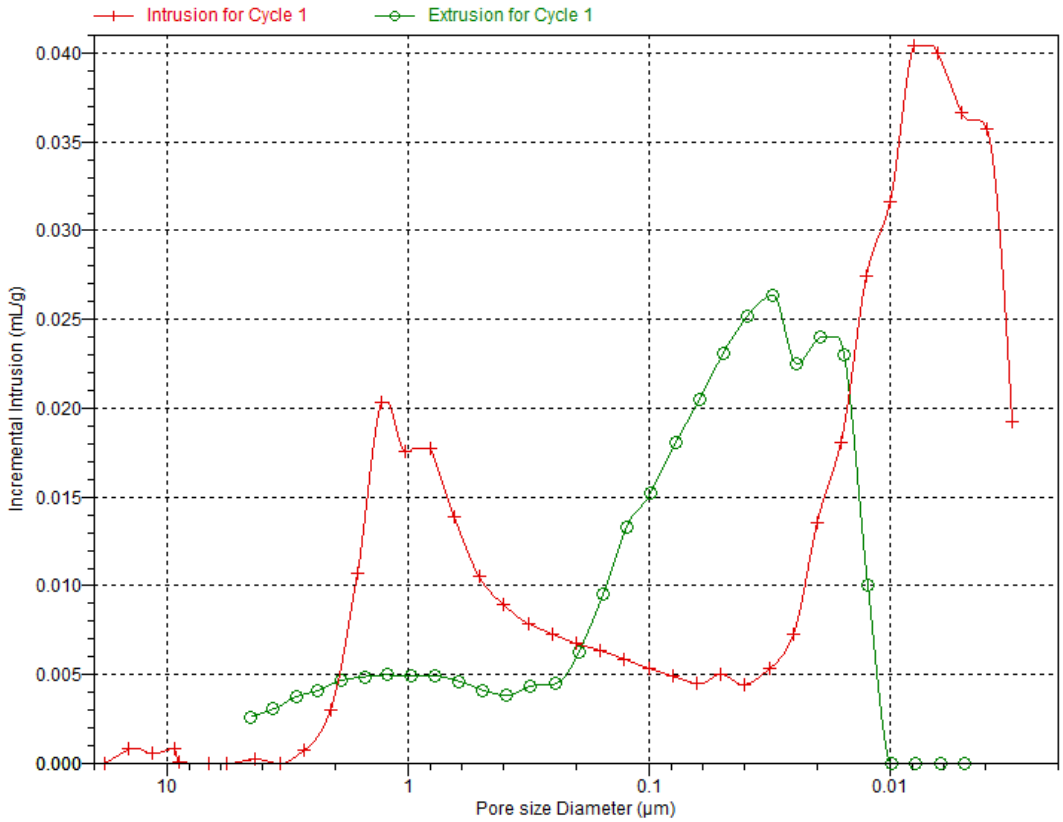
MicroActive AutoPore V 9600
Serial # 102 Port 3/1

Sample: Reverberi Sample
Operator:
Submitter:
File: C:\MicroActive AutoPore...\Reverberi Sample Port 3.SMP

LP Analysis Time: 4/16/2014 11:07:12 AM
HP Analysis Time: 4/16/2014 2:45:09 PM
Report Time: 4/17/2014 6:59:21 AM
Report Range: 0.10 to 61,000.00 psia
Adv. Contact Angle: 130.000 °
Rec. Contact Angle: 130.000 °

Sample Mass: 0.8264 g
Stem Volume Used: 33 %
Show Neg. Int: No
Correction Type: None
Mercury Temperature: 21.02 °C

Incremental Intrusion vs Pore size



SILICA ALUMINA REFERENCE MATERIAL REPORT

Micromeritics Instrument Corporation

MicroActive AutoPore V 9600

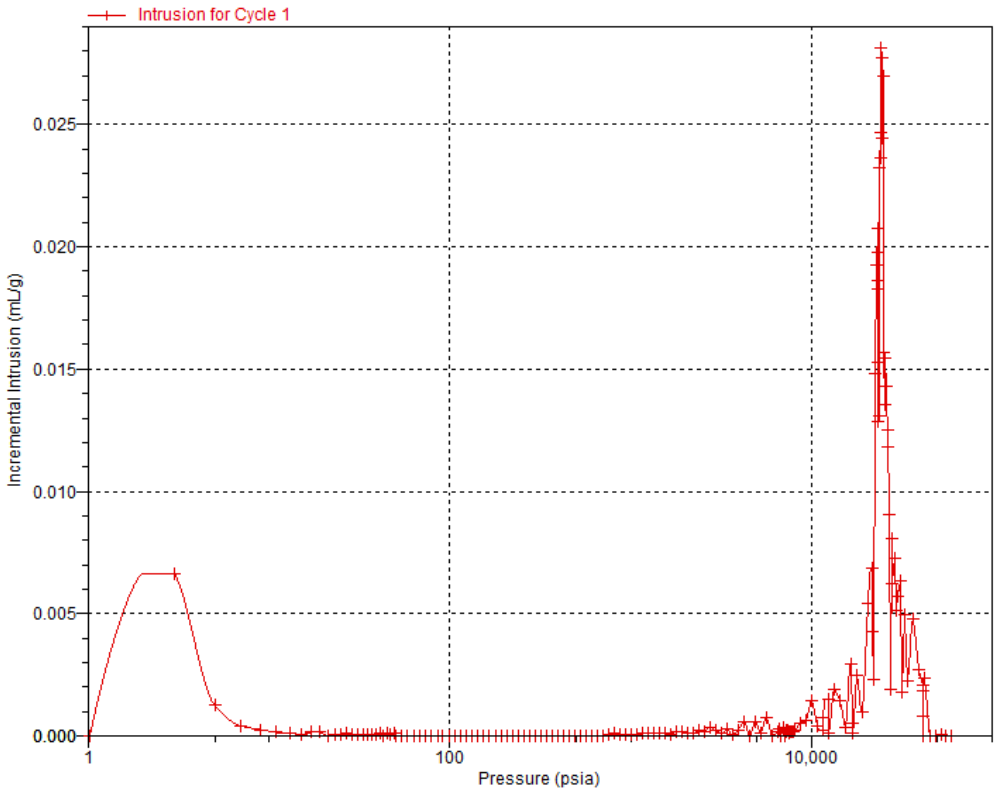
MicroActive AutoPore V 9600
Serial # 102 Port 1/1

Sample: Silica Alumina Reference Material Run A
Operator:
Submitter:
File: C:\MicroActive AutoPore V...\Silica Alumina Port 1.SMP

LP Analysis Time: 4/8/2014 8:52:51 PM
HP Analysis Time: 4/9/2014 8:33:55 AM
Report Time: 4/17/2014 7:24:39 AM
Report Range: 0.10 to 61,000.00 psia
Adv. Contact Angle: 130.000 °
Rec. Contact Angle: 130.000 °

Sample Mass: 1.0652 g
Stem Volume Used: 51 %
Show Neg. Int: No
Correction Type: None
Mercury Temperature: 21.64 °C

Incremental Intrusion vs Pressure



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8 Report Options for Physisorption

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**.

File > Open > [Report file]

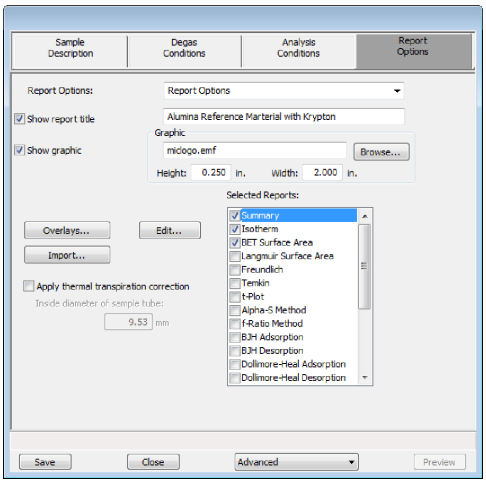
(or select the *Report Options* tab in the Sample Information file when using the *Advanced* presentation option)

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
- manually entered

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



Report Options Fields and Buttons Table

Field or Button	Description
Apply thermal transpiration correction	Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Always use thermal transpiration when performing micropore analysis. See *"Thermal Transpiration Correction" on page 12 - 47*. • **Inside diameter of sample tube.** Enabled when *Apply thermal transpiration correction* is selected. Enter the inside diameter of the sample tube used in the analysis. If filler rods are used, enter the filler rod capillary diameter of 1 mm instead.
Import (for physisorption)	Import up to 25 pore distribution data files. These datasets are shown only in BJH and Dollimore-Heal reports.
Name column	Displays a list of files in the selected directory.
Overlays	See *"Generate Multiple Graph 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. For BJH reports, BJH pore dimension can be calculated in pore width (w), pore radius (R) or pore diameter (D). Go to ***Options > Units*** to specify default calculations.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.

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

ADVANCED REPORT OPTIONS

See *"Python Module - Advanced Reports" on page 11 - 1*

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

1. Create the Python script and save it in the *Scripts* directory.
2. In the *Selected Reports* group box, click the drop-down arrow to select up to 5 Python scripts previously added in the *Available Scripts* box.
3. Click **Pressures** to add pressure points to the report.
4. Click **OK** to return to the *Report Options* tab.
5. On the *Report Options* tab window, click **Preview**. The Python Reports will be included on the tabs across the top portion of the *Reports* window.
6. Select the *Overlay Samples* checkbox to enable the overlay sample feature.

Advanced Report Options Fields and Buttons Table

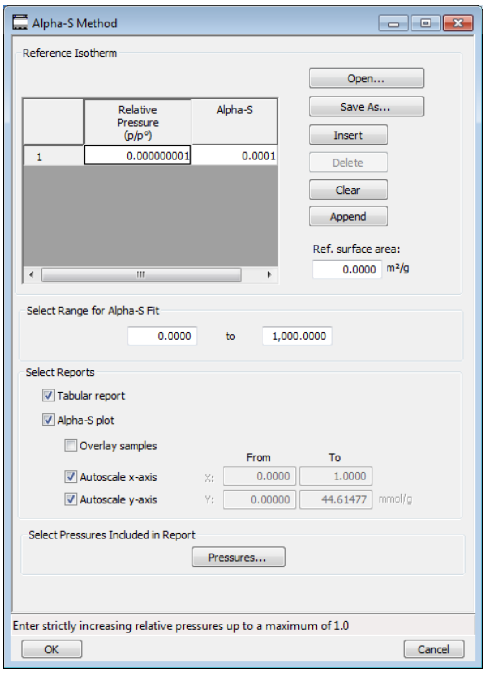
Field or Button	Description
Add	Click to add additional Python reports.
Available Scripts	Lists the available reports and provides the option to add, replace, edit or remove reports.
Overlay samples (if shown)	Use to overlay samples as defined by the function.

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

ALPHA-S METHOD REPORT OPTIONS

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

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

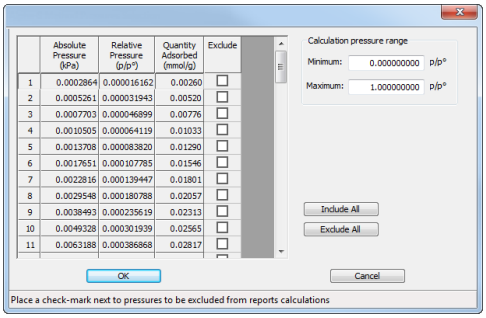


One predefined curve is located in the *Reference* file directory. Use the table buttons to enter relative pressure and the alpha-s values.

Alpha-S Method Report Options Fields and Buttons Table

Field or Button	Description
Alpha-S plot	Use to plot data in graph format. • **Overlay samples.** Use to overlay sample files on the plot. • **Autoscale x-axis.** The x-axis field shows the relative pressure. • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.

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

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

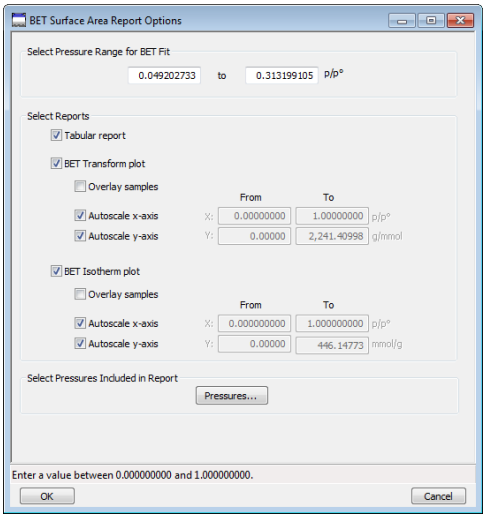
Selected Reports	• **Tabular Report**. Use to have a tabular report of data generated.
	• **Alpha-S Plot.** Use to plot data in graph format.
	  ○ **Overlay samples.** Use to overlay sample files on the plot.
	  ○ **Autoscale x-axis.** The x-axis field shows the relative pressure.
	  ○ **Autoscale y-axis.**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

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

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

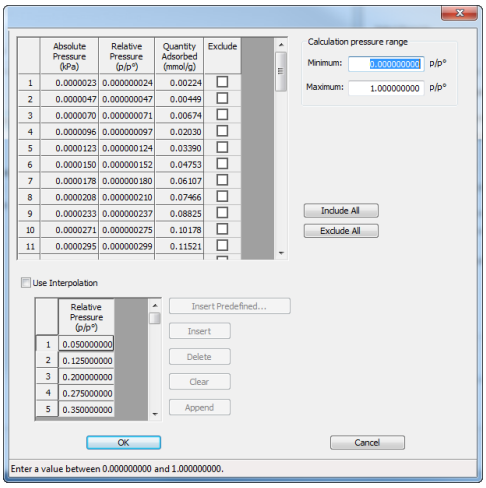
BET SURFACE AREA REPORT OPTIONS

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



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

BET Report Options Fields and Buttons Table

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

BET Report Options Fields and Buttons Table (continued)

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

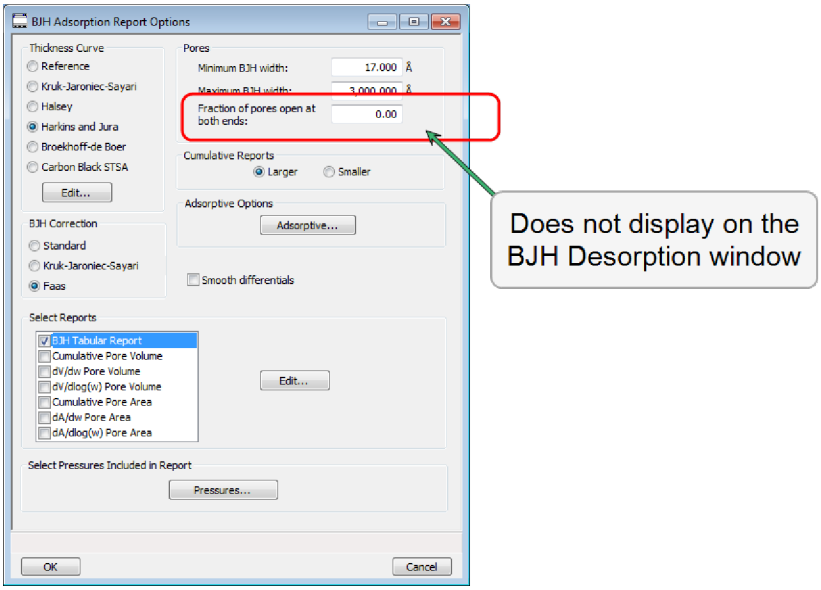
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

BJH Adsorption / Desorption Report Options

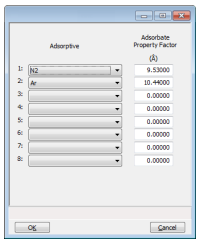
A tutorial is available for this topic. To view the tutorial, click the *Tutorial* tab in Online Help.

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

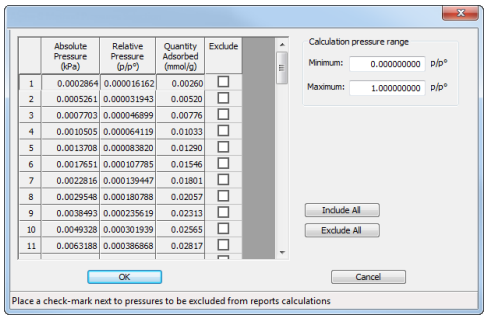
An incomplete pore distribution may be generated if a thickness curve selection is not a good match for the sample being analyzed.



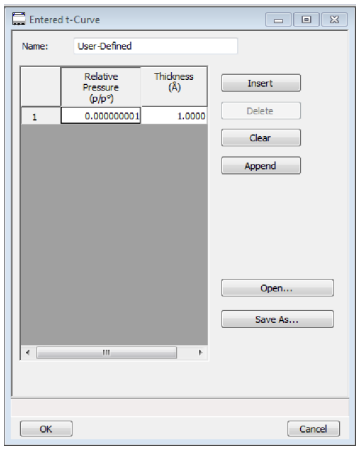
BJH Adsorption / Desorption Report Options Fields and Buttons Table

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

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

Field or Button	Description
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Select Reports	Select the 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.

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

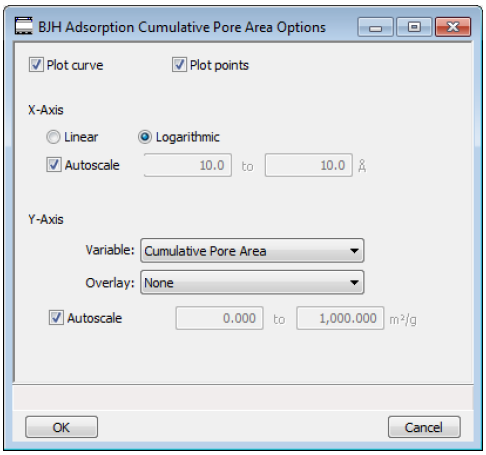
Field or Button	Description
Thickness Curve	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Reference.** Select *Reference*, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory.  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. Use to modify the table contents. • **Insert.** Inserts one row above the selected row. • **Delete.** Deletes the selected row. • **Clear.** Clears all table entries and displays only one default value. • **Append.** Inserts one row at the end of the table. **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.

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

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

BJH PLOT OPTIONS

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



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

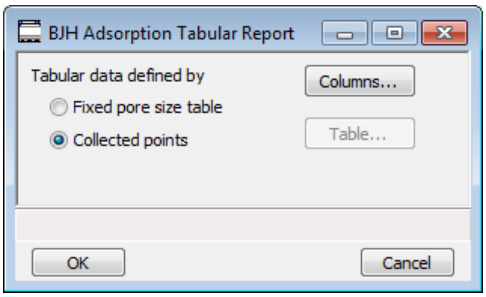
BJH Plot Options Fields and Buttons Table

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

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

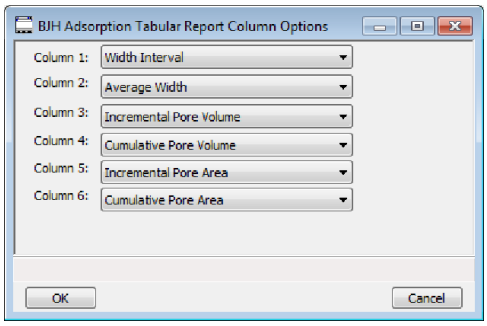
BJH Tabular Report Options

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



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

BJH Tabular Report Options Fields and Buttons Table

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

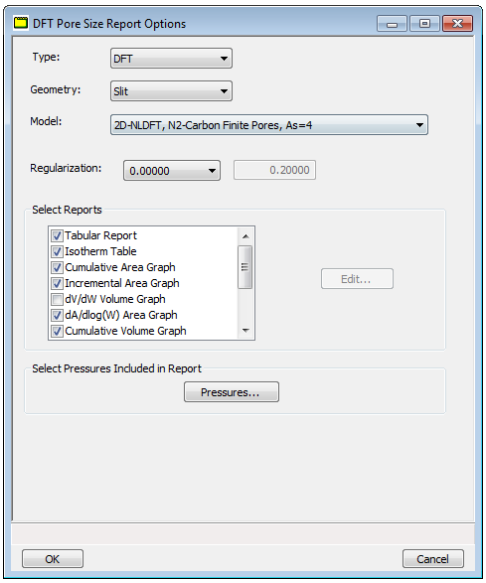
BJH Tabular Report Options Fields and Buttons Table (continued)

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

DFT Pore Size Report Options

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

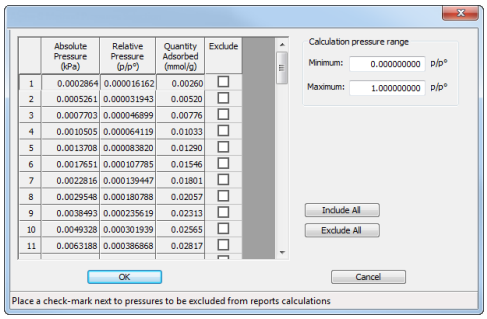
The *DFT Pore Size* report contains the results of pore size distribution analyses using a non-local DFT range of micro and mesopore ranges.



DFT Pore Size Report Options Fields and Buttons Table

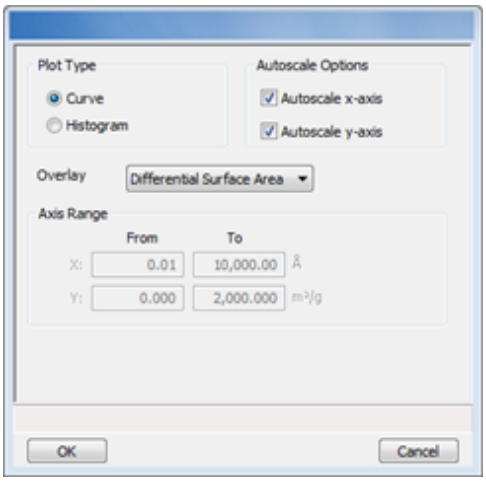
Field or Button	Description
Geometry	Select the pore shape.
Model	Lists the models that meet the specified criteria and match the adsorbate and temperature of the sample data. If no models appear, no models meet the selected criteria. One model must be selected.

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

Field or Button	Description
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Regularization	Select the extent of smoothing to apply to the data. If *0.20000 (user)* is selected, enter a number in the text box giving a relative weight for the smoothing during deconvolution. Larger values produce more smoothing.

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

Field or Button	Description

Select Reports	Select the reports to generate. To edit graph details, highlight the graph option and click **Edit**. The *Log Goodness of Fit* and *Goodness of Fit* graphs cannot be edited.
	 
	
	• **Plot Type.** Select the method for data display.
	• **Autoscale Options.** Use to autoscale the x-axis and / or y-axes.
	• **Overlay.** Select an overlay for the report.
	• **Axis Range.** *From* / *To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and / or y-axes.
	  ○ **X-axis**. Shows the pore size.
	  ○ **Y-axis.** Shows the area.

Type	• **DFT.** Model based on the density functional theory. • **Classical.** Model based on the Kelvin equation and thickness for determining the pore size distribution. See *"DFT Models" on page 13 - 1*.

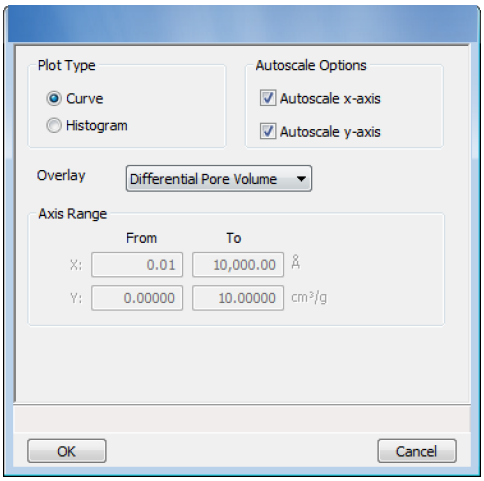
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

DFT SURFACE ENERGY REPORT OPTIONS

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

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

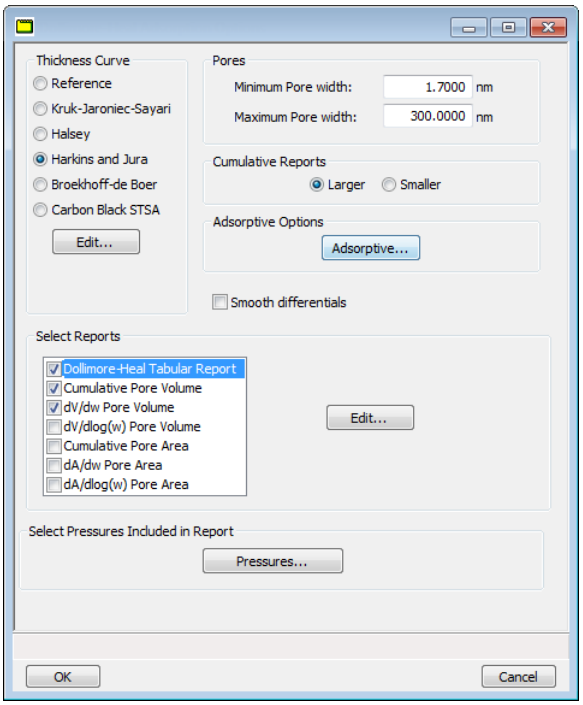
DFT Surface Energy Report Options fields and buttons are identical to the *DFT Pore Size Report Options*. See ***"DFT Pore Size Report Options" on page 8 - 18***.



DOLLIMORE-HEAL ADSORPTION / DESORPTION REPORT OPTIONS

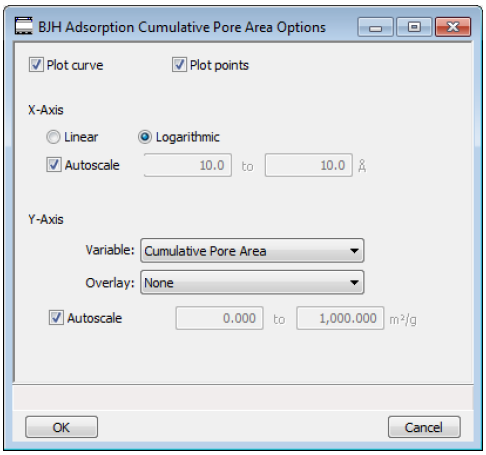
A tutorial is available for this topic. To view the tutorial, click the *Tutorial* tab in Online Help.

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 - 10***.



DOLLIMORE-HEAL PLOT OPTIONS

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



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

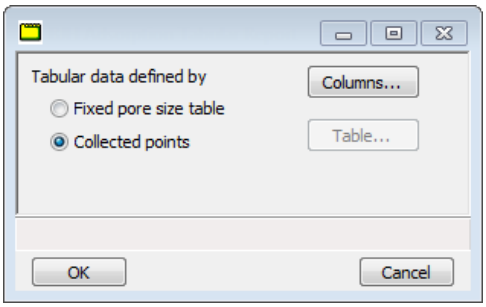
BJH Plot Options Fields and Buttons Table

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

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

DOLLIMORE-HEAL TABULAR REPORT OPTIONS

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

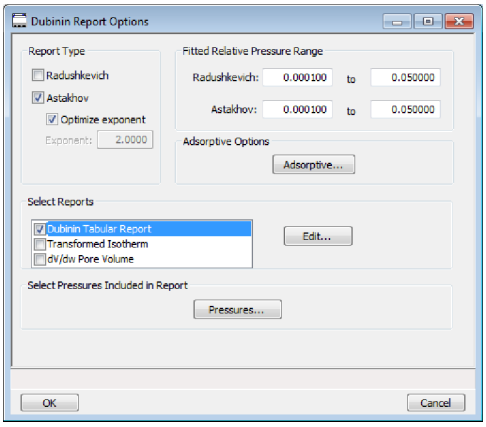


Dollimore-Heal Tabular Report Options are identical to the *BJH Tabular Report Options*. See *"BJH Tabular Report Options" on page 8 - 16*.

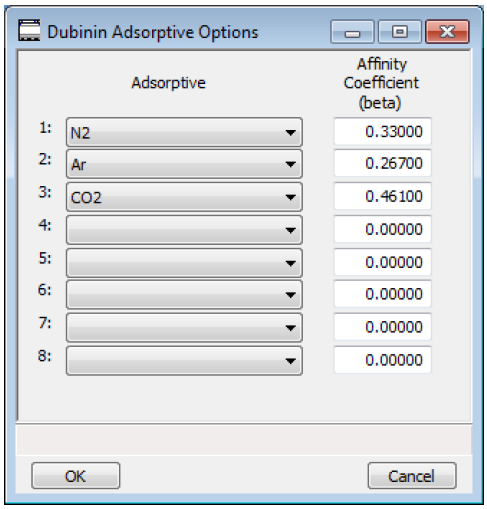
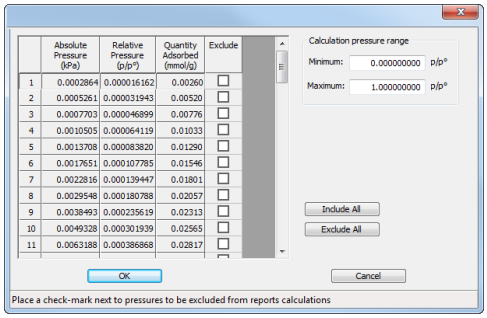
DUBININ REPORT OPTIONS

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

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



Dubinin Report Options Fields and Buttons Table

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

Dubinin Report Options Fields and Buttons Table (continued)

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

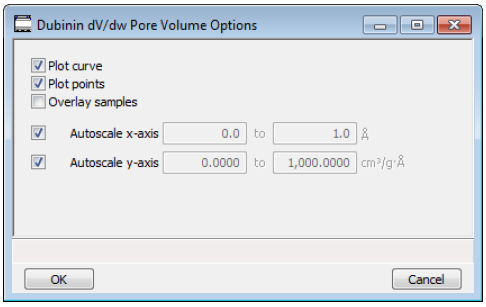
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

DUBININ PORE VOLUME REPORT OPTIONS

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

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

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



Dubinin Pore Volume Report Fields and Buttons Table

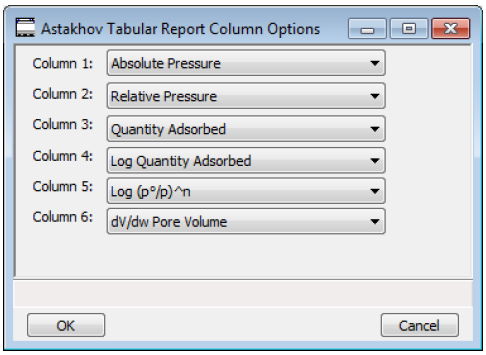
Field or Button	Description
Autoscale x-axis / Autoscale y-axis	Select an option to have the x- and / or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges. Enable to enter beginning and ending values manually.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

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

DUBININ TABULAR REPORT OPTIONS

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

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



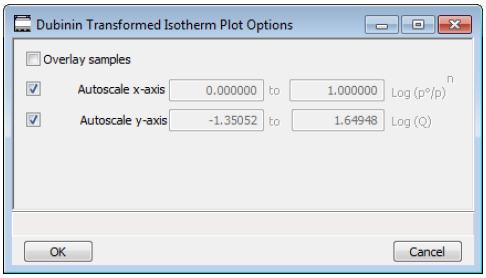
Log (p^0/p)^n. The value for *[n]* is the optimized exponent if *Optimize exponent* is selected on the *Dubinin Report Options* window. If not, then the value for *[n]* is the entered exponent value.

DUBININ TRANSFORMED ISOTHERM PLOT OPTIONS

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

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

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



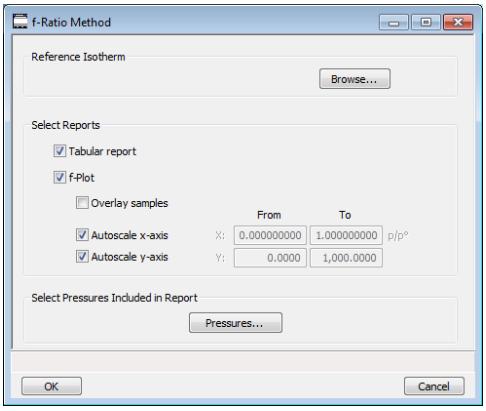
Dubinin Transformed Isotherm Plot Options Fields and Buttons Table

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

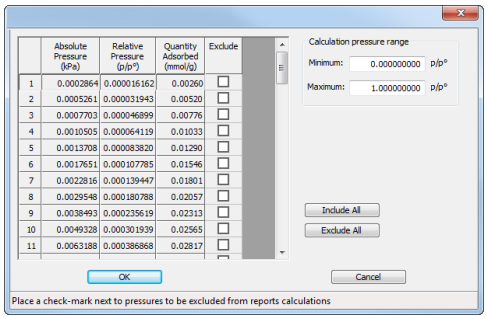
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

F-RATIO METHOD REPORT OPTIONS

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



f-Ratio Method Report Options Fields and Buttons Table

Field or Button	Description
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.

f-Ratio Method Report Options Fields and Buttons Table (continued)

Field or Button	Description
Reference isotherm	Click **Browse** to select a sample file to use as a reference for the isotherm. Select a file containing an isotherm measured from a non-porous sample of the same material as the current sample. When the referenced file is selected, the file name appears to the left of **Browse.**

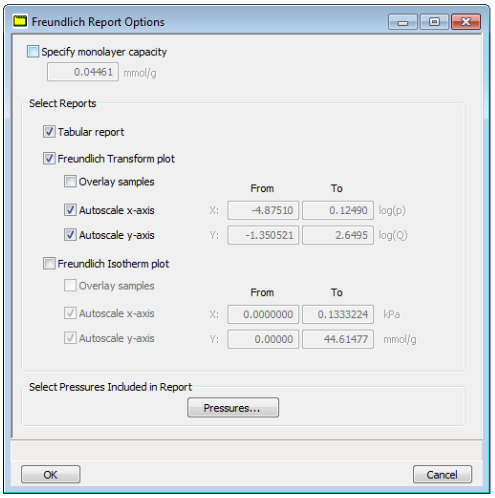
Selected Reports	• **Tabular Report.** Use to have a tabular report of data generated.
	• **f-Plot.** Use to generate a normalized isotherm.
	  ○ **Overlay samples.** Use to overlay sample files on the f-plot.
	  ○ **Autoscale x-axis.** The x-axis field is dimensionless in units of f-ratio.
	  ○ **Autoscale y-axis**. The y-axis field shows the quantity of gas adsorbed.

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

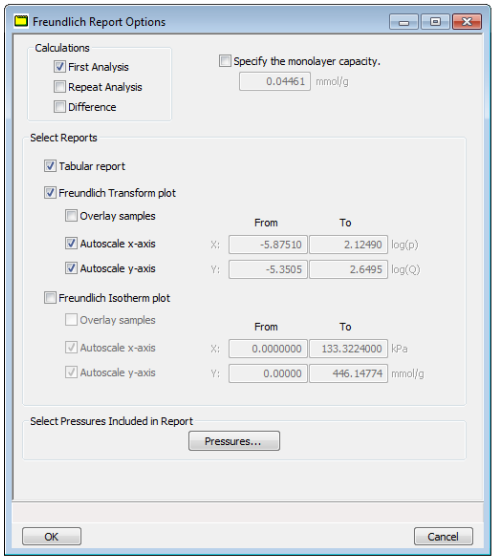
FREUNDLICH REPORT OPTIONS

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

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



Physisorption

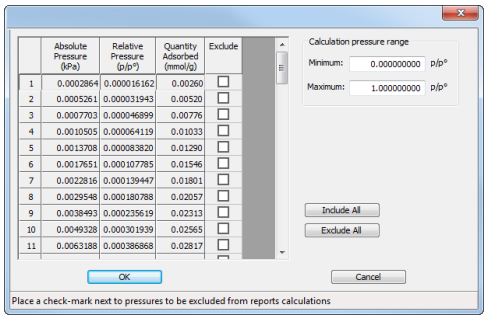
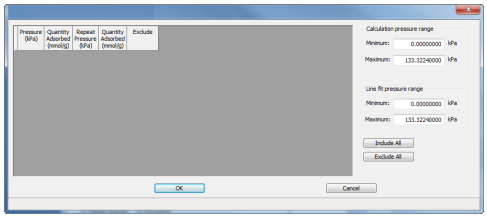


Chemisorption

Freundlich Report Options Fields and Buttons Table

Field or Button	Description
Calculations (for chemisorption)	Select from the various calculation options. • **First Analysis**. Includes a line fit plot for the primary analysis. • **Repeat Analysis.** Includes a line fit plot for the secondary analysis. • **Difference**. Plots the difference between the analysis and repeat analysis lines.

Freundlich Report Options Fields and Buttons Table (continued)

Field or Button	Description
Pressures (for physisorption)	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Pressures (for chemisorption)	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Line fit pressure range.** Enter the minimum and maximum pressures for line fit. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.

Freundlich Report Options Fields and Buttons Table (continued)

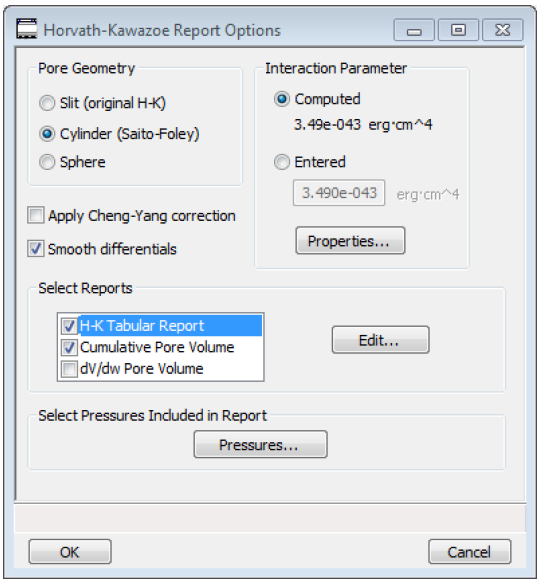
Field or Button	Description
Select Reports	• **Tabular report.** Select to include pressure points included in the report. • **Freundlich Isotherm plot.** Plots the absolute pressure vs quantity adsorbed. Shows best fit line. ◦ **Overlay samples.** Use to overlay sample files on the Freundlich isotherm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

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

HORVATH-KAWAZOE REPORT OPTIONS

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

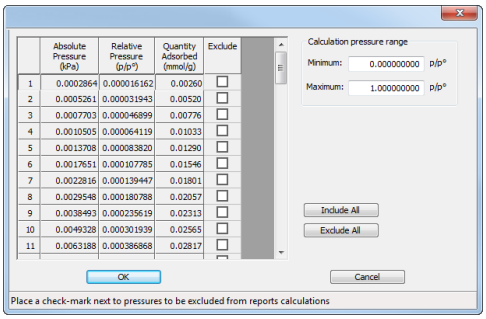
The *Horvath-Kawazoe* method plots individual peaks for different pore sizes even if the difference between one pore size and the next is only one angstrom (0.10 nm) or less.



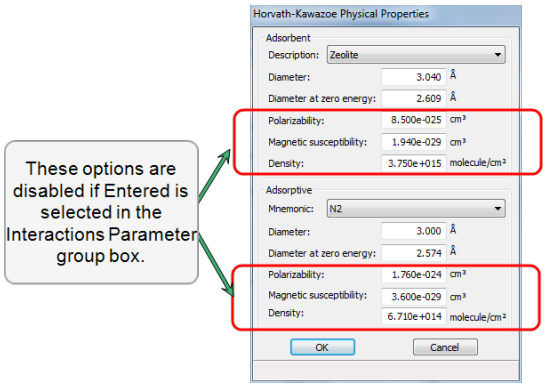
Horvath-Kawazoe Report Fields and Buttons Table

Field or Button	Description
Apply Cheng-Yang correction	Use to apply the Cheng/Yang correction to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath-Kawazoe derivation.
Interaction Parameter	Use to determine which interaction parameter will be used in the report. These options are disabled if *Sphere* is selected in the *Pore Geometry* group box. • **Computed.** Use to calculate using the parameters on the *Horvath-Kawazoe Physical Properties* window (click **Properties** to display the *Physical Properties* window). The interaction parameter is recalculated each time a parameter in the *Physical Properties* window is edited. • **Entered.** Calculates using the value entered in the text box.

Horvath-Kawazoe Report Fields and Buttons Table (continued)

Field or Button	Description
Pore Geometry	Select the option that best represents the physical geometry of the micropores in the sample material. When *Sphere* is selected, options in the *Interaction Parameter* group box are disabled.
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.

Horvath-Kawazoe Report Fields and Buttons Table (continued)

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

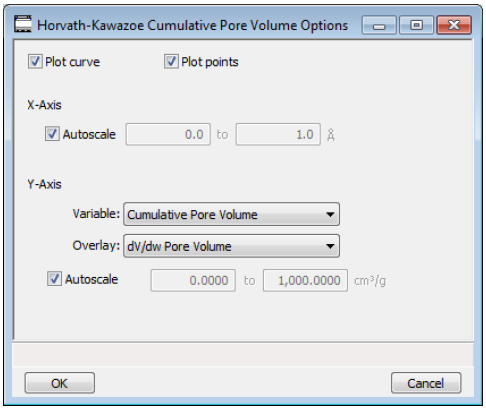
Horvath-Kawazoe Report Fields and Buttons Table (continued)

Field or Button	Description
	adsorptive. • **Density**. Enter the density per unit area of the adsorptive.
Select Reports	Select the types of reports to generate. Highlight the report, then click **Edit** to modify report parameters.
Smooth Differentials	See *" BJH Adsorption / Desorption Report Options" on page 8 - 10*.

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

HORVATH-KAWAZOE PLOT OPTIONS

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



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.

Horvath-Kawazoe Plot Options Fields and Buttons Table

Field or Button	Description
Autoscale	When enabled on a report parameters window, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y- ranges will be set so that all the data is shown. If *Autoscale* is not selected, the entered range is used.
Plot curve / Plot points	Select to plot points on the graph.

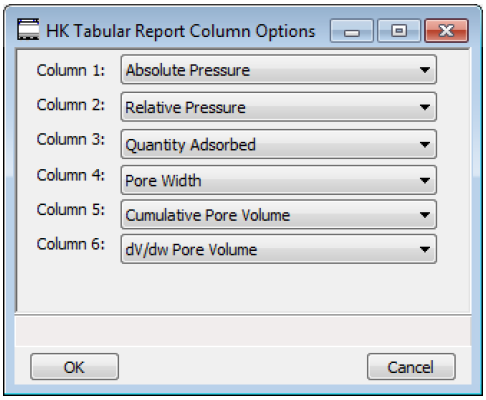
Horvath-Kawazoe Plot Options Fields and Buttons Table (continued)

Field or Button	Description
X-Axis / Y-Axis	• **X-Axis.** The x-axis field shows pore radius or diameter in angstroms or nanometers. • **Y-Axis.** The y-axis field shows the quantity of gas adsorbed. • **Variable.** Select a y-axis variable for the report. • **Overlay.** Select an option to overlay on the current report.

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

HORVATH-KAWAZOE TABULAR REPORT OPTIONS

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

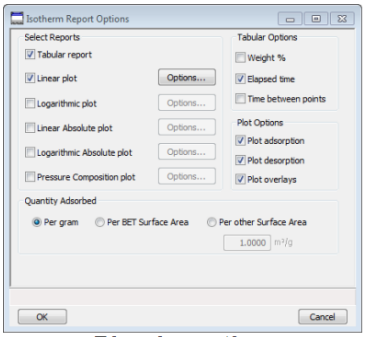


Highlight *H-K Tabular Report* in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit**. Select the data types to include in the report. *Column [n]* indicates the column order and data contents for the report.

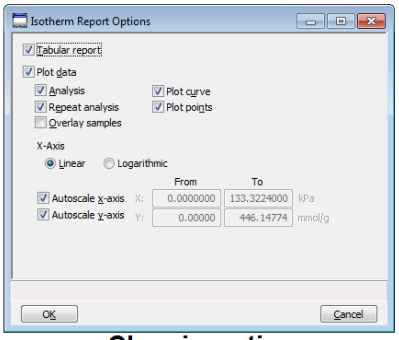
ISOTHERM REPORT OPTIONS

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

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



Physisorption



Chemisorption

Physisorption Isotherm Report Options Fields and Buttons Table

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

Physisorption Isotherm Report Options Fields and Buttons Table (continued)

Field or Button	Description
Tabular Options	Select the options to include on the report: • **Weight %.** Enter the mass percentage when plotting pressure composition • **Elapsed time.** Time elapsed during the analysis • **Time between points.** Time elapsed between points during the analysis

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

Chemisorption Isotherm Report Options Fields and Buttons Table

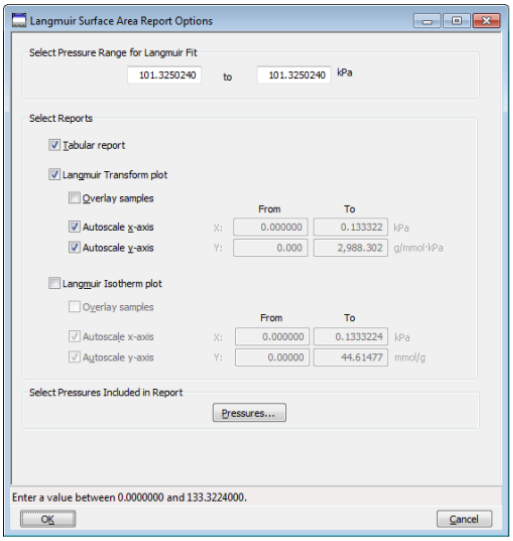
Field or Button	Description
Autoscale	• **Autoscale x-axis.** Linear x-axes begin at zero. Logarithmic x-axes begin at an appropriate value. The x-axis field shows the relative or absolute pressure. • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.
Plot data	Select each option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

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

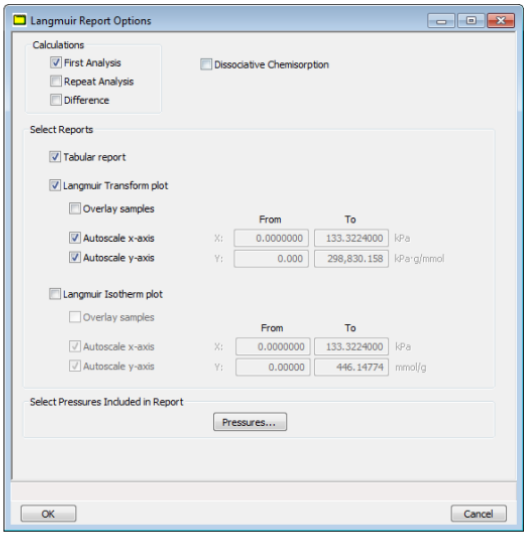
LANGMUIR REPORT OPTIONS

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

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



Physisorption



Chemisorption

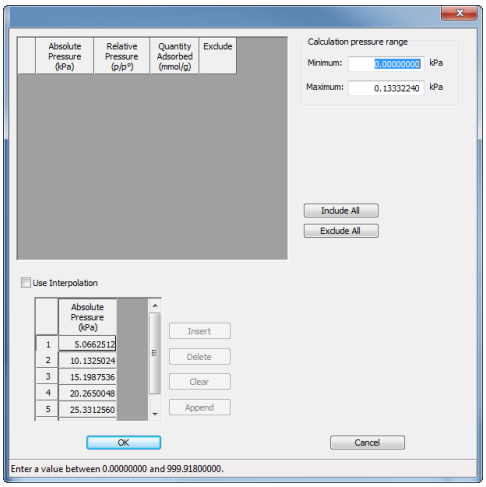
Langmuir Report Options Fields and Buttons Table

Field or Button	Description
Calculations (for chemisorption)	Select one or more of the calculation options to be used for analysis.

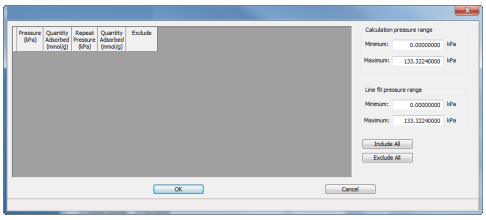
Langmuir Report Options Fields and Buttons Table (continued)

Field or Button	Description
Select Reports	• **Langmuir Transform Plot.** Use to generate a traditional Langmuir surface area plot used to determine monolayer volume constant ◦ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. ◦ **Autoscale y-axis.** The y-axis field shows Langmuir transformation. ◦ **Overlay samples.** Use to overlay sample files on the Langmuir transform plot. • **Langmuir Isotherm Plot.** Uses the Langmuir monolayer volume and constant to produce an isotherm. ◦ **Overlay samples.** Use to overlay sample files on the Langmuir isotherm plot. ◦ **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. ◦ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.

Langmuir Report Options Fields and Buttons Table (continued)

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

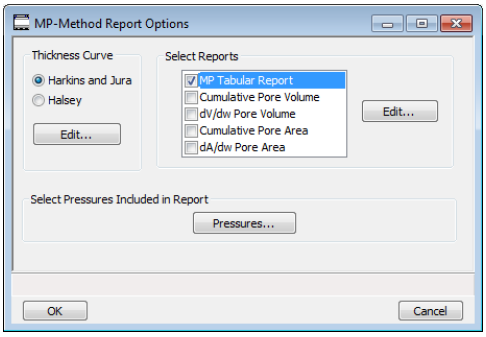
Langmuir Report Options Fields and Buttons Table (continued)

Field or Button	Description
Pressures (for chemisorption)	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Line fit pressure range.** Enter the minimum and maximum pressures for line fit. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Select Pressure Range for Langmuir fit (for physisorption)	Enter values to indicate the fitted pressure range.

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

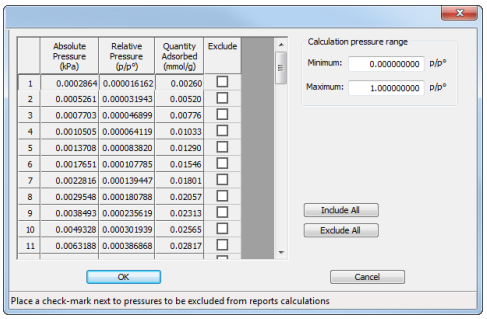
MP-METHOD REPORT OPTIONS

The *MP-Method Report Options* provides pore volume distributions for microporous materials by correlating quantity adsorbed with the thickness of the adsorbed layer as determined from a user-selected thickness curve.



Pore size can be expressed in angstroms or nanometers. Go to ***Options > Units*** to specify the unit.

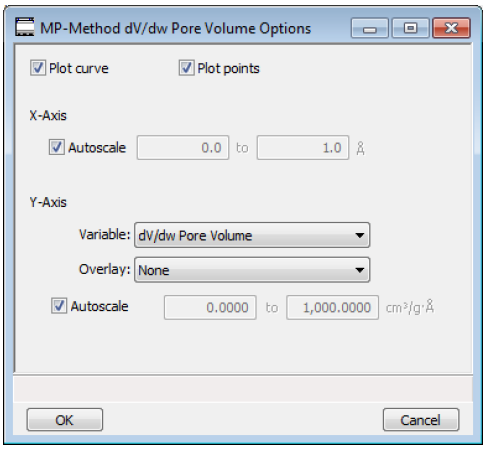
MP-Method Report Options Fields and Buttons Table

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

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

MP-METHOD PLOT REPORT OPTIONS

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



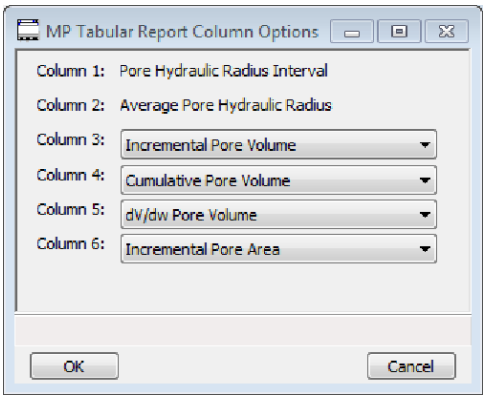
MP Method Plot Options Fields and Buttons Table

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

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

MP-METHOD TABULAR REPORT OPTIONS

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



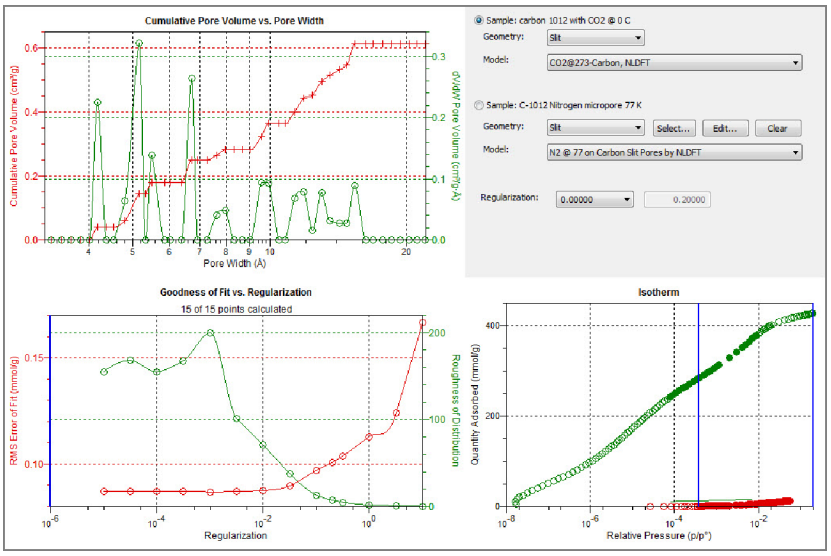
The MP Method reports hydraulic radius only. If Pore size in diameter is selected on the Unit Selection window, pore size in radius will be reports.

NLDFT ADVANCED PSD REPORT

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

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

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



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

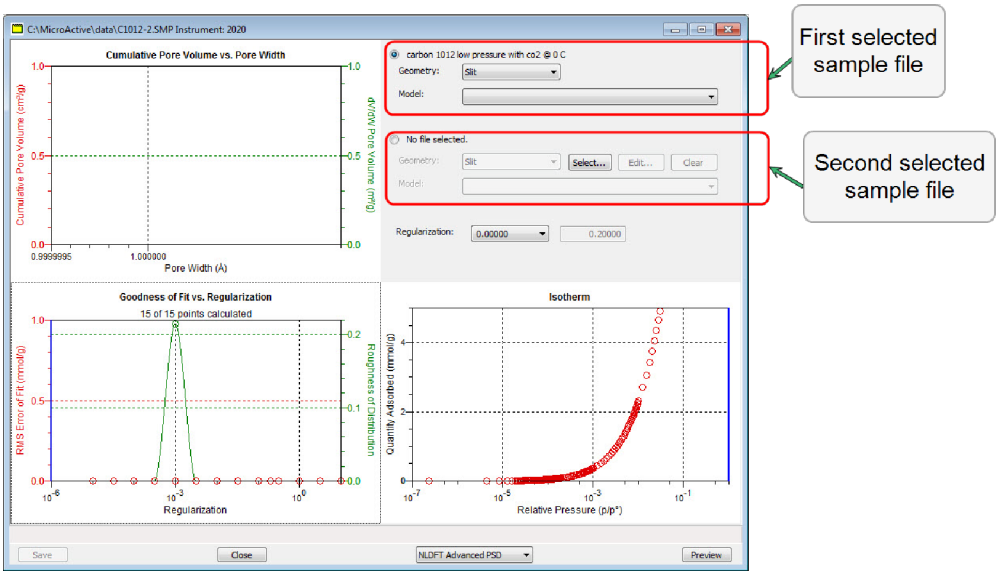
A second sample file and second model is used to compute a more accurate pore size distribution (PSD), which is shown in the upper left quadrant. Typically, the second sample file will have used the same sample material as the first sample file yet will have used a different analysis gas and temperature.

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

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

To run the NLDFT report:

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



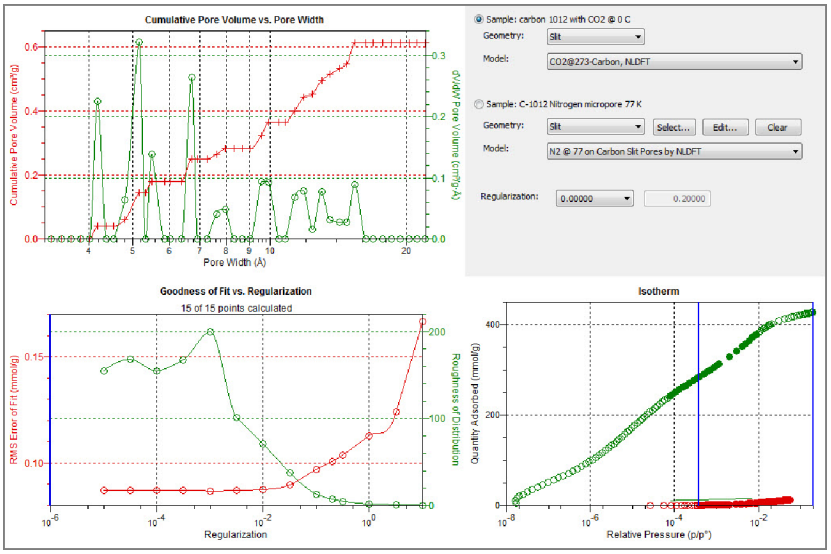
   a. Select the *Geometry* and *Model* from the drop-down lists for the first sample file.
   b. To select isotherm data points for calculation for the first sample file, ensure the option to the left of the first sample file description is selected. Slide the two blue bars on the isotherm graph to select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

3. To calculate data from the second sample file, click **Select** to locate and open the second sample file with a *Complete* status. Graphs for the second sample file display and the sample description displays as the second group box title in the upper right corner of the window.

a. Select the *Geometry* and *Model* from the drop-down lists for the second sample file.

b. To select isotherm data points for calculation for the second sample file, ensure the option to the left of the second sample file description is selected. Slide the two blue bars on the isotherm graph to select data points. Data are automatically calculated for both sample files.

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



NLDFT Advanced PSD Report Fields and Buttons Table

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

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

OPTIONS REPORT

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

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

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

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

Options reports cannot be edited.

SAMPLE LOG REPORT

This report provides information on:

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

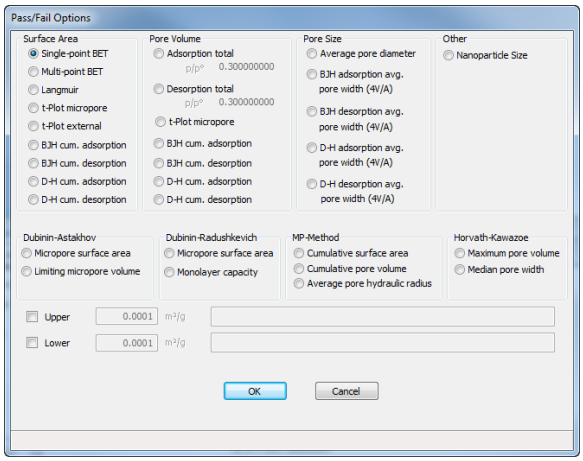
SUMMARY REPORT OPTIONS

Physisorption

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

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

Summary Report Fields and Buttons Table

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

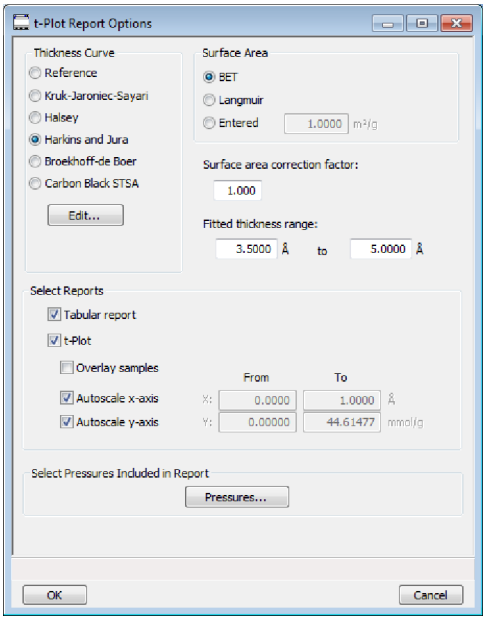
Summary Report Fields and Buttons Table (continued)

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

T-PLOT REPORT OPTIONS

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

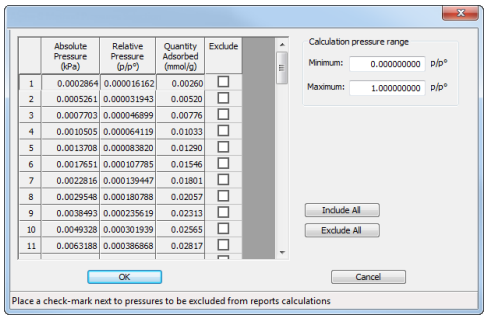
The *t-Plot* calculation allows quantitative analysis of the area and total volume ascribed to micropores. Matrix area (the area external to micropores) is directly determined and often proves to be a valuable way of characterizing complex mixed materials.



t-Plot Report Options Fields and Buttons Table

Field or Button	Description
Fitted thickness range	Enter the minimum and maximum thicknesses (in angstroms or nanometers) to include in the thickness curve. Go to ***Options > Units*** to specify default units.

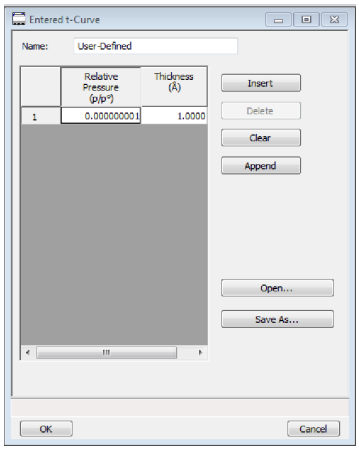
t-Plot Report Options Fields and Buttons Table (continued)

Field or Button	Description
Pressures	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.

Selected Reports	• **Tabular Report.** Use to have a tabular report of data generated.
	• **t-Plot.** Use to have a graphical representation of data generated.
	  ○ **Overlay samples.** Use to overlay sample files on the t-plot.
	  ○ **Autoscale x-axis.** The x-axis field shows the statistical thickness of the adsorbed film.
	  ○ **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.

Surface area correction factor	Enter the value to correct for surface areas that are not smooth. This brings the values for BET surface area and micropore surface area into accordance. For most samples, the default value of 1.000 is adequate.
Surface Area	Select the surface area value used for thickness calculations. BET is the most commonly used option.

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

Field or Button	Description
Thickness Curve	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Reference.** Select *Reference*, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory.  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. Use to modify the table contents. • **Insert.** Inserts one row above the selected row. • **Delete.** Deletes the selected row. • **Clear.** Clears all table entries and displays only one default value. • **Append.** Inserts one row at the end of the table. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed.

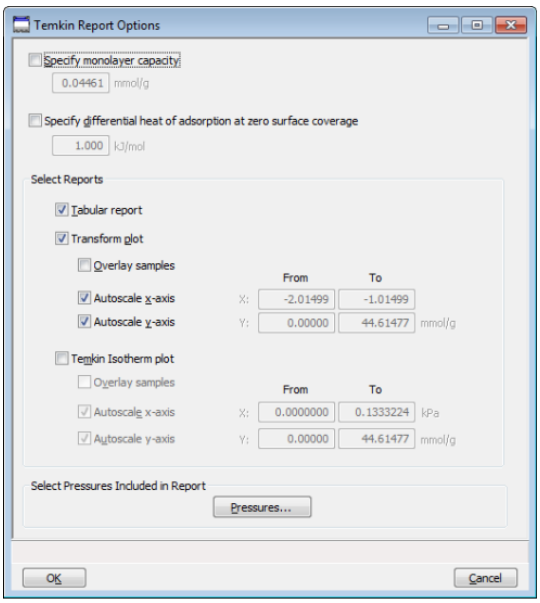
t-Plot Report Options Fields and Buttons Table (continued)

Field or Button	Description
t-Plot	Use to have a graphical representation of data generated. • **Overlay samples.** Use to overlay sample files on the t-plot. • **Autoscale x-axis.** The x-axis field shows the statistical thickness of the adsorbed film. • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed.

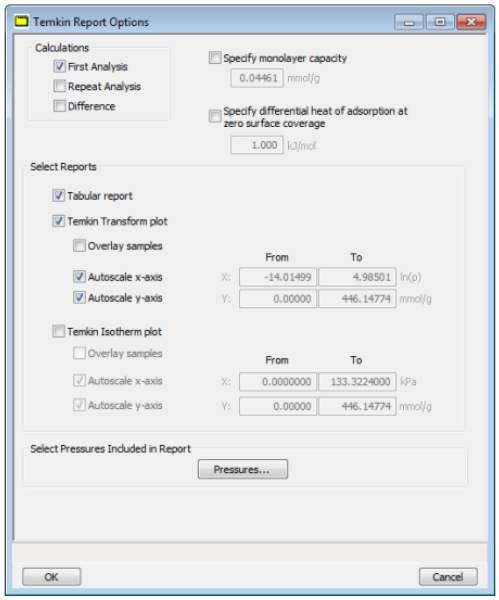
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

TEMKIN REPORT OPTIONS

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



Physisorption

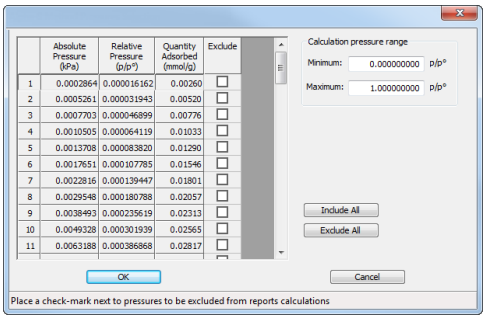
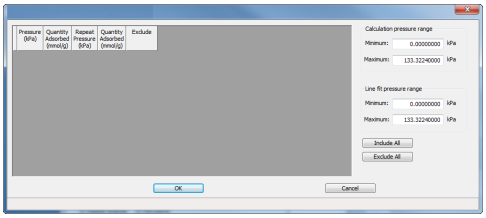


Chemisorption

Temkin Report Options Fields and Buttons Table

Field or Button	Description
Calculation Options (for chemisorption)	Select one or more of the calculation options to be used for analysis.

Temkin Report Options Fields and Buttons Table (continued)

Field or Button	Description
Pressures (for physisorption)	Use to select a pressure range for report calculations and points for exclusion from calculations.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Pressures (for chemisorption)	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Line fit pressure range.** Enter the minimum and maximum pressures for line fit. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.

Temkin Report Options Fields and Buttons Table (continued)

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

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

VALIDATION REPORT OPTIONS

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 Report Options for Chemisorption

File > Open > [Report file]

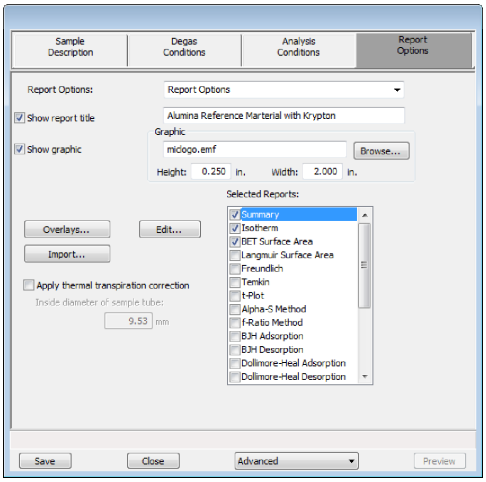
(or select the *Report Options* tab in the Sample Information file when using the *Advanced* presentation option)

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
- manually entered

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



Report Options Fields and Buttons Table

Field or Button	Description
Apply thermal transpiration correction	Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Always use thermal transpiration when performing micropore analysis. See *"Thermal Transpiration Correction" on page 12 - 47*. • **Inside diameter of sample tube.** Enabled when *Apply thermal transpiration correction* is selected. Enter the inside diameter of the sample tube used in the analysis. If filler rods are used, enter the filler rod capillary diameter of 1 mm instead.
Import (for physisorption)	Import up to 25 pore distribution data files. These datasets are shown only in BJH and Dollimore-Heal reports.
Name column	Displays a list of files in the selected directory.
Overlays	See *"Generate Multiple Graph 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. For BJH reports, BJH pore dimension can be calculated in pore width (w), pore radius (R) or pore diameter (D). Go to ***Options > Units*** to specify default calculations.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.

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

ADVANCED REPORT OPTIONS

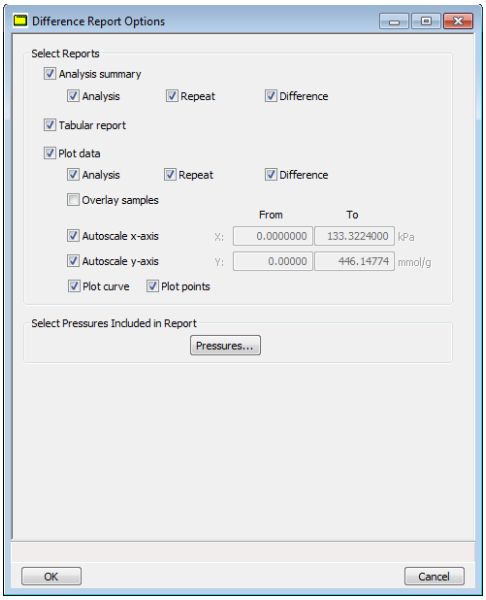
See *"Advanced Report Options" on page 8 - 3*

See *"Python Module - Advanced Reports" on page 11 - 1*

DIFFERENCE METHODS REPORT OPTIONS

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

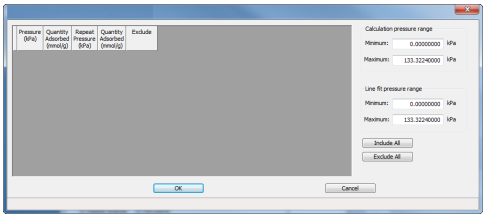
The *Difference Method Report* and the *Sinfelt Method Report* windows are identical unless otherwise specified.



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

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

Difference and Sinfelt Report Options Fields and Buttons Table

Field or Button	Description
Crystallite Size	Select the pore shape. • Spherical • Cubic • Entered
Pressures	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.  • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Line fit pressure range.** Enter the minimum and maximum pressures for line fit. • **Include All.** Select to include all pressure points in the table. • **Exclude All.** Select to exclude all pressure points in the table.
Selected Reports	• **Analysis summary.** Select the analysis summary options to display on the report. • **Tabular report.** Select to have a report of the pressure points generated. • **Plot data**. Select the plot data options to display on the report.

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

FREUNDLICH REPORT OPTIONS

See *"Freundlich Report Options" on page 8 - 33*

LANGMUIR REPORT OPTIONS

See *"Langmuir Report Options" on page 8 - 43*

Options Report

See *"Options Report" on page 8 - 54*

Sample Log Report

See *"Sample Log Report" on page 8 - 54*

Sinfelt and Difference Methods

The *Difference Method Report* and the *Sinfelt Method Report* windows are identical unless otherwise specified. See *"Difference Methods Report Options" on page 9 - 3*.

Temkin Report Options

See *"Temkin Report Options" on page 8 - 60*

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10 Report Options for Mercury Porosimetry

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**.

File > Open > [Report file]

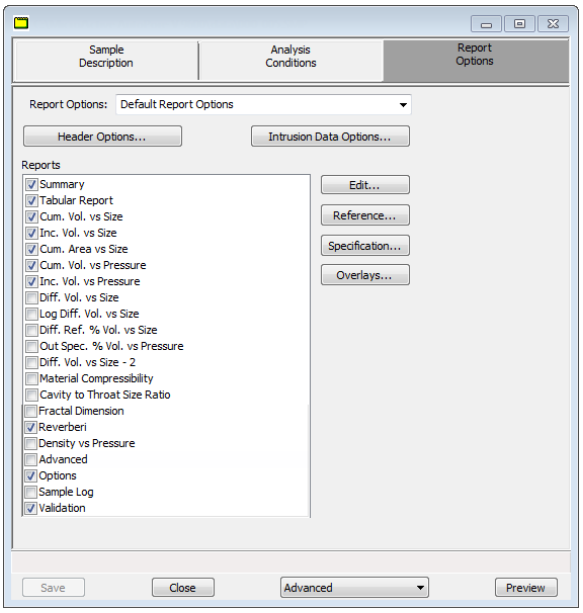
(or select the *Report Options* tab in the Sample Information file when using the *Advanced* presentation option)

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.

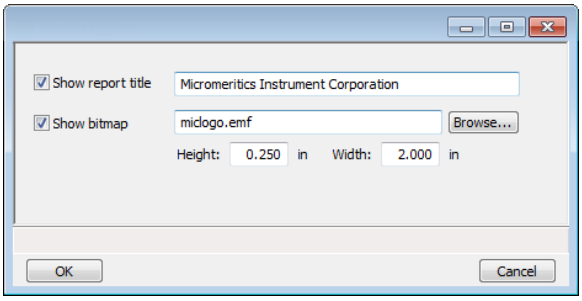
Reports can be generated for data:

- collected on a sample that has completed analysis
- manually entered

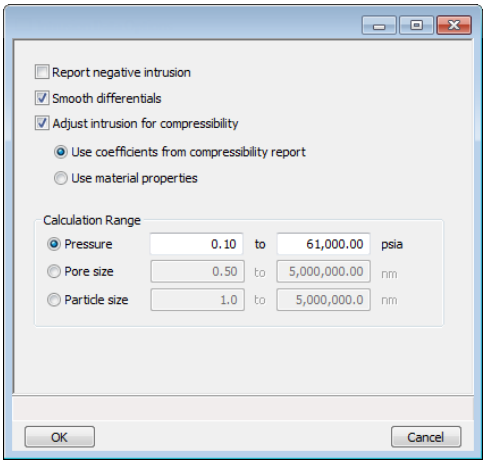
Customized report options files can be created then loaded into a sample file, allowing quick and easy generation of reports.



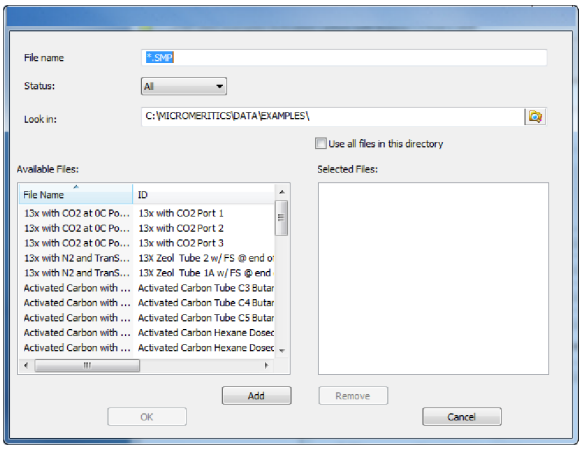
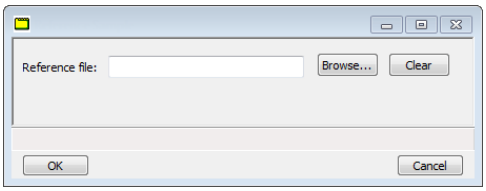
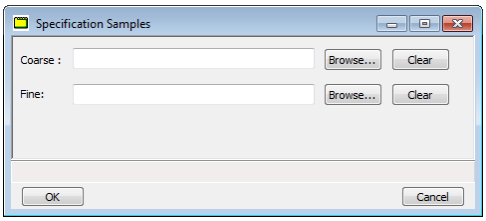
Report Options Fields and Buttons Table

Field or Button	Description
Edit	Click **Edit** to edit graph options. • **Plot Points.** Select to plot points on the graph.. • **Plot Curve.** Select to plot curves on the graph.. • **Show Histogram.** Select to show the graph as a histogram. When selected, the *Plot Points* and *Plot Curve* selections are disabled.
Header Options	 **Show report title**. Enter a report title to appear on the report header. **Show bitmap.** Displays the selected graphic on the report header. Click **Browse** to locate the graphic in either .BMP or .EMF format. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.

Report Options Fields and Buttons Table (continued)

Field or Button	Description
Intrusion Data Options	 **Report negative intrusion.** Select to report small incorrect polarities (negative intrusions or positive extrusions) which may indicate the presence of noise, improper blank correction, or instrument malfunction. **Smooth differentials.** Select to apply smoothing to any differentials reported in tables or graphs. **Use coefficients from compressibility report.** Select to have the application use the coefficients from the *Material Compressibility* report rather than from the *Material Properties*. **Use materials properties.** Select to have the application use the parameters from *Material Properties* rather than from the *Material Compressibility* report. **Calculation Range.** Select to indicate if reports should be limited in range by pore size, pressure, or particle size; then enter the range(s). The *from* value must be less than the *to* value.

Report Options Fields and Buttons Table (continued)

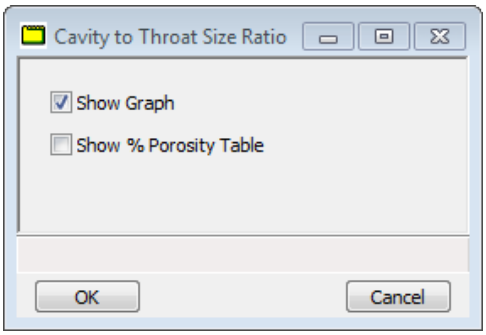
Field or Button	Description
Overlays	See *"Generate Multiple Graph Overlays" on page 7 - 29* 
Reference	Click **Reference** to select a sample file to compare analysis results with the current sample. 
Reports list box	Select the report names to include in the report.
Specification	Click **Specification** to select the sample files to be used for the boundaries of the coarse and fine specifications. This helps in determining if the results of the current sample are within the specified boundaries. 

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

ADVANCED REPORT OPTIONS - PYTHON MODULE

See *"Python Module - Advanced Reports" on page 11 - 1*

CAVITY TO THROAT SIZE RATIO REPORT OPTIONS



Select to show the graph or to show the % porosity table.

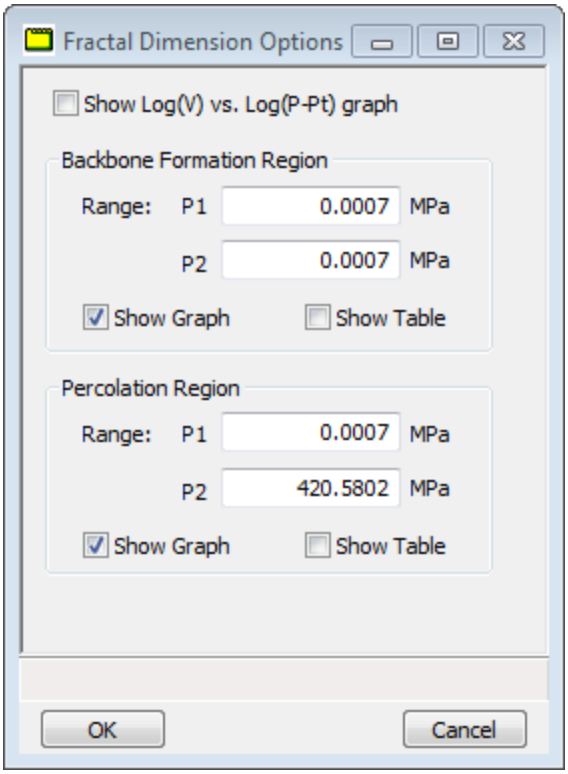
Cavity to Throat Size Ratio Fields and Buttons Table

Field or Button	Description
Show % Porosity Table	Displays percent porosity and pore throat ratio in table format.
Show Graph	Displays a graph that plots percent porosity on a linear scale on the x-axis and pore throat ratio on the Y-axis.

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

FRACTAL DIMENSION REPORT OPTIONS

The fractal dimensions can be shown as a graph, table, or both.The graph and table reports contain the fractal dimension and the RMS error to give an indication of the quality of the fit.



Fractal Dimension Report Fields and Buttons Table

Field or Button	Description
Backbone Formation Region	**Range.** Enter the pressure at which the calculations are to be performed. • **P1.** Enter the beginning pressure. • **P2.** Enter the ending pressure.
Show Graph	Displays the results in graph format. The graph plots pressure on a log scale on the x-axis and the intrusion volume reading as points on the y-axis, with the theoretical curve based on the calculated values overlaid.
Show Table	Displays the results in table format. The table displays pressure, intrusion volume, predicted intrusion volume, and error.

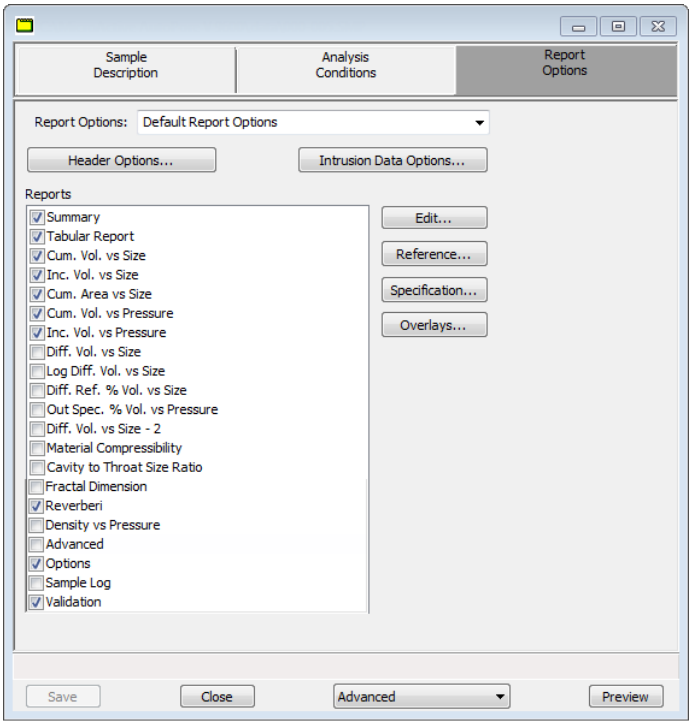
Fractal Dimension Report Fields and Buttons Table (continued)

Field or Button	Description
Percolation Region	**Range.** Enter the pressure at which the calculations are to be performed. • **P1.** Enter the beginning pressure. • **P2.** Enter the ending pressure.
Show Log(V) vs Log (P-Pt) graph	Select to generate an additional graph to help select linear range for calculations (P1, P2).

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

GRAPH REPORT OPTIONS

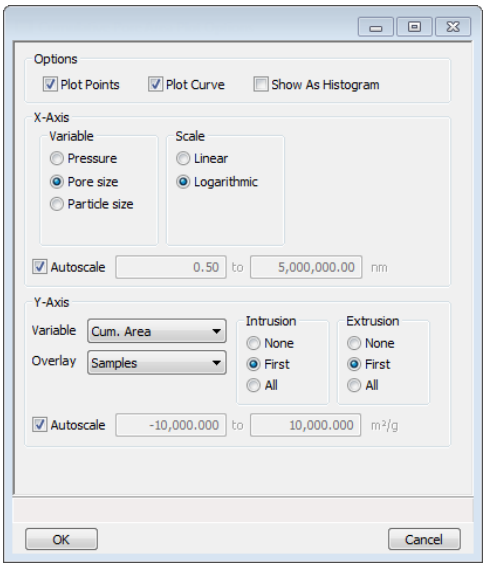
- Cumulative Area vs Size Plot Options
- Cumulative Volume vs Pressure Plot Options
- Cumulative Volume vs Size Plot Options
- Difference from Reference % Volume Plot Options
- Differential Intrusion Plot Options
- Differential Volume vs Size Plot Options
- Incremental Volume vs Pressure Plot Options
- Incremental Volume vs Size Plot Options
- Log Differential Intrusion Plot Options
- Out of Specification % Volume Plot Options



On the *Report Options* tab, highlight the graph report in the *Reports* group box then click one of the buttons to the right.

EDIT GRAPH REPORT OPTIONS

Highlight the graph report in the *Reports* list box and click **Edit**.



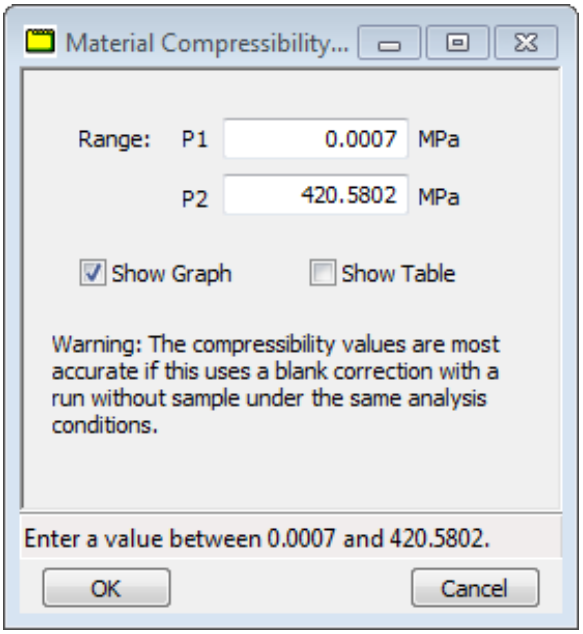
Graph Report Options Fields and Buttons Table

Field or Button	Description
Autoscale	When enabled on the report parameters windows, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y-ranges will be set so that all the data is shown. If *Autoscale* is not selected, the entered range is used.
Options	Select one or more graph display options. **Plot Points.** Select to plot the points on the graph. **Plot Curve.** Interpolated from data points. **Show As Histogram.** When selected, *Plot Points* and *Plot Curve* options are disabled.
Overlays button	Click to select sample files that contain data to be overlayed onto the selected plot.
Reference button	Click to select a sample file to compare analysis results of the current sample.
Specification button	Click to specify sample files to use for the boundaries of the coarse and fine specifications.
X-Axis group box	Select options for the x-axis. **Variable.** **Scale.**
Y-Axis	Select options for the y-axis. **Variable**. Select the y-axis variable from the drop-down list. **Overlay.** [*Optional*]. Select an option to overlay. **Intrusion / Extrusion.** Select the data points to plot. • **None.** No intrusion (or extrusion) data points • **First.** Points from the first intrusion (or extrusion) cycle • **All.** Include all intrusion (or extrusion) data points

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

MATERIAL COMPRESSIBILITY REPORT OPTIONS

The compressibility calculations can be shown as a graph, table, or both.The graph and table reports contain the linear and quadratic *c* compressibility values and the RMS error to give an indication of the quality of the fit. The linear and quadratic compressibility coefficients from this report can be copied into a *Material Properties* parameter file for use in future sample analyses with the same material.'



Material Compressibility Fields and Buttons Table

Field or Button	Description
Range	**P1.** Enter the beginning pressure. **P2.** Enter the ending pressure.
Show Graph	Displays the results in graph format. The graph plots pressure on a log scale on the x-axis and the volume compressed readings as points on the y-axis, with the theoretical curve based on the calculated values overlaid.
Show Table	Displays the results in table format. The table displays pressure, volume compressed, predicted volume compressed, and error.

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

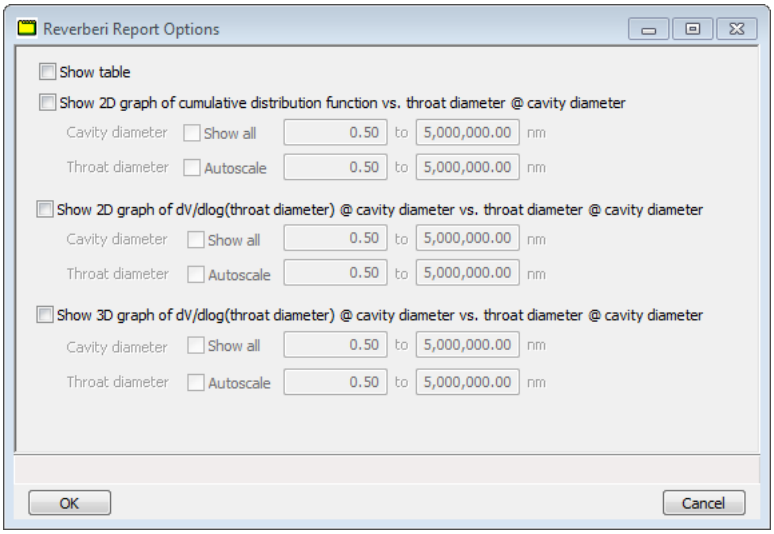
OPTIONS REPORT

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

- Analysis conditions
- Evacuation options
- High pressure options
- Low pressure options
- Material properties
- Mercury properties
- Penetrometer properties
- Reverberi options
- Sample information

Options reports cannot be edited.

REVERBERI REPORT OPTIONS



Reverberi Fields and Buttons Table

Field or Button	Description
Cavity diameter	Select *Show all* to display all diameters or enter a specific range to display.
Show 2D graph of cumulative distribution function vs. throat diameter @ cavity diameter	Select to display a 2D graph with this description.
Show 2D graph of dV/dlog (throat diameter) @ cavity diameter vs. throat diameter @ cavity diameter	Select to display a 2D graph with this description.
Show 3D graph of dV/dlog (throat diameter) @ cavity diameter vs. throat diameter @ cavity diameter	Select to display a 3D graph with this description.
Show table	Select to display a table in the report.
Throat diameter	Select *Autoscale* to autoscale the throat diameter or enter a specific range to display.

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

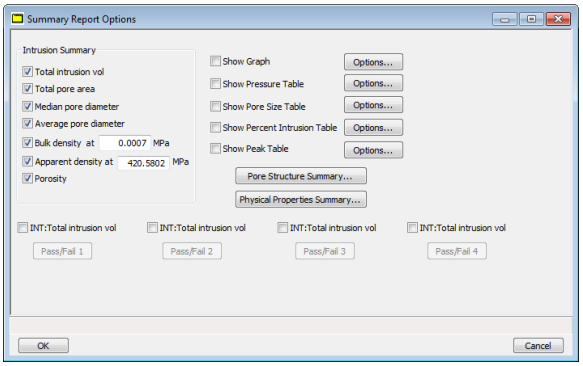
SAMPLE LOG REPORT

This report provides information on:

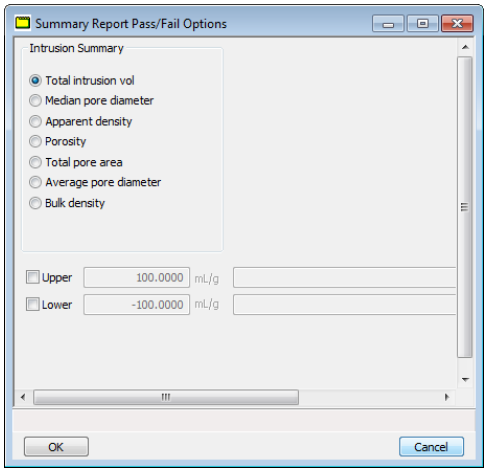
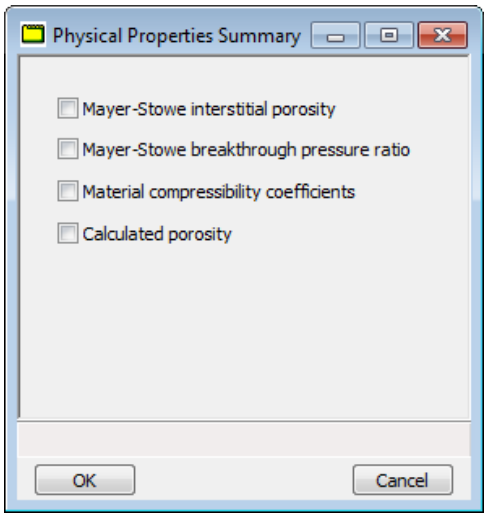
- Manual control operations performed during analysis
- Information entered using *Add Log Entry* on the sample file editor
- Warnings and / or errors which occurred during analysis

SUMMARY REPORT

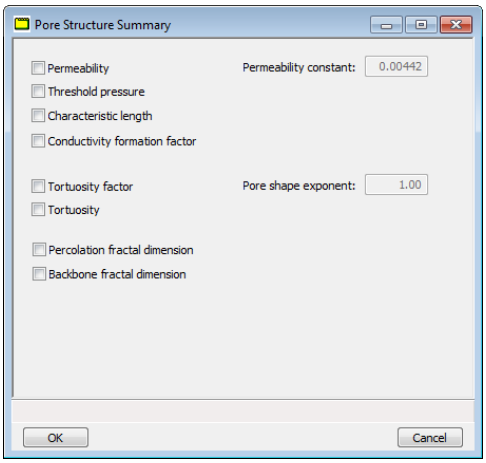
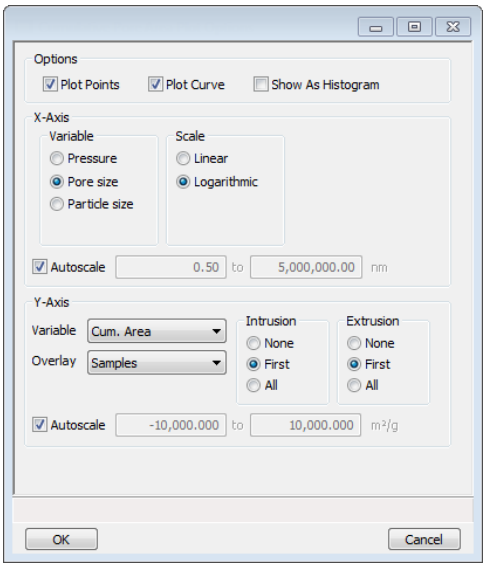
The *Summary Report* provides a condensed listing of selected data results.



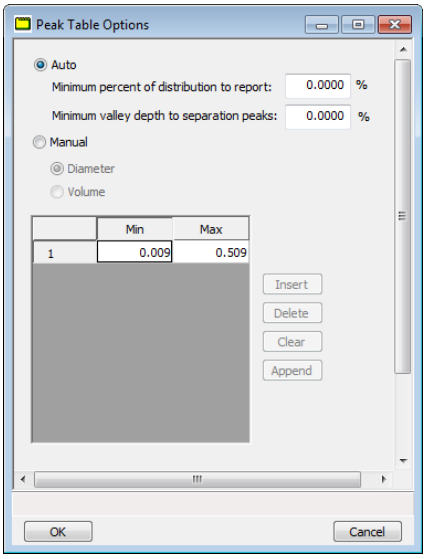
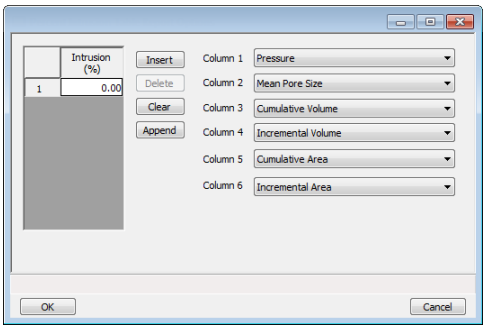
Summary Report Fields and Buttons Table

Field or Button	Description
INT: Total intrusion vol	Use to enable the **Pass/Fail** button. Click **Pass/Fail** to select pass/fail criteria options.  • **Upper / Lower.** Specify upper and lower limits for the selected parameter. A range can be left open by not selecting the limit. In the text box to the right of *Upper / Lower*, enter operator instructions to be displayed if a failure is encountered.
Intrusion Summary	Select the intrusion options to include in the report. If *Bulk Density* is selected, enter the pressure for the measurement. If the entered pressure is below the filling pressure, the filling pressure will be used in the report.
Physical Properties Summary	Select the physical properties to display on the report. 

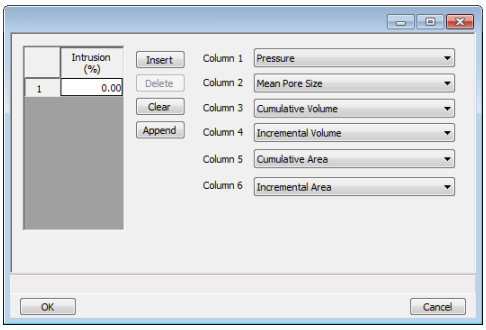
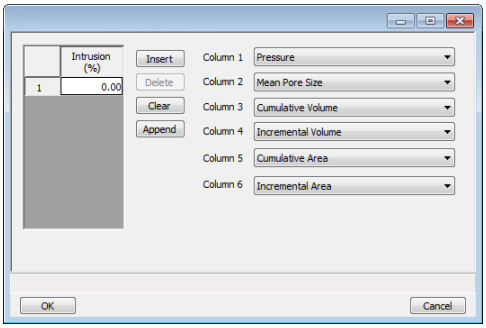
Summary Report Fields and Buttons Table (continued)

Field or Button	Description
Pore Structure Summary	Click to select the pore structure to be included in the report. If permeability is selected on the *Pore Structure Summary* window, enter a permeability constant in the *Permeability constant* field. If *Tortuosity* is selected, enter the pore shape exponent in the *Pore shape exponent* field. 
Show Graph	Select to display the report in graph format. Click **Options** to the right of *Show Graph* to select how the graph should display.  See *"Report Options" on page 6 - 14* for a description of fields and buttons on this window.

Summary Report Fields and Buttons Table (continued)

Field or Button	Description
Show Peak table	Select to display the peak table in the report. Click **Options** to the right of *Show Peak Table* to select how the graph should display.  **Auto.** Select to have the system automatically identify peaks based on the entered minimum valley depth to separation peaks. Enter the minimum settings in the text boxes. **Manual.** Select to manually enter the minimum and maximum diameter or volume for each peak in the table.
Show Percent Intrusion Table	Click **Options** to the right of *Show Percent Intrusion Table* to enter the percentile intrusion for the report. Use the drop-down fields to specify the data to appear in the specified columns for report generation. 

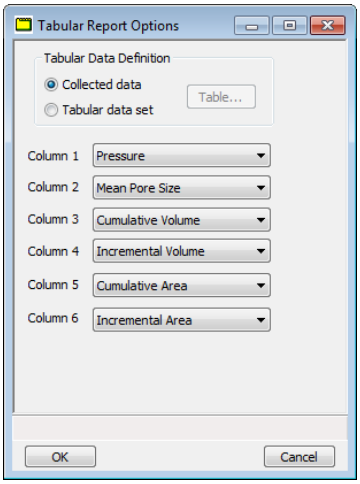
Summary Report Fields and Buttons Table (continued)

Field or Button	Description
Show Pore Size Table	Select to show pore size in the report. Click **Options** to select points to display. Use the drop-down fields to specify the data to appear in the specified columns for report generation. 
Show Pressure Table	Select to display pressure points in the report. Click **Options** to the right of *Show Pressure Table* to select points to display. Use the drop-down fields to specify the data to appear in the specified columns for report generation. 

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

TABULAR REPORT OPTIONS

Tabular reports display the numerical values for the data points. Up to six columns of data can be selected to display on the report.

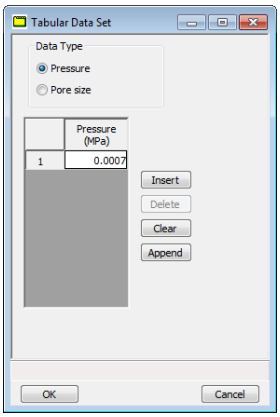


In the *Tabular Data Definition* group box, indicate select either *Collected data* or *Tabular data set* for this report.

Tabular Report Options Fields and Buttons Table

Field or Button	Description
Column [n]	Use the drop-down fields to specify the data to appear in the specified columns for report generation.

Tabular Report Options Fields and Buttons Table (continued)

Field or Button	Description
Tabular Data Definition	Indicate if the report should use: **Collected data.** Select if the report should use data points collected during analysis. Data are collected at equilibration points on or about the pressure points specified in the pressure table used for each analysis. **Tabular data set**. Select to have a table of specific pressure points included in tabular reports. Allows for the comparison of data from various runs, because it interpolates values from each sample run at the points specified in the table. When this option is selected, the **Table** button is enabled.  • **Data Type group box**. Select either *Pressure* or *Pore size* as the data type. • Click **Insert** to insert a data point immediately before the selected point. To complete the table quickly, enter the highest value in the set, then click **Insert** to enter points below that vale.

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

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11 Python Module - Advanced Reports

The mic Python module is automatically imported when running a user supplied script. The module provides access to data and provides support for summary, tabular, and graphical reports.

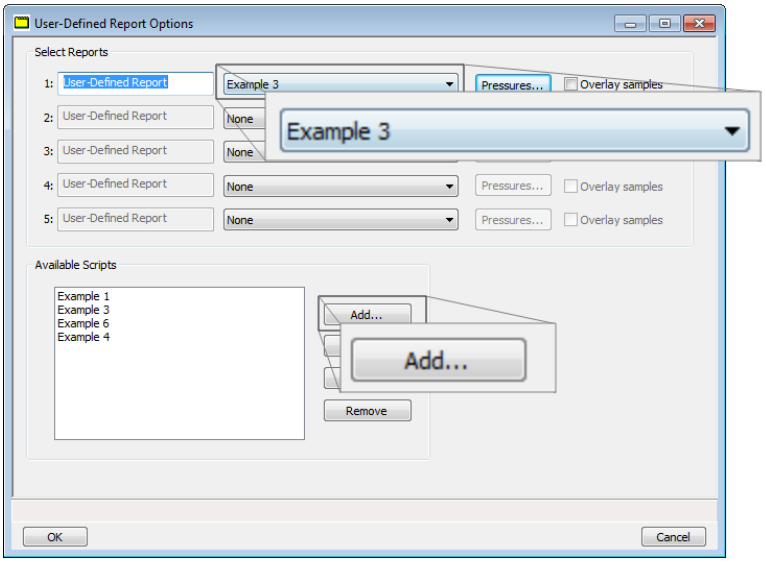
- **Summary reports.** Consist of summary sections, each containing a two-column table of label and value pairs. Summary reports are created with the *mic.summary* call.
- **Tabular reports.** Consist of one or more tables each containing one or more labeled columns of data.Tabular reports are created with the *mic.table* call.
- **Graphical reports**. Consist of a single graph with one or more curves on one or two y-axes. Graphical reports are created with the *mic.graph* call.

Calls for accessing the sample file data can be found in the *Mic Module Python Calls* section of this appendix. More advanced example python scripts are included in the analyzer software. Application specific discussions can be found on http://www.micro-report.com

The examples in this topic are also included as a part of the Micromeritics installation process and are located in the Scripts sub-directory.

Run a Script

1. Open a sample file with a *Complete* file status.
2. Select *Advanced* in the drop-down list at the bottom of the window.
3. Select the *Report Options* tab.
4. Highlight *Advanced* in the *Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add**. Locate and select one or more python scripts then click **Select**. The selected scripts become a part of the drop-down list in the *Available Scripts* section of the *Advanced Report Options* window.



6. In the *Selected Reports* section, select up to five Advanced reports in the drop-down lists. Use the **Pressures** button to include or exclude available pressures in the report.
7. Click **OK** to close the window.
8. Click **Preview** on the *Report Options* tab to view all reports selected in the previous window.

EDIT A SCRIPT

When a script is added, the code is stored within the application. If the script changes outside of the application, the script file will have to be re-added to the Advanced Report Options window for the changes to take affect.

Field or Button	Description
Add	Adds one or more scripts to the *Available Scripts* box. The added scripts then become available as options in the *Selected Reports* section.
Edit	Edits the script stored within the application but does not affect the original .py text file.
Remove	Removes the script from the *Available Scripts* box but does not affect original .py text file
Replace	Replaces the contents of the selected script however, the script name remains the same.

REMOVE A SCRIPT

Select the script in the *Available Scripts* box then click **Remove**. The script is removed from the application however, the original .py text file is not affected.

SUMMARY REPORT

This script produces a summary report with two summaries:

```
import mic
mic.summary( "My Summaries" )

mic.summary.add( "Summary A",
                 ["label 1:", "label 2:", "label 3:"],
                 ["val1", "val2", "val3"] )


mic.summary.add( "Summary B",
                 ["label 4:", "label 5:", "label 6:"],
                 ["val4", "val5", "val6"] )
```

The result is:

Summary A
label 1: val1
label 2: val2
label 3: val3

Summary B
label 4: val4
label 5: val5
label 6: val6

TABULAR REPORT

If more than one column is required, the call *mic.table* is employed. This script produces a tabular report consisting of two tables. **NOTE**: This script uses the Python package "numpy" and c-style formatting of the numerical values.

```
import mic
import numpy as np

mic.table("My Tables")

mic.table.addtable( "My set A" )
mic.table.addcolumn( "x", ["1.0", "2.0", "3.0"] )
mic.table.addcolumn( "y", ["0.5", "1.0", "1.5"] )

x1 = 0.2
x2 = 0.5
x3 = 3.14159/2
mic.table.addtable( "My set B" )
mic.table.addcolumn( "x", ["%8.3f" % x1,
                           "%8.3f" % x2,
                           "%8.3f" % x3 ] )

mic.table.addcolumn( "sin(x)", ["%8.3f" % np.sin(x1),
                                "%8.3f" % np.sin(x2),
                                "%8.3f" % np.sin(x3)] )


mic.table.addcolumn( "cos(x)", ["%8.3f" % np.cos(x1),
                                "%8.3f" % np.cos(x2),
                                "%8.3f" % np.cos(x3)] )
```

The result is:

My set A

x	y
1.0	0.5
2.0	1.0
3.0	1.5

My set B

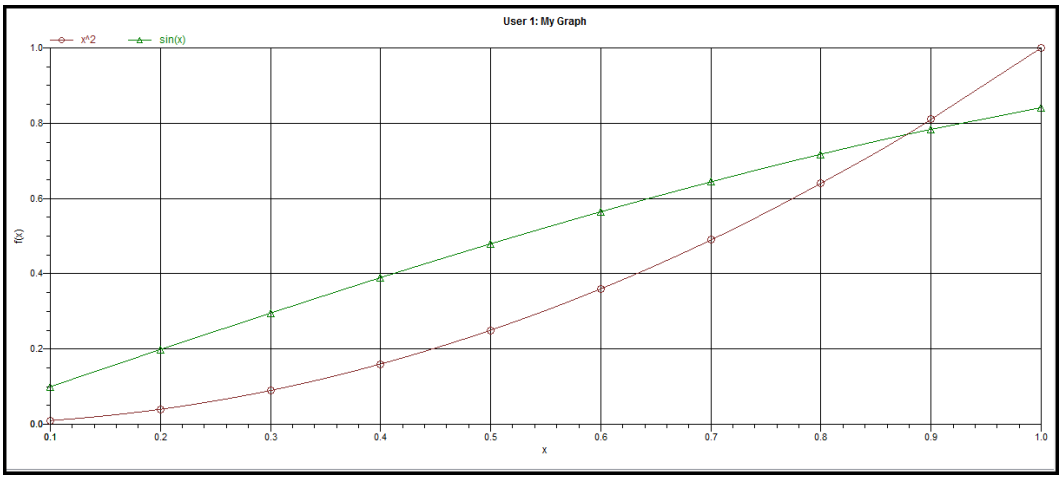
x	sin(x)	cos(x)
0.200	0.199	0.980
0.500	0.479	0.878
1.571	1.000	0.000

GRAPHIC REPORT

This script is an example of the mic module producing a graph with two curves:

```
import mic
import numpy as np

mic.graph( 'My Graph', 'x', 'f(x)' )

myx = np.array( [0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0 ] )
mic.graph.add( 'x^2', myx, myx*myx, marker='o' )
mic.graph.add( 'sin(x)', myx, np.sin(myx), marker='^' )
```

The results are:



ACQUIRING BASIC INFORMATION

Physisorption

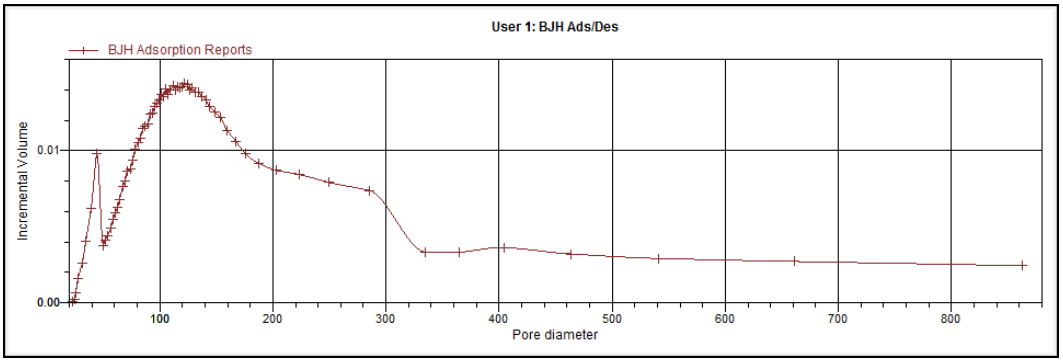
This script produces a graph of the adsorption and desorption isotherms for both relative and absolute pressure, and prints summaries of the sample information and the adsorptive properties.

To acquire the adsorption isotherm and other basic information about the sample being edited, the calls *mic.isotherm*, *mic.sample_information* and *mic.adsorptive_data* are applied.

Note the calls to *mic.isotherm* and *mic.adsorptive_data* above are each returning results as a list with elements of varying return type.

```
import mic

prel, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('rel')
mic.graph( 'Graphical Report 1', 'Rel. Press', 'Quantity Adsorbed' )
mic.graph.add( 'Sample isotherm', prel, qads )

pabs, qads, n_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('abs')

mic.graph( 'Graphical Report 2' 'Abs. Press', 'Quantity Adsorbed')
mic.graph.add('Sample Isotherm', pabs, qads)

mass = mic.sample_information('sample mass' )
Tanl = mic.sample_information('analysis temperature' )
dens = mic.sample_information('sample density')

mic.summary( "Sample Information" )
mic.summary.add( "Sample Information:",
                 [ "Number of adsorption points:",
                   "Warm Free space:",
                   "Cold Free space:" ,
                    "Sample mass (g):",
                    "Description:",
                    "Analysis Temp:",
                    "Sample Density (g/cm^3):" ],
                 [ "%8.3f" % n_ads,
                   "%8.3f" % warm_fs,
                   "%8.3f" % cold_fs,
                   "%8.3f" % mass,
                   desc,
                   "%8.3f" % Tanl,
                   "%8.3f" % dens ] )

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()

mic.summary.add( "Adsorptive Data",
                 [ "Cross Sectional Area",
                   "Hard Sphere Diameter",
                   "Density Conversion Factor",
                   "Molecular Weight",
                   "Analysis gas"],
                 [ "%8.3f" % csa,
                   "%8.3f" % hsd,
                   "%8.3f" % dcf,
                   "%8.3f" % mol_weight,
                   analysis_gas  ]  )
```

The result is:



Chemisorption

This script produces a graph of the primary, repeat, and difference isotherms; and prints summaries of the sample information and the adsorptive properties.

To acquire the adsorption isotherm and other basic information about the sample being edited, the calls *mic_chem.isotherm*, *mic.sample_information* and *mic.adsorptive_data* are applied.

Note the calls to *mic_chem.isotherm* and *mic.adsorptive_data* above are each returning results as a list with elements of varying return type.

```
import mic

p_primary,    q_primary    = mic.chem_isotherm('primary')
p_repeat,     q_repeat     = mic.chem_isotherm('repeat')
p_difference, q_difference = mic.chem_isotherm('difference')
mic.graph( 'Graphical Report 1', 'Abs. Press', 'Quantity Adsorbed')
mic.graph.add('Primary', p_primary   , q_primary)
mic.graph.add('Repeat', p_repeat    , q_repeat)
mic.graph.add('Difference', p_difference, q_difference)

mic.summary( "Sample Information" )
mic.summary.add( "Sample Information:",
                 [ "Ambient Free space (cm^3):",
                   "Analysis Free space (cm^3):" ,
                    "Sample mass (g):",
                    "Description:",
                    "Analysis Temp (K):",
                    "Sample Density (g/cm^3):" ],
                 [ "%8.3f" % mic.sample_information('ambient freespace'),
                   "%8.3f" % mic.sample_information('analysis freespace'),
                   "%8.3f" % mic.sample_information('sample mass'),
                   mic.sample_information('sample description'),
                   "%8.3f" % mic.sample_information('analysis temperature'),
```

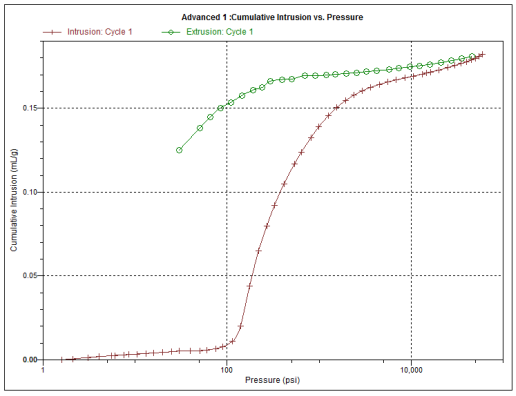
```
                    "%8.3f" % mic.sample_information('sample density') ] )

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()

mic.summary.add( "Adsorptive Data",
                 [ "Cross Sectional Area",
                   "Hard Sphere Diameter",
                   "Density Conversion Factor",
                   "Molecular Weight",
                   "Analysis gas"],
                 [ "%8.3f" % csa,
                   "%8.3f" % hsd,
                   "%8.3f" % dcf,
                   "%8.3f" % mol_weight,
                   analysis_gas  ]  )
```

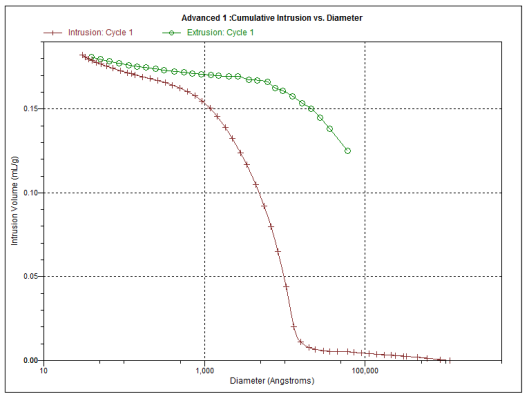
Mercury Porosimetry

This script produces a graph of the intrusion and extrusion data, and a graph of the corresponding distribution of pores. It applies the mic module python calls mic.intrusion and mic.extrusion.

```
import mic

xdat1, ydat1 = mic.intrusion('pressure', 1)
xdat2, ydat2 = mic.extrusion('pressure', 1)
mic.graph( 'Cumulative Intrusion vs. Pressure',
           'Pressure (psi)', 'Cumulative Intrusion (mL/g)', xlinear = False )
mic.graph.add( 'Intrusion: Cycle 1', xdat1, ydat1 )
mic.graph.add( 'Extrusion: Cycle 1', xdat2, ydat2 )

xdat3, ydat3 = mic.intrusion('diameter', 1)
xdat4, ydat4 = mic.extrusion('diameter', 1)
mic.graph( 'Cumulative Intrusion vs. Diameter',
           'Diameter (Angstroms)', 'Intrusion Volume (mL/g)', xlinear = False)
mic.graph.add( 'Intrusion: Cycle 1', xdat3, ydat3 )
mic.graph.add( 'Extrusion: Cycle 1', xdat4, ydat4 )
```

The results are:





The following script applies the generic mic module python calls mic.sample_information and mic.report and also applies the AutoPore application specific calls mic.material_properties, and mic.mercury_ properties. Three summaries are produced:

- Sample Information
- Material Mercury Properties
- Intrusion Summary Results

```
import mic

mic.summary( "Summaries" )

mic.summary.add( "Sample Information:",
    [ "Description:",
      "Sample mass (g):",
      "Assembly mass (g):",
      "Penetrometer mass (g):"],
    [ mic.sample_information("sample description"),
      "%8.3f" % mic.sample_information("sample mass"),
      "%8.3f" % mic.sample_information("assembly mass"),
      "%8.3f" % mic.sample_information("penetrometer mass") ] )

mic.summary.add( "Material & Mercury Properties",
    [ "Material name:",
      "BET surface area (m^2/g):",
      "Mercury Density (g/ml):",
      "Mercury Surface Tension (dynes/cm):",
      "Advancing Contact Angle (degrees):",
      "Receding Contact Angle (degrees):" ],
    [ mic.material_properties("material name"),
      "%8.3f" % mic.material_properties("bet surface area"),
      "%8.3f" % mic.mercury_properties("density"),
      "%8.3f" % mic.mercury_properties("surface tension"),
      "%8.3f" % mic.mercury_properties("advancing contact angle"),
```

```
      "%8.3f" % mic.mercury_properties("receding contact angle") ] )

mic.summary.add( "Intrusion Summary Results",
    [ "Total intrusion volume (mL/g):",
      "Pore area (m^2/g):",
      "Bulk density (g/mL):",
      "Apparent density (g/mL):",
      "Median diameter by volume (Angstroms):",
      "Median diameter by area (Angstroms):",
      "4 V/A average diameter (Angstroms):",
      "Porosity (%):",
      "Tortuosity:",
      "Tortuosity factor:",
      "Permeability (mdarcy):",
      "Permeability constant:",
      "Break-through pressure ratio:",
      "linear compressibility coefficient (1/psi):",
      "quadratic compressibility coefficient (1/psi^2):" ],
    [ "%8.3f" % mic.report("hgsum", "total intrusion volume"),
      "%8.3f" % mic.report("hgsum", "pore area"),
      "%8.3f" % mic.report("hgsum", "bulk density"),
      "%8.3f" % mic.report("hgsum", "apparent density"),
      "%8.3f" % mic.report("hgsum", "median diameter by volume"),
      "%8.3f" % mic.report("hgsum", "median diameter by area"),
      "%8.3f" % mic.report("hgsum", "4 V/A average diameter"),
      "%8.3f" % mic.report("hgsum", "porosity"),
      "%8.3f" % mic.report("hgsum", "tortuosity"),
      "%8.3f" % mic.report("hgsum", "tortuosity factor"),
      "%8.3f" % mic.report("hgsum", "permeability"),
      "%8.3f" % mic.report("hgsum", "permeability constant"),
      "%8.3f" % mic.report("hgsum", "break-through pressure ratio"),
      "%8.3f" % mic.report("hgsum", "linear compressibility coefficient"),
      "%8.3f" % mic.report("hgsum", "quadratic compressibility coefficient")])
```

The results are:

Sample Information:

Description:	Clay
Sample mass (g):	2.110
Assembly mass (g):	140.390
Penetrometer mass (g):	62.379

Material & Mercury Properties

Material name:	Garnet
BET surface area (m^2/g):	200.000
Mercury Density (g/ml):	13.533
Mercury Surface Tension (dynes/cm):	485.000
Advancing Contact Angle (degrees):	130.000
Receding Contact Angle (degrees):	130.000

Intrusion Summary Results

Total intrusion volume (mL/g):	0.182
Pore area (m^2/g):	12.041
Bulk density (g/mL):	1.833
Apparent density (g/mL):	2.751
Median diameter by volume (Angstroms):	5595.058
Median diameter by area (Angstroms):	53.917
4 V/A average diameter (Angstroms):	605.072
Porosity (%):	33.384
Tortuosity:	25.407
Tortuosity factor:	1.853
Permeability (mdarcy):	0.729
Permeability constant:	0.004
Break-through pressure ratio:	6.272
linear compressibility coefficient (1/psi):	-0.000
quadratic compressibility coefficient (1/psi^2):	0.000

ACQUIRING REPORT RESULTS

Physisorption

aSample file report results may be accessed using the *mic.report* call. This script prints a summary of the results of the t-plot and BET reports.

```
import mic

sa   = mic.report("bet",  "surface area")
c    = mic.report("bet",  "bet constant")
vm   = mic.report("bet",  "monolayer capacity")
esa  = mic.report("tplot", "external surface area")
vol  = mic.report("tplot", "micropore volume")

mic.summary( "BET and T-plot Results" )

mic.summary.add( "Report Results",
                 [ "bet surface area",
                   "bet constant",
                   "bety 6" ,
```

```
                    "tplot external surface area",
                    "tplot micropore volume"],
                 [ "%10.5f" % sa,
                   "%10.5f" % c,
                   "%10.5f" % vm,
                   "%10.5f" % esa,
                   "%10.5f" % vol ] )
```

Acquiring the results from a pore-distribution report such as the BJH method is done in a similar way as in the previous script except the return values from the *mic.report* call are slightly different since they involve lists of data. For example,

```
import mic
xdat, ydat, desc = mic.report('bjhads' ,'incremental distribution' )
mic.graph( 'BJH Ads/Des', 'Pore diameter', 'Incremental Volume' )
mic.graph.add( desc, xdat, ydat )
```

See ***"Mic Module Python Calls" on page 11 - 18***for a more complete description of the usage and scope of the *mic.report* call.

The result is:

Report Results

bet surface area	796.36286
bet constant	137786.85871
bet monolayer capacity	182.96348
tplot external surface area	416.38843
tplot micropore volume	0.17931

ACQUIRING OVERLAY SAMPLE DATA

Physisorption

The call to obtain overlay sample data is similar to the calls for the primary sample. This script involves two overlay sample files.

The calls to obtain adsorptive data and report results for an overlay sample file using *mic.report* and *mic.adsorptive_data* have a very similar interface as the *mic.overlay call*, and a summary of their usage is shown in the example in this topic.

```
import mic

p, q, n, fsw, fsc, mass, desc = mic.isotherm('rel')
p1, q1, n1, fsw1, fsc1, mass1, desc1 = mic.overlay( 1, 'rel')
p2, q2, n2, fsw2, fsc2, mass2, desc2 = mic.overlay( 2, 'rel')

mic.graph( 'Three Sample Isotherms',
```

```
            'Rel. Press',
            'Quantity Adsorbed (cm^3/g)' )

mic.graph.add( 'Primary Isotherm ', p, q )
mic.graph.add( 'Overlay Isotherm 1', p1, q1 )
mic.graph.add( 'Overlay Isotherm 2', p2, q2 )

mic.summary( "A summary report" )

mic.summary.add( "Two samples",
                 [ "Primary Sample:",
                   "Overlay Sample 1:",
                   "Overlay Sample 2:" ],
                 [ desc,
                   desc1,
                   desc2] )
```

To enable the use of overlay data in the Advanced reports, the following two actions must be taken prior to running the script. Instructions for each of the following actions are provided below.

- Sample files to overlay must be selected, and
- The *Overlay samples* checkbox on the *Advanced Report Options* window must be selected

Chemisorption

The call to obtain overlay sample data is similar to the calls for the primary sample. This script involves two overlay sample files.

The calls to obtain adsorptive data and report results for an overlay sample file using *mic.report* and *mic.adsorptive_data* have a very similar interface as the *mic.chem.overlay call*, and a summary of their usage is shown in the example in this topic.

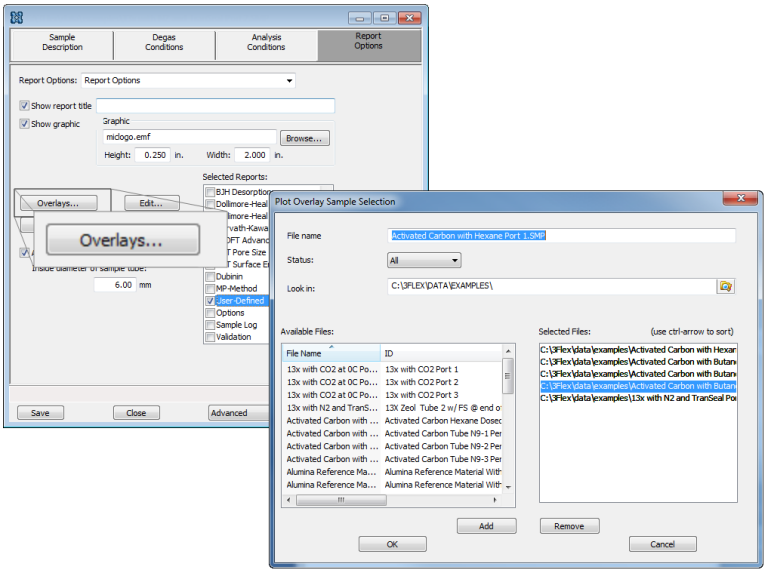
```
import mic


p0, q0    = mic.chem_isotherm('primary')
p0r,q0r = mic.chem_isotherm('repeat')
p1, q1 = mic.chem_overlay(1,'primary')
p1r, q1r = mic.chem_overlay(1,'repeat')
p2, q2 = mic.chem_overlay(2,'primary')
p2r, q2r = mic.chem_overlay(2,'repeat')
mic.graph( 'Graphical Report 1', 'Abs. Press', 'Quantity Adsorbed')
mic.graph.add('prim 0',p0,q0)
mic.graph.add('rep 0',p0r, q0r)
mic.graph.add('prim 1',p1,q1)
mic.graph.add('rep 1',p1r, q1r)
```

```
mic.graph.add('prim 2',p2,q2)
mic.graph.add('rep 2',p2r, q2r)

mic.summary( "A summary report" )

mic.summary.add( "Sample and Two Overlays",
                 [ "Primary Sample:",
                   "Overlay Sample 1:",
                   "Overlay Sample 2:" ],
                 [  mic.sample_information('sample description'),
                    mic.sample_information('sample description',1),
                    mic.sample_information('sample description',2)  ] )
```

ENABLE THE USE OF OVERLAY DATA

1. On the *Report Options* tab, click **Overlays**.

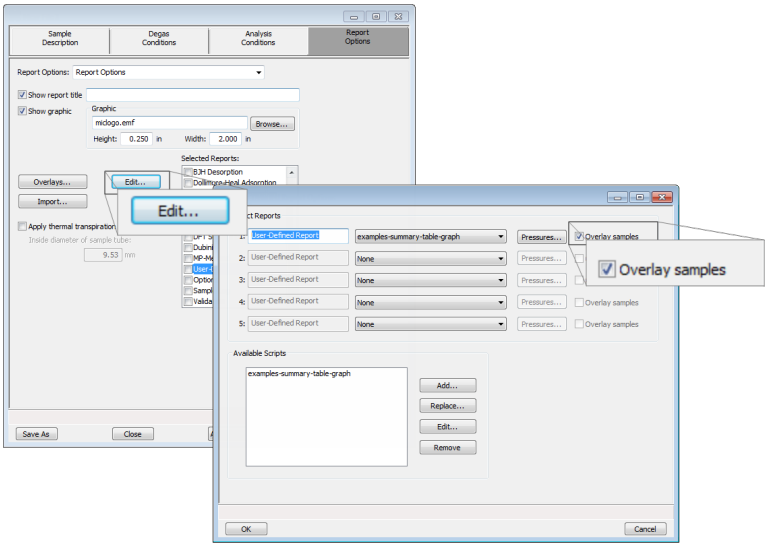
2. On the *Plot Overlay Sample Selection* window, use one of the following options to move up to 25 files from the *Available Files* box to the *Selected Files* box:



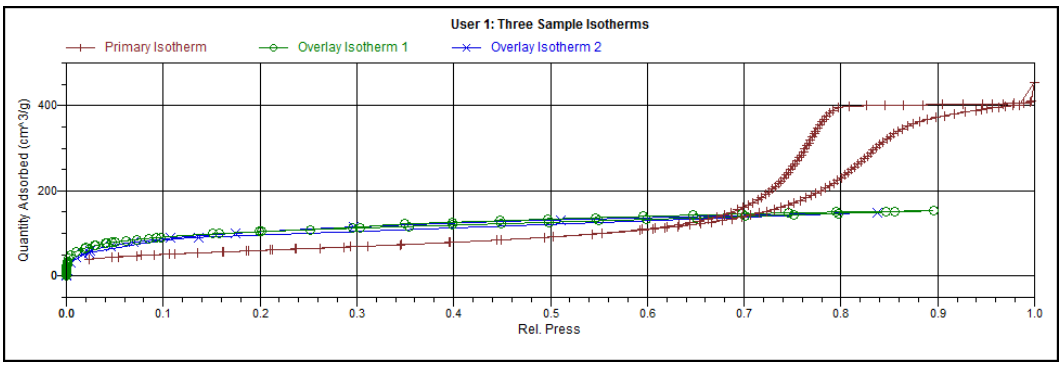
   - Double click a file name in the *Available Files* box to move the file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, double click the file name in the *Selected Files* box, or
   - Select a file name in the *Available Files* box. Click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

3. Click **OK**.

4. On the *Report Options* tab, highlight *Advanced* in the *Selected Reports* list box.
5. Click **Edit** to the left of the *Selected Reports* list box.
6. Select the *Overlay samples* checkbox to the right of the selected report.
7. Click **OK**.
8. Run the script using the instructions found in *"Enable the Use of Overlay Data" above*.



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

ACQUIRING METAL COMPOSITION DATA

Chemisorption

The call to obtain information about active metals in a chemisorption sample is *mic.metal_composition*. Specifically, this call provides access to the data shown in the table of the *Active Metals* window. With no arguments specified, the call returns a list of all the active metals in the sample. When called with a metal specified, the method returns an associative array (python dictionary) of the metal's properties. With both the metal and property specified, the call returns the value for the specified metal property. The following example script illustrates these three usage patterns.

```
import mic
import pprint as pp

mnames = mic.metal_composition()
mic.summary( "Metal Composition:" + pp.pformat( mnames ) )

mprops = sorted( mic.metal_composition( mnames[0] ).items() )
mkeys = []
mvals = []
for k, v in mprops :
    if ( 'cross sectional area' in k ) :
        mkeys.append( k + ' (nm^2)' )
    elif ( 'atomic weight' in k ) :
        mkeys.append( k + ' (amu)' )
    elif ( 'density' in k ) :
        mkeys.append( k + ' (g/cm^3)' )
    else :
        mkeys.append( k )
    mvals.append("%8.3f" % v )
mic.summary.add( "Properties for " + mnames[0], mkeys, mvals)

mweights = []
for mname in mnames :
    mweights.append( "%8.3f" % mic.metal_composition(mname, 'atomic weight') )
mic.summary.add("Active Metals and Atomic Weight (amu)", mnames, mweights)
```

MIC MODULE PYTHON CALLS

TABLES

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

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

Create a New Graphical Report

```
mic.graph( title='User Graph', xlabel='X axis', ylabel='Y axis', yylabel='YY
axis', xlinear=True, ylinear=True, yylinear=True )

Keyword arguments:

   title    --- the graphical report title (default = 'User Graph')
   xlabel   --- x-axis label (default = 'X axis')
   ylabel   --- y-axis label (default = 'Y axis')
   yylabel  --- yy-axis label (default = 'YY axis')
   xlinear  --- x-axis linear scale; if false, use log scale
                (default = True)
   ylinear  --- y-axis linear scale; if false, use log scale
                (default = True)
   yylinear --- yy-axis linear scale; if false, use log scale
                (default = True)
```

Add a Curve

This script adds a curve to the last created graphical report:

```
mic.graph.add( name, x, y, yyaxis=False, color=None, linestyle='-', mark-
er='a', graphtype='both' )

Keyword arguments:

   name      --- the curve name
   x         --- list of x values; must be a list of floats
                 (or convertible) and the same length as y
   y         --- list of y values; must be a list of floats
                 (or convertible) and the same length as x
   yyaxis    --- place this curve on the yy-axis if True
                 otherwise place on the  y-axis (default = False)
   color     --- RGB color as an HTML hex string (e.g., '#4169e1')
```

```
                 or a three-element list or tuple (e.g., [65,105,225]);
                 if None, color is automatically selected (default = None)
   linestyle --- line style; (default = '-')
                    '-'        : solid
                    '--'       : dash
                    '.'        : dot
                    '-.'       : dash dot
                    '-..'      : dash dot dot
marker    --- marker shape; (default = 'a')
                 '+'        : plus
                 'o' or '0' : circle
                 'x'        : cross
                 '^'        : up triangle
                 'v'        : down triangle
                 's'        : square
                 'd'        : diamond
                 '8'        : hourglass
                 '~'        : horizontal hourglass
                 '' or None : no marker
                 'a'        : automatically selected
graphtype --- graph type; (default = 'both')
                 'curve'  or 'c' : curve
                 'points' or 'p' : points
                 'both'   or 'b' : curve-and-points
                 'hist'   or 'h' : histogram
```

Add a Curve Using the Second Y-Axis

This script adds a curve to the last created graphical report using the second y-axis:

```
mic.graph.addyy( name, xx, yy )

    Add a curve to the last created graphical report using the second
    y-axis.  The arguments to this call are the same as to mic.graph.add
    with the argument
```

GET PRIMARY ISOTHERM DATA

Physisorption

```
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
```

Chemisorption

```
mic.chem_isotherm( branch='primary' ) :
Get primary, repeat and difference isotherm data.
```

```
Keyword arguments:
  branch --- Specifies which analysis to get isotherm data;
             use 'primary' for the first analysis,
                 'repeat' for the repeat analysis
             and 'difference' for the difference of these two

Usage:

   p, q = mic.chem_isotherm('primary')
   p, q = mic.chem_isotherm('repeat')
   p, q = mic.chem_isotherm('difference')
   p    --- array of absolute pressures
   q    --- array of cumulative quantity adsorbed
```

GET OVERLAY ISOTHERM DATA

Physisorption

```
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
```

Chemisorption

```
mic chem_overlay( overlay_number = 1, branch='primary' ) :

Get overlay isotherm data.

Keyword arguments:

  overlay_number --- the overlay number (1 through 8; default = 1)

   branch --- Specifies which analysis to get isotherm data;
              use 'primary' for the first analysis,
                  'repeat' for the repeat analysis
              and 'difference' for the difference of these two

Usage:

  p, q = mic.chem_overlay(1, 'primary')
  p, q = mic.chem_overlay(1, 'repeat')
  p, q = mic.chem_overlay(1, 'difference')
  p    --- array of absolute pressures
  q    --- array of cumulative quantity adsorbed
```

GET 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

Physisorption

This script gets report results for the indicted report and sample.

```
mic.report( report_name, result, sample_number = 0 )


Keyword arguments:

  sample_number --- Identifier for the sample data to retrieve
                    0           : the current sample file
                    1 through 8 : the corresponding overlay sample file

Usage:

  sa                      = mic.report( 'bet' , 'surface area' )
  porewidth, incvol, desc = mic.report( 'bjhads' ,
                                        'incremental distribution'  )

The available report keywords, result keywords and a corresponding
description of the result is listed in the table below:

Report keyword  Result keyword              Description
--------------  --------------              -----------
    bet         surface area               Surface area ( m^2/g )
    bet         bet constant               BET constant ( dimensionless )
```

```
   bet          monolayer capacity         Monolayer capacity ( cm^3/g )
   tplot        external surface area      External surface area (m^2/g)
   tplot        micropore volume           Micropore volume (cm^3/g)
   bjhads       incremental distribution   Incremental Distribution
   bjhdes       incremental distribution   Incremental Distribution
   dhads        incremental distribution   Incremental Distribution
   hk           incremental distribution   Incremental Distribution
   dft          incremental distribution   Incremental Distribution
   nldft        incremental distribution   Incremental Distribution

where the incremental pore distribution result above (for those
reports which return this) is a list with three components being,

porewidth --- array of pore dimension boundaries (angstroms);
              empty-array if unavailable.
incvol    --- array of incremental pore volumes (cm^3/g);
              empty-array if unavailable.
desc      --- Name of data set; empty-string if unavailable.
```

GET IMPORTED PORE DATA

```
mic.imported_pore_data( import_number = 1 )

Keyword arguments:

  import_number --- the import number (1 through 8)

Usage:

  xdat, ydat, desc = mic.imported_pore_data(1)


  xdat --- array of pore dimension boundaries (angstroms);
           empty-array if unavailable.
  ydat --- array of incremental pore volumes (cm^3/g);
           empty-array if unavailable.
  desc --- Name of data set; empty-string if unavailable.
```

GET METAL COMPOSITION

Chemisorption

```
mic metal_composition( metal='' , metal_property='' , sample_number = 0 ) :

Get information about the active metals in this sample
```

```
Keyword arguments:

  metal           --- the metal to return information about
                      if '' or None, then return a list of the
                      active metals

  metal_property  --- the specific property to return information on
                      if '' or None, then return all the properties
                      for the specified metal (requires metal to be
                      specified)

  sample_number --- Identifier for the metal data to retrieve
                    0           : current sample file (default)
                    1 through 8 : corresponding overlay sample file

Usage:

  metal_list  = mic.metal_composition()
  copper_prop = mic.metal_composition( 'copper')
  copper_perc = mic.metal_composition( 'copper',
                                        'percent of sample mass' )

In the above first usage case, the list of active metals is returned.
In the above second usage case, a python dictionary type
is returned which includes all the properties of the metal
available and their corresponding values.  The last case returns
a single value (int, float, or string) for the specified property.

The metal_properity keywords which one can use are

  atomic weight
  oxygen atoms
  density
  percent of sample mass
  metal atoms
  cross sectional area
  percent reduced
  stoichiometry H2
  stoichiometry O2
  stoichiometry He

Or as just mentioned, one can make the call
metal_composition( metalname ) without any metal_property
keyword provided to see the whole dictionary.
```

12 CALCULATIONS

ALPHA-S METHOD

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

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

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

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

a. Slope (s cm^3/g STP)
b. Y-intercept (Q_0 cm^3/g STP)
c. Uncertainty of the slope (u(s) cm^3/g STP)
d. Uncertainty of the Y-intercept (u(Q_0) cm^3/g STP)
e. Correlation coefficient

Surface area is calculated as:

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

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

Pore size is calculated as:

$$V_p = Q_0 / D$$

BET SURFACE AREA

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

BJH Pore Volume and Area Distribution

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

The data used in these calculations must be in order of strictly decreasing numerical value. Points which do not meet this criterion are omitted. The remaining data set is composed of relative pressure (P), quantity adsorbed (Q) pairs from (P_1, Q_1) to (P, Q_n) where ($P_n = 0$, $Q_n = 0$) is assumed as a final point. Each data pair represents an interval boundary (or desorption step boundary) for intervals i=1 to i=n-1 where n = total number of (P, Q) pairs.

Generally, the desorption branch of an isotherm is used to relate the amount of adsorbate lost in a desorption step to the average size of pores emptied in the step. A pore loses its condensed liquid adsorbate, known as the core of the pore, at a particular relative pressure related to the core radius by the Kelvin[1] equation. After the core has evaporated, a layer of adsorbate remains on the wall of the pore. The thickness of this layer is calculated for a particular relative pressure from the thickness equation. This layer becomes thinner with successive decreases in pressure, so that the measured quantity of gas desorbed in a step is composed of a quantity equivalent to the liquid cores evaporated in that step plus the quantity desorbed from the pore walls of pores whose cores have been evaporated in that and previous steps. Barrett, Joyner, and Halenda[2] developed the method (known as the BJH method) which incorporates these ideas. The algorithm used is an implementation of the BJH method.

Explanation of Terms

A pore filled with condensed liquid nitrogen has three zones:

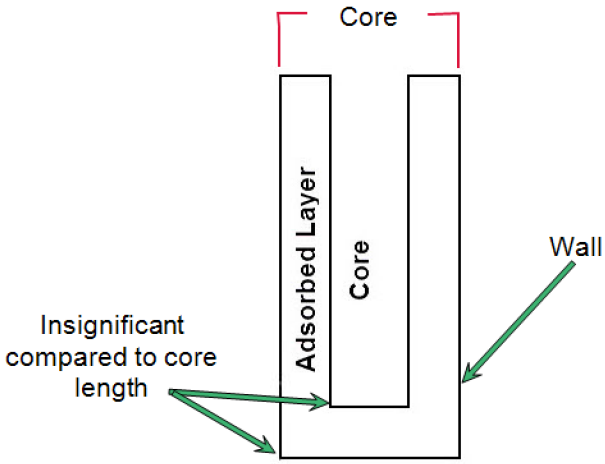
a. The *core* - evaporates all at once when the critical pressure for that radius is reached; the relationship between the core radius and the critical pressure is defined by the Kelvin equation.

b. The *adsorbed layer* - composed of adsorbed gas that is stripped off a bit at a time with each pressure step; the relationship between the thickness of the layer and the relative pressure is defined by the thickness equation.

c. The *walls of the cylindrical pore* - the diameter of the empty pore is required to determine the pore volume and pore area. End area is neglected.

---

1) Kelvin, J. (published under the name of Sir William Thomson), Phil. Mag. 42, 448-452 (1871).
2) Barrett, E.P.; Joyner, L.S.; and Halenda, P.., J. Am. Chem. Soc. 73, 373-380 (1951).



CALCULATIONS

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

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

where V_{mol} is the liquid molar volume from the fluid property information.

The relative pressure (P_i) is assumed to be close to unity so that substantially all the pores in the sample are filled.

The corresponding Kelvin core radius is calculated. Only pores smaller than this size will be included:

$$Rc_i = \frac{-A}{(1+F)\ \ln(P_i)}$$

where

A	=	adsorbate property factor (from the *BJH Adsorptive Options* window)
F	=	fraction of pores open at both ends (from the *BJH Adsorption Report Options* window or the *BJH Desorption Report Options* window); assumed to be zero for desorption
Rc	=	Kelvin radius (Å) of core

This radius will be adjusted for the thickness of the adsorbed layer during subsequent calculation steps.

The following calculations (a-c) are made for each relative pressure interval based on the increment of volume desorbed during that interval:

where

i	=	interval number, that is i=1 for the first interval from P_1 to P_2, and so on
j	=	each previous interval during which new pores were found
k	=	the total number of intervals in which new pores have been found. It is also the number of lines reported on the BJH table for collected data

a. The thickness of the adsorbed layer at the end of the interval is calculated using the equation located in *"Thickness Curve" on page 12 - 48*.

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 12 - 48*.

For the first pressure interval, there are no previously opened pores so the volume of liquid desorbed from walls of previously opened pores is zero ($Vd_1 = 0$), and the remainder of Step (a) is skipped.

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

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

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

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

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

$$Vd_i = \sum_j \left(LP_j\right)\left(CSAa_j\right)$$ for all previously opened pores

where LP_j = length of previously opened pores as calculated in Step b(2).

b. The physical processes occurring for this pressure interval are determined as:

1. If Vd_i is greater than the current increment of volume desorbed (Vl_i - Vl_{i+1}), desorption from walls only is occurring. Total surface of walls exposed thus far (cm^2/g) is calculated as:

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

where

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

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

$$\Delta Tw = \frac{\left(Vl_i - Vl_{i+1}\right)\left(10^8 \frac{\text{Å}}{cm}\right)}{SAw_i}$$

Since no cores are evaporated in this pressure interval, no new pores are revealed. Thus no ending Kelvin radius and average pore diameter are calculated for this interval. Note that this means the report may have fewer tabulated intervals on the collected data report than experimental pressure intervals.

2. If Vd_i is less than the volume increment desorbed during this interval (Vl_i-Vl_{i+1}), the remaining volume is due to new pores with core evaporation taking place in this interval. *K*, the number of intervals with new pores exposed, is increased by 1. (For the interval from the lowest Pr_1 to zero relative pressure, no new pore volume is calculated and the rest of Step b is skipped.)

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

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

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

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

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

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

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

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

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

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

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

The decrease in thickness of the wall layer by desorption from the walls of new pores during the lower portion of the pressure interval is calculated as:

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

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

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

The length of the newly opened pores is calculated as:

$$LP_k = \frac{Vc_i}{CSAc_k}$$

Pore diameters and radii are adjusted for the change in thickness of the adsorbed wall layer during this interval. If new pores were opened during this interval, the average diameter is adjusted by the change in layer thickness during the second portion of the desorption interval as:

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

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

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

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

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

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

for all except Rc_{k+1}.

Steps a to c are repeated for each pressure interval.

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

$$Dp_j = 2\left(Rc_j\right)$$

for all Rc_j including Rc_{k+1}.

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

(1) Incremental Pore Volume (Vp_i, cm^3/g):

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

(2) Cumulative Pore Volume ($Vp_{cum,i}$, cm^3/g):

$$VP_{cum,i} = \sum_j Vp_j for\left(J \le 1\right)$$

(3) Incremental Surface Area (SA_i, m^2/g):

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

(4) Cumulative Surface Area ($SA_{cum,i}$, m^2/g):

$$SA_{cum,10} = \sum SA_j forJ \le 1$$

(5) *dV/dD* pore volume (dV/dD_i, cm^3/g-A):

$$\frac{dV}{dD_i} = \frac{VP_i}{Dp_i - Dp_{i+1}}$$

(6) *dV/dlog(D)* pore volume ($dV/dlog(D)_i$, cm^3g):

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

(7) *dA/dD* pore area (dA/dD_i, m^2/g-A):

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

(8) *dA/dlog(D)* pore area [$dA/dlog(D)_i$, m^2/g]:

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

For fixed pore size tables (if selected), the following calculations are performed:

(1) Average Fixed Pore Size ($DF_{avg,j}$, *A*):

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

calculated for all intervals in the fixed pore size table.

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

(2) Cumulative Pore volume ($VpF_{cum,i}$, cm^3/g):

$$VpF_{cum,i} = INTERP\left(DpF_{i+1}\right)$$

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

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

(3) Incremental Pore Volume (VpF_i, cm^3/g):

$$VpF_i = VpF_{cum,i} - VpF_{cum,i-1}$$

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

(4) Cumulative Surface Area ($SAF_{cum,i}$, m^2/g):

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

where *INTERP(x)* is the value interpolated from the function $X = Dp_{j+i}$ and $Y = SA_{cum,j}$.

(5) Incremental Surface Area (SAF_i, m^2/g):

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

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

(6) *dV/dD* pore volume ($dV/dDpF_i$, cm^3/g-A):

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

where *INTERP(x)* is the value interpolated from the function $X = D_{avg'j}$ and $Y = dV/dD_j$.

(7) *dV/dlog(D)* pore volume [$dV/dlog(DpF_i)$, cm^3/g]:

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

where *INTERP(x)* is the value interpolated from the function $X = D_{avg,j}$ and $Y = dV/dlog(D)_j$.

(8) *dA/dD* pore area ($dA/dDpF_i$, m^2/g-A):

$$\frac{dA}{dDpF1} = INTERP\left(DpF_{i+1}\right)$$

where *INTERP(x)* is the value interpolated from the function $X = D_{avg'j}$ and $Y = dA/dD_j$.

(9) *dA/dlog(D)* pore area [$dA/dlog(DpF_i)$, m^2/g]:

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

where *INTERP(x)* is the value interpolated from the function $X = D_{avg,j}$ and $Y = dA/dlog(D)_j$.

COMPENDIUM OF VARIABLES

ΔTd	=	thickness of layer desorbed from walls of newly opened pores (Å)
ΔTw	=	thickness of adsorbed layer desorbed during interval (Å)
A	=	adsorbate property factor; from the *BJH Adsorptive Options* window
CSA	=	analysis gas molecular cross-sectional area (nm^2), user-entered on the *Adsorptive Properties* window
$CSAa$	=	annular cross-sectional area of the desorbed layer (cm^2)
$CSAc$	=	cross-sectional area of opening of newly opened pores (cm^2)
D_{avg}	=	average pore diameter (Å)
Dp	=	pore (or core) diameter (Å)
F	=	fraction of pores open at both ends; from the *BJH Adsorption Report Options* window or the *BJH Desorption Report Options* window
LP	=	length of pore (cm/g)
P	=	relative pressure
Q	=	quantity adsorbed expressed as a volume (cm^3/g STP)
Rc	=	Kelvin radius (Å) of core
SAw	=	total surface area of walls exposed (cm^2/g)
Tw	=	thickness of remaining adsorbed wall (Å)
Vc	=	volume desorbed from cores of newly opened pores (cm^3/g)
Vd	=	volume of gas desorbed from walls of previously opened pores (cm^3/g)
Vl	=	liquid equivalent volume of volume adsorbed (cm^3/g)
V_{mol}	=	liquid molar volume, from the fluid property information

CRYSTALLITE SIZE

Chemisorption

$$d_{xtal} = \frac{1000k}{\bar{\rho} A_{metal}}$$

Where

k	=	shape factor; 6 for sphere, 5 for cube
$\bar{\rho}$	=	weighted average density of the active metals

DFT (DENSITY FUNCTIONAL THEORY)

The adsorption isotherm is known to convey a great deal of information about the energetic heterogeneity and geometric topology of the sample under study. The data of physical adsorption have been used for many years as the basis for methods to characterize the surface area and porosity of adsorbents. Real solid surfaces rarely approach ideal uniformity of structure. It is accepted that in general, the surface of even a nonporous material presents areas of greater or lesser attraction for adsorbed molecules.

This energetic heterogeneity greatly affects the shape of the adsorption isotherm with the result that simple theories such as the Langmuir and BET formulas can, at best, give only approximate estimates of surface area. Porous solids virtually are never characterized by a single pore dimension, but instead exhibit a more or less wide distribution of sizes. The observed adsorption isotherm for a typical material is therefore the convolution of an adsorption process with the distribution of one or more properties which affect that process. This was first stated mathematically by Ross and Olivier[1)] for the case of surface energy distribution and has become known as the integral equation of adsorption.

The Integral Equation of Adsorption

In a general form for a single component adsorptive, the integral equation of adsorption can be written as

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

where

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

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

Equation (1), a Fredholm integral of the first kind, is a member of a class of problems known as ill-posed, in that there are an infinite number of functional combinations inside the integral that will provide solutions. Even when the kernel function is known, experimental error in the data can make solving for even a single distribution function a difficult task. Solving for multiple distribution functions requires more data than provided by a single adsorption isotherm.

Application to Surface Energy Distribution

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

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

where

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

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

where

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

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

Performing the Deconvolution

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

$$Q\left(p\right) = \sum_i q\left(p, Z_i\right) f\left(Z_i\right) \tag{4}$$

Given a set of model isotherms, $q(p,Z)$, from a model chosen from the models library and an experimental isotherm, $Q(p)$, contained in a sample information file, the software determines the set of positive values $f(Z)$ that most nearly, in a least squares sense, solves equation (4). The distributed property, surface energy or pore size, is then displayed on the *Report Options* window as a selection of tables or graphs.

Regularization

DFT allows a selectable regularization (also referred to as smoothing) constraint to be applied during the deconvolution process to avoid over-fitting in the case of noisy data or ill-fitting models. The method used is based on co-minimization of the second derivative of the distribution. The relative weight given to this term is determined by the value of the regularization parameter, which is set on the *DFT Pore Size* or *Surface Energy* window and also is shown in the header of reports. The value of the regularization parameter varies from zero (for no second derivative constraint) to ten (indicating a weight equal to minimizing the residuals), or even larger. When the distribution and residuals obtained change little with the value of the regularization parameter, it indicates that the chosen model provides a good representation of the data. Conversely, a large sensitivity to the regularization parameter might indicate inadequate data or a poor choice of model to represent the data.

DOLLIMORE-HEAL ADSORPTION

The calculations for the Dollimore-Heal reports are the same as those for BJH, except for the calculation of average pore diameter and pore length.

PORE DIAMETER

Pore diameter is determined from the Kelvin radius and thickness equation:

$$D_i = 2r_k(P_i) + t(P_i)$$

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

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

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

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

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

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

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

where

ΔV_p	=	Change in pore volume
$A_{p,i}$	=	Pore surface area
$A_{p,i,cum}$, $l_{i,cum}$	=	Summations over the lengths and areas calculated so far
Cv	=	Volume correction factor
D	=	Density conversion factor
$\overline{r_k}$	=	Average Kelvin radius
$\bar{t}$	=	Average thickness

DUBININ-ASTAKHOV

The Dubinin-Astakhov equation is:

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

where

β	=	the affinity coefficient of the analysis gas relative to the Po gas, from the *Dubinin Adsorptive Options* window
E_0	=	characteristic energy *(kj/mol)*
N	=	Astakhov exponent, may be optimized or user entered from the *Dubinin Report Options* window
P	=	equilibrium pressure
Po	=	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:

$$LV = \log(Q)$$

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

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

a. Slope (*S* cm^3/g STP)
b. Y-intercept (*YI* cm^3/g STP)
c. Error of the slope (S_{err} cm^3/g STP)
d. Error of the y-intercept (YI_{err} cm^3/g STP)
e. Correlation coefficient
f. Optimized Astakhov exponent (*N*)

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

Monolayer Capacity (cm^3/g STP):

$$Q_0 = 10^{YI}$$

Micropore Volume (cm^3/g):

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

where

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

Limiting Micropore Volume (cm^3/g):

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

where

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

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

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

Characteristic Energy (KJ/mol):

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

Modal Equivalent Pore Diameter (Å):

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

where

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

Maximum Differential Pore Volume (cm^3/g-Å):

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

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

Mean Equivalent Pore Width (Å):

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

Micropore surface area (m^2/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 $0 \leq \times \leq 1$:

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

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

where

b_1	=	-0.57719 1652
b_2	=	0.98820 5891
b_3	=	-0.89705 6937
b_4	=	0.91820 6857
b_5	=	-0.75670 4078
b_6	=	0.48219 9394
b_7	=	-0.19352 7818
b_8	=	0.03586 8343

and where

$$x + 1 = \left(\frac{3N+1}{3N}\right)$$

Equivalent Pore Diameter (Å):

$$D_i = 2\left[\frac{-\left(\frac{10^3 nm^3 / \text{Å}^3}{\beta \cdot E_0}\right)^N}{\ln(W_i) - \ln(W_0)}\right]^{1/3N}$$

dV/dD Pore Volume (cm^3/g-Å):

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

DUBININ-RADUSHKEVICH

The Dubinin-Radushkevich[1] equation is:

$$\log\left(Q\right) = \log\left(Q_0\right) - \frac{BT^2}{\beta} \times \left[\log\frac{Po}{P}\right]^2$$

where:

β	=	the affinity coefficient of analysis gas relative to Po gas (for this application β is taken to be 1)
B	=	a constant
Po	=	saturation vapor pressure of gas at temperature T
P	=	equilibrium pressure
Q	=	quantity adsorbed at equilibrium pressure (cm^3/g STP)
Q_0	=	the micropore capacity (cm^3/g STP)
T	=	analysis bath temperature (*K*), from the *Po and Temperature Options* window

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

$$LV = \log(Q)$$

$$LP = \log\left(\frac{Po}{P}\right)^2$$

The intercept, *log(Vo)* can be found by performing a least-squares fit on the (*LP,LV*) designated pairs where *LP* is the independent variable and *LV* is the dependent variable. Assuming the adsorption of gas is restricted to a monolayer, *Vo* is the monolayer capacity. Based on this assumption, the following are calculated:

a. Slope (*S* cm^3/g STP)
b. Y-intercept (*YI* cm^3/g STP)

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

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

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

e. Correlation coefficient

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

Monolayer Capacity (cm^3/g STP):

$$Q_0 = 10^{YI}$$

Error of Monolayer Capacity (cm^3/g STP):

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

Micropore surface area (m^2/g):

$$SDP = \frac{\sigma\, Q_0\, N_A}{22414 cm^3\left(\frac{10^{18}\ nm^2}{m^2}\right)}$$

where

σ = molecular cross sectional area of gas (nm^2) from the *Adsorptive Properties* window

EQUATION OF STATE

EQUILIBRATION

Equilibration is reached when the pressure change per equilibration time interval (first derivative) is less than 0.01% of the average pressure during the interval. Both the first derivative and average pressure are calculated using the Savitzky-Golay[1)] convolution method for polynomial functions. The following equations are those used to compute weighted average and first derivative, respectively, for the 6th point of an 11-point window.

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

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

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

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

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 Po, equilibration will not take place until the *Minimum equilibration delay for P/Po 0.995* has expired, in addition to the standard equilibration criteria.

F-RATIO METHOD

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

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

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

FREE SPACE

Chemisorption

The free space is the physical volume below the sample valve. The different temperatures in the sample tube, stem, and port must be accounted for.

Free space volumes are calculated as:

$$n_p = \frac{P_s V_p}{z\left(P_s, T_p\right) T_p}$$

$$n_s = n_d - n_p$$

$$V_s = \frac{n_s z(P_s, T_s) T_s}{P_s}$$

The reported free space is

$$V_f = V_p + V_s$$

The quantity of gas in the free space for a given data point is:

$$n_p = P_s \left(\frac{V_p}{z\left(P_s, T_p\right) T_p} + \frac{V_s}{z(P_s, T_s,) T_s} \right)$$

where:

n_d	=	quantity of gas dosed
n_p	=	quantity of gas in the port
n_s	=	quantity of gas in the sample tube
P_s	=	sample (and port) pressure
T_p	=	port temperature
T_s	=	sample temperature
V_p	=	volume of the sample port
V_s	=	volume of the sample tube
$z(P,T)$	=	gas compressibility factor P and temperature T for the gas used

FREUNDLICH ISOTHERM

The Freundlich isotherm has the form

$$\frac{Q}{Q_m} = CP^{\frac{1}{m}}$$

where

C	=	temperature-dependent constant
m	=	temperature-dependent constant
P	=	equilibrated collected pressure measured by gauge at temp T_{amb}
Q	=	quantity of gas adsorbed
Q_m	=	quantity of gas in a monolayer

The pressure is absolute; typically, m > 1. In terms of quantity adsorbed,

$$Q = Q_m CP^{\frac{1}{m}}$$

Taking the log of both sides yields

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

HORVATH-KAWAZOE

A relative pressure lower limit is determined such that L-d_0 never equals zero. All pressure points less than this limit are discarded. For each collected relative pressure point, values of L are chosen in an iterative manner, and the relative pressure (P/Po) determined by solving one of the following equations:

- Slit Pore Geometry (original Horvath-Kawazoe)
- Cylinder Pore Geometry (Saito/Foley)
- Sphere Pore Geometry (Cheng/Yang)

Slit Pore Geometry (original Horvath-Kawazoe)

When using the original Horvath-Kawazoe[1)] method, the following equation is solved for each value of P. The value of L is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$ln\frac{P}{Po} = \frac{K}{RT}\frac{IP\times 10^{32}}{\sigma^4 L - 2d_0}\frac{\sigma^4}{3L - d_0{}^3} - \frac{\sigma^{10}}{9L - d_0{}^9} - \frac{\sigma^4}{3d_o{}^3} + \frac{\sigma^{10}}{9d_0{}^9}$$

where

1) Horvath, G. and Kawazoe, K., J. Chem. Eng. Japan 16(6), 470 (1983).

10^{32} = the number of cm^4 that are equal to $Å^4$

α = gas solid nuclear separation at zero interaction energy (Å), $\frac{Z_S + Z_A}{2}$

d_0 = $\frac{D_A + D_S}{2}$

where:

D_A = molecular diameter (Å) from the *Horvath-Kawazoe Physical Properties* window

D_S = diameter of sample atom (Å) from the *Horvath-Kawazoe Physical Properties* window

IP = interaction parameter (erg-cm^4) from the *Horvath-Kawazoe Report Options* window

K = Avogadro Constant (N_A)

L = pore width (nucleus to nucleus) (Å)

P = equilibrium pressure

Po = saturation pressure

R = gas constant (8.31441×10^7 erg/mol *K*)

T = analysis bath temperature (*K*), from an entered or calculated value on the *Po and Temperature Options* window

where:

Z_s = sample equilibrium diameter at zero interaction energy (Å) from the *Horvath-Kawazoe Physical Properties* window

Z_A = zero interaction energy diameter from the *Horvath-Kawazoe Physical Properties* window

Cylinder Pore Geometry (Saito/Foley)

When using the Saito-Foley[1)] method, the following equation is solved for each value of *P*. The value of *L* is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

1) Saito, A. and Foley, H. C., AlChE Journal 37(3), 429 (1991).

$$\ln\left(\frac{P}{Po}\right) = \frac{3}{4}\frac{\pi K}{RT} \times \frac{IPx10^{32}}{d_0{}^4} \times \sum_{k=0}^{\infty}\left[\frac{1}{k+1}\left(1-\frac{d_0}{r_p}\right)^{2k} \times \left\{\frac{21}{32}\alpha_k\left(\frac{d_0}{r_p}\right)^{10} - \beta_k\left(\frac{d_0}{r_p}\right)^{4}\right\}\right]$$

where

Sphere Pore Geometry (Cheng/Yang)

When using the Cheng/Yang[1)] method, the following equation is solved for each value of *P*. The value of *L* is determined when the solved-for relative pressure is within 0.1% of the collected absolute pressure.

$$\ln\left(\frac{P}{P0}\right) = \frac{6N_1\varepsilon^*{}_{12} + N_2\varepsilon^*{}_{22}\,L^3 \times 10^{32}}{RTL - d_0{}^3}\left[-\left(\frac{d_0}{L}\right)^6\left(\frac{T_1}{12}+\frac{T_2}{8}\right) + \left(\frac{d_0}{L}\right)^{12}\left(\frac{T_3}{90}+\frac{T_4}{80}\right)\right]$$

where

1) Cheng, Linda S. and Yang, Ralph T., Chemical Engineering Science 49(16), 2599-2609 (1994).

10^{32}	=	the number of cm^4 that are equal to $Å^4$
ε^*_{12}	=	$\frac{Å_S}{4d_s^6}, where Å_s = \frac{6(mc^2)a_S a_A}{\frac{a_S}{X_S}+\frac{a_A}{X_A}}$
ε^*_{22}	=	$\frac{A_A}{4D_A^6}, where Å_A = \frac{3(mc^2)(a_A)(X_A)}{2}$
d_0	=	$\frac{D_A + D_S}{2}$

where

D_A	=	molecular diameter (Å) from the *Horvath-Kawazoe Physical Properties* window
D_S	=	diameter of sample atom (Å) from the *Horvath-Kawazoe Physical Properties* window

L	=	pore width (nucleus to nucleus) (Å)
N_1	=	$4\pi L^2 N_S$, where N_S = number of sample atoms/cm^2 at monolayer
N_2	=	$4\pi (L - d_0)^2 N_A$, where N_S = number of gas molecules/cm^2
P	=	equilibrium pressure
Po	=	saturation pressure
R	=	gas constant (8.31441×10^7 erg/mol K)
T	=	analysis bath temperature (*K*), from an entered or calculated value on the *Po and Temperature Options* window

$$T_1 = \frac{1}{(1-S)^3} - \frac{1}{(1+S)^3}$$

$$T_2 = \frac{1}{(1+S)^2} - \frac{1}{(1-S)^2}$$

$$T_3 = \frac{1}{(1-S)^9} - \frac{1}{(1+S)^9}$$

$$T_4 = \frac{1}{(1+S)^8} - \frac{1}{(1-S)^8}$$

where $S = \frac{L - d_0}{L}$

Cheng/Yang Correction

This factor corrects for the nonlinearity of the isotherm. It adds an additional term to the equations for the different geometrics:

$$\ln\left(\frac{P}{Po}\right) = G\left(L\right) - \left[1 - \frac{1}{\theta}\ \ln\left(\frac{1}{1-\theta}\right)\right]$$

where

$G(L)$	=	one of the Horvath-Kawazoe equations given above
θ	=	degree of void filling; θ is estimated by first computing the monolayer capacity (Q_m) with the Langmuir equation over the range of data points from relative pressure 0.02 to 0.2 or the maximum relative pressure included in the Horvath-Kawazoe analysis. θ is computed as the quantity adsorbed over Q_m.

Interaction Parameter

The interaction parameter (IP) results from the following calculations:

The Kirkwood-Muller dispersion coefficients

$$A_S = \frac{6mc^2\alpha_S\alpha_A}{\frac{\alpha_S}{X_S} + \frac{\alpha_A}{X_A}}$$

$$A_A = \frac{3mc^2 a_A x_A}{2}$$

where

α_A	=	polarizability of gas molecule (cm^3)
α_S	=	polarizability of sample atoms (cm^3)
mc^2	=	kinetic energy of electron (0.8183×10^{-6} erg)
X_A	=	diamagnetic susceptibility of gas molecule (cm^3)

$$IP = (N_A A_A) + (N_S A_S)$$

N_A	=	number of gas molecules/cm^2 at monolayer from the *Horvath-Kawazoe Physical Properties* window
N_S	=	number of sample atoms/cm^2 from the *Horvath-Kawazoe Physical Properties* window

X_s	=	diamagnetic susceptibility of sample atom (cm^3)

See ***"Interaction Parameter Components Table" on the next page*** for recommended values.

Additional Calculations

Based on the previous calculations, the following can be calculated:

Adjusted Pore Width (Å):

(Shell to Shell)

$$AL_i = L_i - D_S$$

Cumulative Pore Volume (cm^3/g):

$$V_{cum,i} = \frac{Q_i V_{mol}}{22414 cm^3 STP}$$

where

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

dV/dD Pore Volume (cm^3/g-Å):

$$\frac{dV}{dD_i} = \frac{V_{cum,i} - V_{cum,i-1}}{AL_i - AL_{i-1}}$$

Median Pore Width (Å):

$$V_{half} = \frac{V_{cum,n}}{2}$$

$$D_{med} = 10\left[\log\left(D_1\right) + \left[\log\left(V_{half}\right) - \log\left(V_1\right)\right] \times \frac{\log\left(D_g\right) - \log\left(D_1\right)}{\log\left(V_g\right) - \log\left(V_1\right)}\right]$$

where

D_l	=	pore width (L_i) that corresponds to V_1
D_g	=	pore width (L_i) that corresponds to V_g
$V_{cum,n}$	=	total cumulative pore volume ($V_{cum,i}$) for points designated for Horvath-Kawazoe calculations
V_g	=	cumulative pore volume ($V_{cum,i)}$ for first point greater than V_{half}
V_{half}	=	50% of total cumulative pore volume
V_l	=	cumulative pore volume ($V_{cum,i}$) for first point less than V_{half}

Interaction Parameter Components Table

Gas	Bath Temperature (K)	Sample Type	Interaction Parameter Calculated Value *
Argon	87.3	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.61 5.89 3.19
Carbon Dioxide	298.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.20 9.20 5.08
	273.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.34 9.35 5.22
	194.65	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	4.72 9.72 5.60
Nitrogen	77.15	Carbon (Ross/Olivier value) Carbon (Horvath/Kawazoe value) Zeolite	2.84 6.53 3.49
* The interaction parameter is entered in the *Horvath-Kawazoe Report Options* window in the following field: Interaction parameter: (calculated value) × 10^{-43} erg-cm^4			

The following values were used to calculate the values in the previous table.

Carbon-Graphite

D_S = 3.40
N_S = 3.845×10^{15}
X_S = 1.05×10^{-29} (Ross/Olivier)
13.5×10^{-29} (Horvath/Kawazoe, implicit)
α_s = 1.02×10^{-24}

Zeolite

D_S = 3.04
N_S = 3.75×10^{15}
X_S = 1.94×10^{-29}
α_s = 0.85×10^{-24}

Nitrogen

D_A = 3.00
N_A = 6.71×10^{14}
X_A = 3.6×10^{-29}
α_A = 1.76×10^{-24}

Argon

D_A = 2.95
N_A = 7.608×10^{14}
X_A = 3.22×10^{-29}
α_A = 1.63×10^{-24}

Carbon Dioxide

D_A = 3.23
N_A = 4.567×10^{14} (25 ºC)
5.45×10^{14} (0 ºC)
7.697×10^{14} (-78 ºC)
X_A = 5.0×10^{-29}
α_A = 2.7×10^{-24}

D_A values are from van der Waal's constant.

N_A values are from liquid densities.

x and *a* values are derived from data found in Ross and Olivier[1] .

The physical parameters referenced in Saito/Foley are:

Aluminophosphate

D_S = 2.60
N_S = 1.48×10^{15}
X_S = 1.3×10^{-29}
α_s = 2.5×10^{-24}

Aluminosilicate

D_S = 2.76
N_S = 1.31×10^{15}
X_S = 1.3×10^{-29}
α_s = 2.5×10^{-24}

[1] Ross and Olivier, J.P., “On Physical Adsorption,” J. Wiley and Sons, New York (1964)

LANGMUIR SURFACE AREA

Physisorption

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

$$L = \frac{P_{rel}}{N_{ads}}$$

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

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

a. Slope (S g/cm^3 STP)
b. Y-intercept (Y_{int} g/cm^3 STP)
c. Error of the slope (S_{err} g/cm^3 STP)
d. Error of the y-intercept (YI_{err} g/cm^3 STP)
e. Correlation coefficient

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

Langmuir Surface Area (m^2/g):

$$SA_{Lan} = \frac{CSA \times N_A}{\left(22414 cm^3 \ STP\right)\left(\frac{10^{18} \ nm^2}{m^2}\right) S}$$

where

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

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

$$Q_m = \frac{1}{S}$$

[1] Langmuir, I., J. Am. Chem. Soc. 38, 2267 (1916); J. Am. Chem. Soc. 40, 1361 (1918); Phys. Rev 8, 149 (1916).

Langmuir b Value:

$$b = Y_{\text{int}} V_m$$

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

$$LAN_{err} = \frac{SA_{Lan} S_{err}}{S}$$

TRANSFORM

Chemisorption

The Langmuir isotherm is

$$\frac{Q}{Q_m} = \frac{bP}{1 + bP}$$

The isotherm is transformed so that *P/Q* is plotted as a function of pressure. The transformed data are fitted with a straight line. the slope (m) and intercept (y_0) of the fit line are used in the calculations below.

SURFACE AREA

$$A_{Lang} = \frac{\overline{A}_{atom} \overline{S} N_A}{V_{mol} m} \cdot 10^{-18} \frac{m^2}{nm^2}$$

MONOLAYER CAPACITY

$$Q_{m=\frac{1}{m}}$$

LANGMUIR b VALUE

$$b = \frac{1}{y_0 Q_m}$$

DISSOCIATIVE CHEMISORPTION

Chemisorption

The Langmuir isotherm may be derived for dissociative chemisorption.

$$\frac{Q}{Q_m} = \frac{b\sqrt{P}}{1 + b\sqrt{P}}$$

The calculations are performed with the slope and intercept of a fit of $\frac{\sqrt{P}}{Q}$ as a function of $\sqrt{P}$.

METAL DISPERSION

Chemisorption

$$D = 100\,\% \cdot 100\,\% \frac{Q_o \overline{S}}{V_{mol} \Sigma \frac{P_i}{W_i}}$$

Where

$V_{mol} \simeq 22414 cm^3 / mol$ the molar volume of an ideal gas at standard temperature and pressure

METALLIC SURFACE AREA

Chemisorption

The metallic surface area is the total active metal surface area available for interaction with the adsorbate.

$$A_{metal} = \frac{N_A Q_o \overline{SA}_{atom}}{V_{mol}}$$

Where

$N_A \simeq 6.023 \times 10^{23}$ the number of atoms per mole

MP-METHOD

With the (t_i, Q_i) data pairs[1)], the Akima semi-spline interpolation method is used to interpolate quantity adsorbed values based on thickness values that are evenly spaced 0.2 angstrom apart starting at the first outlier point. Outliers are defined as those points that have the maximum instantaneous slope within an iteratively shrinking subset of all points. The remaining pore surface area calculation result is the slope of the line defined by two consecutive interpolated points. The slopes of each pair of consecutive points from the origin to the last point must be monotonically decreasing and non-negative. With the interpolated points set the following can be calculated:

Average pore hydraulic radius (Å):

$$R_i = \frac{t_i + t_{i-1}}{2}$$

Remaining pore surface area for the ith point (m²/g):

$$S_i = \frac{Q_i - Q_{i-1}}{t_i - t_{i-1}} \frac{V_{mol}}{22414 \ cm^3 STP} \times 10^4$$

where

10^4	=	unit conversions
V_{mol}	=	liquid molar volume from the fluid property information

Incremental pore surface area occluded for the ith point (m²/g):

$$S_{inc,i} = S_{i-1} - S_i$$

Cumulative pore surface area occluded for the ith point (m²/g):

$$S_{cum_i} = S_{inc,i} + S_{inc,i-1} + \ldots + S_{inc,i}$$

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):

$$V_{inc,i} = S_{inc,i} R_i \times 10^{-4}$$

---

1) Mikhail, R., Brunauer, S. and Bodor, E., J. Colloid and Interface Sci. 24, 45 (1968).

Cumulative pore volume occluded for the ith point (cm^3/g):

$$V_{cum,\,i} = V_{inc,\,i-1} + V + \ldots + V_{inc,\,i}$$

dV/dR pore volume for the ith point (cm^3/g-Å):

$$\frac{dV}{dR_i} = \frac{V_{inc,\,i}}{t_i - t_{i-1}}$$

QUANTITY ADSORBED

Physisorption

For the ith dose, the quantity dosed is

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

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

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

The quantity of gas in the free space is

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

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

where:

T_{amb}	=	approximate room temperature (298 K)
T_{amb}	=	analysis bath temperature (K)
V_{fc}	=	volume of free space, cold (cm^3 at standard temperature)
V_{fw}	=	volume of free space, warm (cm^3 at standard temperature)

The specific quantity adsorbed is

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

where m is the sample mass.

Free Space - Measured

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

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

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

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

where:

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

Free Space - Calculated

The calculated free space is determined by subtracting the gas capacity of the volume occupied by the sample from the measured free space of the empty tube.

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

Chemisorption

A portion of the dosing volume may be at a slightly elevated temperature due to heating of the sample ports. The manifold volume is partitioned into a volume at the temperature of the manifold block and a volume at the average temperature of the ports.

$$n_a = n_d - n_f$$

$$n_d = P_{1m} C\left(P_{1m}, T_{1m}, \overline{T}_{1p}\right) - P_{2m} C\left(P_{2m}, T_{2m}, \overline{T}_{2p}\right)$$

$$C\left(P, T_m, T_p\right) = V_m \left(\frac{\alpha}{z(P, T_m) T_m} + \frac{\beta}{z\left(P, T_p\right) T_p} \right)$$

where:

α and β	=	constants that determine the relative weights of the manifold and port temperatures
n_a	=	quantity of gas adsorbed
n_d	=	quantity of gas dosed
n_f	=	quantity of gas in the free space
P_{1m}	=	manifold pressure before dosing onto the sample
P_{2m}	=	manifold pressure after dosing
T_{1m}	=	manifold temperature before dosing onto the sample
T_{1p}	=	average of all port temperatures before dosing onto the sample
T_{2m}	=	manifold temperature after dosing
T_{2m}	=	average of all port temperatures after dosing
V_m	=	volume of the dosing manifold

REAL GAS EQUATION OF STATE

Chemisorption

All chemisorption gas accounting calculations utilize the real gas equation of state and compressibility factor data traceable to NIST.

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

where

n	=	quantity of gas
P	=	pressure
T	=	temperature
V	=	volume
$z(P,T)$	=	compressibility factor for the gas of interest at the given pressure and temperature

Quantity of gas in cm^3 STP is given by

$$Q = n\frac{T_{STD}}{P_{STD}}$$

RELATIVE PRESSURE

SATURATION PRESSURE

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

1. Po is measured in the Po tube for each isotherm point.
2. The saturation pressure is measured in the sample tube after all adsorption data points have been collected. This pressure is used as Po for all data points.
3. Po is measured for all points as with #1. After all adsorption points have been taken Po is measured in the sample tube. The measured Po points are shifted so that the Po measured in the Po tube matches the Po measured in the sample tube. That is, $Po(\mathrm{i}) = Po(\mathrm{i}) + Po_s - Po_n$ where Po_n is the Po measured in the Po tube when Po in the sample tube (Po_s) was measured.
4. Determine Po from pressure measured over the dosing source. Note that the *Adsorptive Properties* must specify dosing from Psat tube, Sample port 3, or Vapor source.
5. The saturation pressure of a gas is measured in the Po tube for each data point. The bath temperature is found by looking up the temperature for the measured saturation pressure in the fluid properties. Po of the analysis gas is found from the bath temperature as in #6. If dosing is done from the Psat tube, Po is determined once at the beginning of the analysis and used for all data points. Otherwise, Po is measured for each data point.
6. Po is found by looking up the saturation pressure for the entered bath temperature in the fluid property information.

   Lookup of saturation pressure in the fluid properties is done by interpolating the Psat data using the Clausius-Clapeyron equation, $\ln\left(P\right) = \frac{a}{T} + b$. The constants *a* and *b* are determined from the pressures and temperatures that bound the bath temperature. Temperature lookup is done by solving for *T*, $T = \frac{a}{\ln(P) - b}$, where *a* and *b* are determined from the pressures that bound the given saturation pressure.

7. If entered, Po = user-entered value.

SPC REPORT VARIABLES

REGRESSION CHART VARIABLES

The line of best fit for the Regression Chart is calculated by the usual Least Squares method. (Refer to *BASIC Scientific Subroutines Vol II*, by F.R. Ruckdeschel, Copyright 1981 BYTE Publications/McGraw Hill, p. 16.) If there is only a single point or all *N* points have the same x-value, there can be no line of best fit in the standard form.

$$\overline{X} = \frac{\Sigma\, x_i}{N}$$

$$\overline{Y} = \frac{\Sigma\, y_i}{N}$$

$$\text{Slope} = \frac{\Sigma\left(x_i - \overline{X}\right)\left(y_I - \overline{Y}\right)}{\Sigma\left(x_i - \overline{X}\right)^2}$$

The coefficient of Correlation for this line is also calculated in the usual way. (Refer to *Mathematical Handbook for Scientists and Engineers*, by Granino A. Korn and Theresa M. Korn, Copyright 1961, 1968 McGraw Hill, Sec. 18.4.)

$$\sigma_x = \sqrt{\frac{\Sigma\left(x_i - \overline{X}\right)2}{N}}$$

$$\sigma_y = \sqrt{\frac{\Sigma\left(y_i - \overline{Y}\right)^2}{N}}$$

$$Cov\left(x, y\right) = \frac{\Sigma\left(x_i - \overline{X}\right)\left(y_i - \overline{Y}\right)}{N}$$

$$\text{Correlation Coefficient} = \frac{Cov\,(x, y)}{\sigma_x \sigma_y}$$

CONTROL CHART VARIABLES

$$\text{Mean} = \frac{\Sigma y_i}{N}$$

$$\text{StdDev} = \sqrt{\frac{\Sigma\left(y - Mean\right)^2}{N-1}}$$

$$\text{CoefVar} = \frac{StdDev}{Mean}$$

PlusNSig =Mean + n •StdDev

MinusNSig =Mean - n •StdDev

SUMMARY REPORT

The following calculations and the results of previous calculations (as noted) are used to generate the summary report:

a. Single-point Surface Area (m^2/g)

$$S_{1PT} = \frac{\left[Q\left(1-P\right)\right] \times CSA\left(6.023 \times 10^{23}\right)}{22414cm^3 \times STP\left(\frac{10^{18}nm^2}{m^2}\right)}$$

where

P	=	pressure closest to 0.3 of the relative pressure points designated for surface area calculations.
Q	=	quantity adsorbed corresponding to P

b. Multi-point Surface Area. See *"BET Surface Area" on page 12 - 1*

c. Langmuir Surface Area. See *"Langmuir Surface Area" on page 12 - 32*

d. t-Plot Micropore Surface Area. See *"t-Plot" on page 12 - 45*

e. t-Plot External Surface Area. . See *"t-Plot" on page 12 - 45*

f. BJH Cumulative Adsorption

g. BJH Cumulative Desorption

h. Adsorption Total Pore Volume

i. Desorption Total Pore Volume

j. t-Plot Micropore Pore Volume.. See *"t-Plot" on page 12 - 45*

k. Freundlich. See *"Freundlich Isotherm" on page 12 - 23*

l. Temkin. See *"Temkin Isotherm" on page 12 - 46*

m. Alpha-S. See *"Alpha-S Method" on page 12 - 1"Alpha-S Method" on page 12 - 1*

n. DFT Pore Size and DFT Surface Energy. See *"DFT (Density Functional Theory)" on page 12 - 11*

o. Nanoparticle Size

$$d = \frac{6 \times 10^4}{A\rho}$$

where

ρ	=	sample density
A	=	BET surface area
d	=	side length (for cubic particles or diameter (for spherical particles)

p. Dubinin-Astakhov Micropore Surface Area. See *"Dubinin-Astakhov" on page 12 - 15*

q. Dubinin-Astakhov Micropore Volume. See *"Dubinin-Astakhov" on page 12 - 15*

r. Dubinin-Radushkevich Micropore Surface Area. See *"Dubinin-Radushkevich" on page 12 - 19*

s. Dubinin-Radushkevich Monolayer Capacity. See *"Dubinin-Radushkevich" on page 12 - 19*

t. MP-Method Cumulative Surface Area of Pores

S_{total}l = S_{cum}'i, See *"MP-Method" on page 12 - 35*for the last collected data point used in the MP-method Calculations, and the range of hydraulic pore radii over which the cumulative surface area was computed.

u. MP-Method Cumulative Pore Volume of Pores

$V_{total} = V_{cum,i}$'

See *"MP-Method" on page 12 - 35* for the last collected data point used in the MP-method calculations, and the range of hydraulic pore radii over which the cumulative pore volume was computed.

v. Average Pore Hydraulic Radius (Å)

$$\bar{r} = \frac{V_{total}}{S_{total}} \times 10^4$$

w. Horvath-Kawazoe. See *"Horvath-Kawazoe" on page 12 - 24*

T-PLOT

A least-squares analysis fit is performed on the (t_i, $N_{ads,i}$) data pairs where t_i is the independent variable and $N_{ads,i}$ is the dependent variable. Only the values of t_i between t_{min} and t_{max}, the minimum and maximum thickness, are used. The following are calculated:

a. Slope (S cm^3/g-Å STP)
b. Y-intercept (Y_{int} cm^3/g STP)
c. Error of the slope (S_{err} cm^3/g-Å STP)
d. Error of the Y-intercept (YI_{err} cm^3/g STP)
e. Correlation coefficient

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

External Surface Area (m^2/g):

$$\frac{SV_{mol}}{F \times 22414 cm^3 STP} \times 10^4$$

where

10^4	=	unit conversions
F	=	surface area correction factor, user-entered on the t-Plot Report Options screen
V_{mol}	=	liquid molar volume, from the fluid property information

Micropore Surface Area (m^2/g):

$$SA_{\mu\rho} = SA_{total} + SA_{ext}$$

where SA_{total} is the BET surface area if the user enabled the BET report exclusively, or Langmuir surface area if the user enabled the Langmuir report exclusively. If neither report has been selected, SA_{total} is the BET surface area value calculated using a set of default parameters.

Micropore Volume (cm^3 liquid/g):

$$\frac{Y_{int} V_{mol}}{22414 cm^3 STP}$$

TEMKIN ISOTHERM

The Temkin isotherm has the form

$$\frac{Q}{Q_m} = \frac{RT}{q_0\alpha} ln\left(A_o P\right)$$

where

A	=	$a_0 \exp, \left\{\frac{-q_0}{RT}\right\}$ where α_0 and A_0 are adjustable constants
P	=	equilibrium pressure measured by gauge at temp T_{amb}
q_0	=	the differential heat of adsorption at zero surface coverage
Q	=	quantity of gas adsorbed
Q_m	=	quantity of gas in a monolayer
R	=	molar gas constant $8.31441 \times 10^{-3} \frac{kJ}{molK}$
T	=	bath temperature

In terms of quantity adsorbed

$$Q = \frac{RTQ_s}{q_0\alpha}\left[\ln A_0 + \ln\left(\frac{P}{P_0}\right)\right]$$

Thus, the plot of the natural log of absolute pressure vs. quantity adsorbed yields a straight line with slope $\frac{RTQ_m}{q_0}$ and intercept $\ln A \frac{RTQ_m}{q_0 a}$.

THERMAL TRANSPIRATION CORRECTION

During data reduction, thermal transpiration correction is applied to the data if the user selected *Apply thermal transpiration correction* from the *Report Options* window. Starting with the first collected pressure, the following calculations are performed until the pressure ratio (PC/P) is greater than or equal to 0.99.

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

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

$$F = \frac{1}{\alpha Y^2 + \beta Y + \mu}$$

$$P = \left(1 - F\left(1 - \sqrt{\frac{T_{bath}}{T_{amb}}}\right)\right)$$

where:

α	=	Weber's coefficient, 0.033
β	=	Weber's coefficient, 0.245
$F, Y\ \mu$	=	intermediate values for subsequent calculations
G	=	Weber's coefficient, 2.5
H	=	Weber's coefficient, 2
MD	=	thermal transpiration hard sphere diameter of gas (Å), from the *Adsorptive Properties* window
P	=	equilibrated collected pressure measured by gauge at temp T_{amb}
SD	=	inside diameter of sample tube (mm), from the *Report Options* window
T	=	average temperature $\frac{T_{amb}+T}{2}$
T_{amb}	=	room temperature (298 K)
T_{bath}	=	analysis bath temperature (K), from the *Po and Temperature Options* window

THICKNESS CURVE

For each point designated, the following parameters are used in thickness curve calculations:

C_1	=	parameter #1
C_2	=	parameter #2
C_3	=	parameter #3
$P_{rel,\ i}$	=	relative pressure for the ith point (mmHg)
t_i	=	thickness for ith point

REFERENCE

Interpolated from table.

KRUK-JARONIEC-SAYARI

$$t = \left(\frac{C_1}{C_2 = \log\left(P_{rel,\ i}\right)} \right)^{c3}$$

HALSEY

$$t_i = C_1 \left[\frac{C_2}{\ln\left(P_{rel,\ i}\right)} \right]^{C_3}$$

Halsey[1)]

HARKINS AND JURA

$$t_i = \left[\frac{C_1}{C_2 - \log\left(P_{rel,\ i}\right)} \right]^{C_3}$$

Harkins and Jura[2)]

1) Halsey, G., J. Chem. Phys. 16, 931-937 (1948).
2) Harkins, W.D. and Jura, G., J. Chem. Phys. 11, 431 (1943).

BROEKOFF-DE BOER

$$\log\left(P_{rel,i}\right) = \frac{C_1}{t^2_{,i}} + C_2 \exp\left(c_3 t_i\right)$$

CARBON BLACK STSA

$$t_i = C_1\left(P_{rel,i}\right)^2 + C_2\left(P_{rel,i}\right) + C_3$$

WEIGHTED METAL PARAMETERS

Chemisorption

The stoichiometry factor, atomic weight, and density used in calculations are averages weighted by the number of moles of each active metal. For example, the average stoichiometry factor is

$$\overline{S} = \frac{\sum_i n_i S_i}{\sum_i n_i}$$

Where

n_i = number of moles or metal

$$n_i = \frac{\alpha\beta X}{XW_m + YW_0}$$

where

α	=	fraction of sample mass
β	=	fraction reduced
X	=	number of metal atoms in the oxide
Y	=	number of oxygen atoms in the oxide
W_m	=	atomic weight of metal
W_o	=	atomic weight of Oxygen

Average density and atomic cross-sectional area are calculated similarly.

13 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.

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).

MODELS BASED ON STATISTICAL THERMODYNAMICS

Included in this group are methods that model the adsorption system in terms of forces acting between individual molecules.

Theoretical Background

Traditional adsorption theories attempt to describe experimental adsorption isotherms with an isotherm equation containing a small number of parameters. At a minimum, these parameters include the extent of the surface, such as the monolayer capacity (Q_m), and the molar intensity of the gas-surface interaction, such as the Langmuir "K" constant or the BET "C" constant. In some equations, additional parameters take into account the lateral interaction of adsorbed molecules with each other. Other theories, such as the Dubinin-Astakhov approach, also include parameters for the effect of adsorbent porosity.

Instead of this classical kinetic or phenomenological approach, we can use a molecular-based statistical thermodynamic theory that allows us to relate the adsorption isotherm to the microscopic properties of the system: the fluid-fluid and fluid-solid interaction energy parameters, the pore size, the pore geometry, and the temperature.

The following example is given so that you may understand how such a theory is constructed:

> A clean sample of a solid material containing slit-shaped pores of a single width is placed in an evacuated space. It is kept at a fixed temperature as a known quantity of pure argon gas is admitted into the space surrounding the sample. The pressure within the space is recorded over time. In this situation, the pressure falls rapidly from its initial value and gradually approaches a steady reading, called the equilibrium pressure. The amount adsorbed corresponds to the quantity of gas effectively removed from the gas phase by the solid surface. A graph that plots amount adsorbed versus equilibrium pressure is called an adsorption isotherm.
>
> Under such conditions, the argon atoms that randomly enter the pore space feel the presence of the solid surface as the action of an external attractive force (the dispersion forces or Van der Waal's forces) and spend more time near the surface. As a result, the space near the surface acquires a greater average density of argon atoms than regions farther removed.
>
> If the equilibrium distribution of the gas atoms near the surface could be described as a function of pressure and the molecular properties of the components of the system, then a model could be constructed for the adsorption isotherm for the system. Modern physical chemistry provides several ways to calculate this distribution. All these methods are based on the fundamental thermodynamic law that such a system adopts a configuration of minimum free energy at equilibrium. Also needed is a description of the pairwise interaction energy between atoms, U(s), commonly given by a Lennard-Jones potential:
>
> $$U\left(s\right) = 4\epsilon\left(\frac{\sigma}{s}\right)^{12} - \left(\frac{\sigma}{s}\right)^{6}$$
>
> where
>
> ε = a characteristic energy of the adsorptive,
> σ = the diameter of the adsorptive molecule, and
> s = the separation distance.

Molecular Simulation Methods

Two simulation techniques are commonly used to determine the distribution of gas molecules in a system in equilibrium: the molecular dynamics method and the Monte Carlo method. Both of these are used as reference methods because their results are considered exact.

Molecular Dynamics Method

In the molecular dynamics method, the position and velocity of individual gas particles are calculated over time at very short intervals. This method takes into account both the forces acting between the gas particles themselves and those acting between the gas particles and the atoms of the simulated surface. As the simulated particles collide with each other and with the surface, the average concentration of particles in the space near the surface is calculated; this calculation yields the amount of gas adsorbed.

This method can be thought of as a way to determine the chronological record of the movement of each particle in the system using time steps of 10-14 seconds. Although the mathematics are simple, the number of calculations required for a system of even a few hundred particles is astronomical and challenges even the fastest computers.

Monte Carlo Method

In the Monte Carlo method, determination of the system equilibrium distribution begins with an assumption (which may be only approximate) about the initial configuration of particles in the system. The system is “equilibrated” through a process of randomly selecting one particle and conditionally moving it a random distance in a random direction.

If the move results in a configuration of *lower total energy*, then the move is completed and another particle is randomly selected to be moved.

If the move results in a configuration of *higher energy*, a probability for that event is calculated, and a random number between zero and one is generated. If the generated number is smaller than the probability of the event, then the move is accepted; otherwise, another particle is selected and the process is repeated. This process continues until the average total energy of the system no longer decreases; at this point, average configuration data are accumulated to yield the mean density distribution of particles in the system.

Monte Carlo simulations require considerably less computation time than molecular dynamic simulations and can yield the same results; however, neither method provides a really practical way to calculate complete isotherms.

Density Functional Formulation

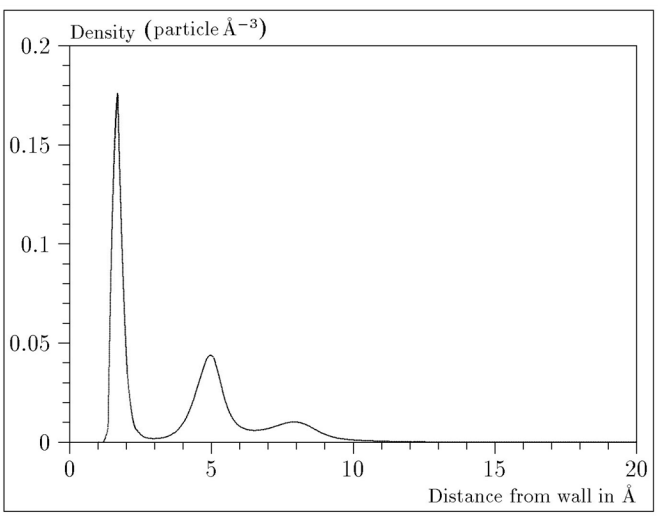
Density functional theory offers a practical alternative to both molecular dynamic and Monte Carlo simulations. When compared to reference methods based on molecular simulation, this theory provides an accurate method of describing inhomogeneous systems yet requires fewer calculations. Because the density functional theory provides accuracy and a reduced number of calculations, it is the basis embodied in the DFT models.

The system being modeled consists of a single pore represented by two parallel walls separated by a distance H. The pore is open and immersed in a single component fluid (adsorptive) at a fixed temperature and pressure. Under such conditions, the fluid responds to the walls and reaches an equilibrium distribution. In this condition (by the definition of equilibrium), the chemical potential at every point equals the chemical potential of the bulk fluid. The bulk fluid is a homogenous system of constant density; its chemical potential[1)] is determined by the pressure of the system using well-known equations. The fluid near the walls is not of constant density; its chemical potential is composed of several position-dependent contributions that must total at every point to the same value as the chemical potential of the bulk fluid.

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 13 - 1* at the end of this appendix.

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

Figure 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.



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).

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 teh corresponding points of the isotherm.

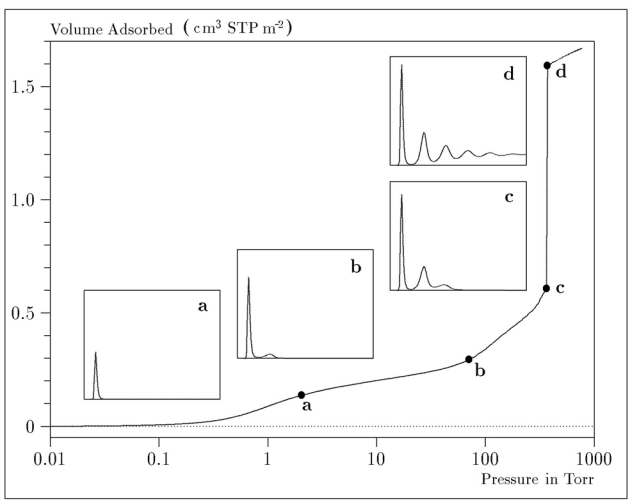


Figure 2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

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.

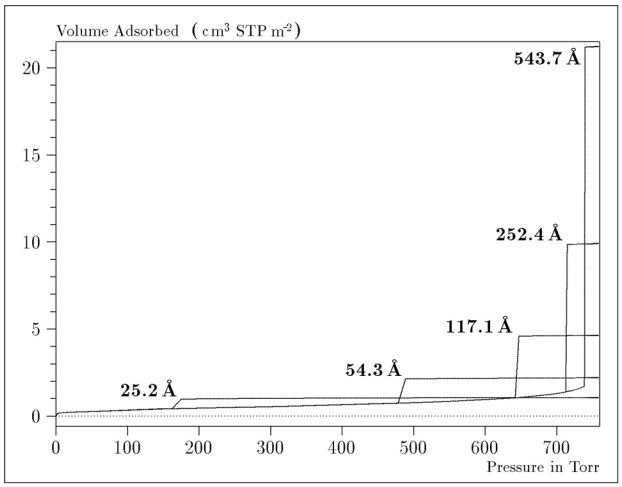


Figure 3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

Figure 4 shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.

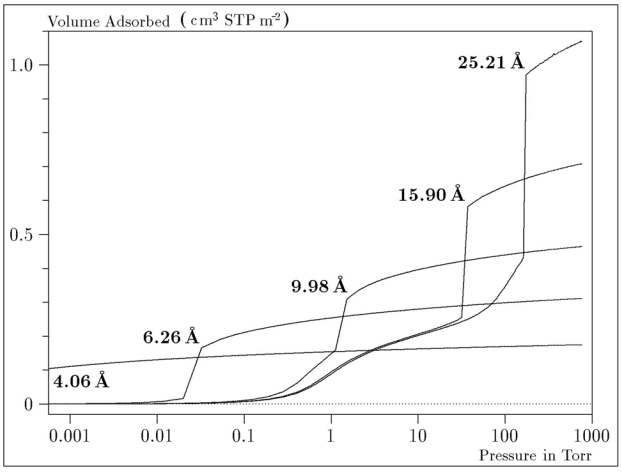


Figure 4. Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

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 13 - 1* [items 3 and 4]), model isotherms were calculated for a wide range of adsorptive energies to a relative pressure of 0.6. The model makes no provision for pore filling in the micropore region. If the sample solid contains small mesopores, the isotherm data should be truncated (using the *Select Data Points* window) to a suitably low relative pressure to avoid trying to fit this region; mesopore filling reports as a large area of low energy in the calculated distribution of adsorptive potential.

The surface energy is reported in terms of the effective Lennard-Jones interaction parameter, ie, for the adsorptive/adsorbent pair divided by Boltzmann constant. The units are therefore Kelvin.

N2 - Cylindrical Pores - Oxide Surface
AR - Cylindrical Pores - Oxide Surface

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using a combination of statistical mechanical calculations and experimental observations for macroporous silicas and MCM-41 mesoporous silicas as well as zeolites. The pore-filling pressures were determined as a function of the pore size from adsorption isotherms on MCM-41 materials characterized by X-ray and other techniques. The variation of the pore fluid density with pressure and pore size has been accounted for by density functional theory calculations. The N2 model reports pore sizes ranging from 3.8 to 387 Å and the AR model from 3.8 to over 500 angstroms.

References: M. Jaroniec, M. Kruk, J.P. Olivier, and S. Koch, "A New Method for the Accurate Pore Size Analysis of MCM-41 and Other Silica-Based Mesoporous Materials," Proceedings of COPS-V, Heidelberg, Germany (1999).

N2 – Cylindrical Pores – Pillared Clay Surface (Montmorillionite)

Geometry: Cylinder
Substrate: Crystalline Silicate
Category: Porosity
Method: Nitrogen at 77K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFT) calculations and experimental isotherms for reference samples of montmorillionite.The construction method for the hybrid models was analogous to that described in the first reference below (Jaroniec et al,1999). The additional references add additional theoretical details as well as examples of the application of the model to pillared clay catalysts.This model reports pore widths from 3.8 to 387 angstroms.

References:

Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V* , Unger, et al, Eds, Elsevier, Amsterdam, 2000.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of a Pillared Interlayered Clay (PILC) from a Hybrid Density Functional Theory (DFT) Method," *The Journal of Physical Chemistry B*; 2001, 105(3), 623-629.

M. L. Occelli, J. P. Olivier, J. A. Perdigon-Melon, and A. Auroux, "Surface Area, Pore Volume Distribution, and Acidity in Mesoporous Expanded Clay Catalysts from Hybrid Density Functional Theory (DFT) and Adsorption Microcalorimetry Methods," *Langmuir* 2002, 18, 9816-9823.9b.

James P. Olivier, "The Importance of Surface Heterogeneity in Developing Characterization Methods." *6th International Symposium on the Characterization of Porous Solids*, Studies in Surface Science and Catalysis 144, Elsevier, 2002.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of Pillared Rectorite Catalysts from a Hybrid Density Functional Theory Method," *Microporous and Mesoporous Materials* 2003, 57, 291-296.

C02 - DFT Model

Geometry: Slit
Substrate: Carbon
Category: Porosity
Method: Carbon dioxide at 273 K

Model isotherms were calculated using the non-local prescription of Tarazona, employing molecular parameters derived from the known bulk properties of carbon dioxide.

AR - Modified Density Functional Model

Geometry: Free surface
Substrate: Any
Category: Surface energy
Method: Argon at 87K

This model was produced in the same manner as the N2 Modified Density Functional model listed earlier, except applicable to argon adsorbed at 87.3 K.

N2 - Tarazona NLDFT, Esf = 30.0K

Geometry: Cylinder
Substrate: Oxide
Category: Porosity
Method: Nitrogen at 77K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a cylindrical pore geometry. The wall potential used is k = 30 K, typical for a silica or alumina surface.

This model file is particularly useful for sizing zeolites or zeolite containing materials that have substantial micropore volume. The reported pore size range is 3.8 to 387 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Slit Pores by NLDFT
Ar - Carbon Slit Pores by NLDFT

Geometry: Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a slit-like pore geometry. These models are slightly different from N2-DFT and Ar-DFT models that were calculated using NLDFT with density independent weighting functions.

The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Finite Pores, As=6, 2D-NLDFT
Ar - Carbon Finite Pores, As=6, 2D-NLDFT

Geometry: Finite Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions assuming 2D model of finite slit pores having a diameter-to-width aspect ratio of 6.

This model is particularly useful for microporous carbon materials. The reported pore size range is from 3.5 to 250 angstroms

References: Jacek Jagiello and James P. Olivier. "A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.," The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N2 - Carbon Finite Pores, As=12, 2D-NLDFT
Ar - Carbon Finite Pores, As=12, 2D-NLDFT

Geometry: Finite Slit
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the same methods and assumptions that were used in the model above except in this model, the aspect ratio is equal to 12.

These two finite pore models may be used as a research tool in conjunction with independent analytical techniques such as high-resolution transmission electron microscopy (HRTEM) and / or X-ray diffraction (XRD) to obtain comprehensive information about the structure of studied carbon material.

References: Jacek Jagiello and James P. Olivier. "A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.," The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N2 - Carbon Cylinder, single-wall nanotube by NLDFT
Ar - Argon Cylinder, single-wall nanotube by NLDFT

Geometry: Cylinder
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the graphitic surface of an infinitely long cylinder.

This model is particularly useful for characterizing carbon single-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Cylinder, multi-wall nanotube by NLDFT
Ar - Argon Cylinder, multi-wall nanotube by NLDFT

Geometry: Cylinder
Substrate: Carbon
Category: Porosity
Method: Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and multiple concentric graphitic surfaces of infinitely long cylinders.

This model is particularly useful for characterizing carbon multi-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987)

Ar - Zeolites H-Form by NLDFT

Geometry: Cylinder
Substrate: Zeolite
Category: Porosity
Method: Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is particularly useful for characterizing oxides and H+ and (NH4)+ exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

Ar - Zeolites Me-Form by NLDFT

Geometry:	Cylinder
Substrate:	Zeolite
Category:	Porosity
Method:	Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is similar to the model above, but it more appropriate is for characterizing alkali metal exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

Models Based on Classical Theories

Both surface energy distribution and pore size distribution may be evaluated using classical approaches to model kernel functions for use with equation (1) of the DFT Theory in *"DFT (Density Functional Theory)" on page 12 - 11*. 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:

$$t = 3.54\left(\frac{-5.00}{\ln(P/Po)}\right)^{1/3}$$

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:

$$t = \left(\frac{13.99}{0.034 - \log(P/Po)} \right)^{1/2}$$

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).

14 Blank and Sample Compression Corrections for Mercury Porosimetry

Baseline errors in AutoPore V data are errors that occur even when no sample is placed in the sample bulb and when a zero intrusion or extrusion volume of mercury would be expected as the pressure is increased to its maximum pressure and then decreased again. The material which follows relates the causes of these errors and discusses ways to minimize and compensate for them when maximum accuracy is required.

Baseline Errors

When the AutoPore applies pressure to the mercury, penetrometer, and surrounding high pressure oil, compression occurs.

Compressibility effects account for a substantial portion of the baseline errors. The penetrometer bulb and capillary are made of glass which decreases in linear dimensions by about 0.8% and in volume by 2.3% at 60,000 psia. If the mercury were incompressible, a typical penetrometer having a 400 microliter capillary and a 5 milliliter bulb would experience a rise of mercury in the capillary of about 124 microliters or 31% of the capillary. Fortunately, mercury compresses also, but slightly more than glass such that the capillary actually falls some as the pressure is increased. The compressibility amounts to about 150 microliters in this example leaving a net fall of 26 microliters or about 6% of the capillary. The oil which surrounds the penetrometer and transmits the pressure to the mercury compresses at more than 10 times the rate of the mercury and occupies only 3/4 the original volume at 60,000 psia. Some of the oil is in the electric field of the capacitor, especially around the sample bulb and its connection to the exterior. The dielectric constant of the oil increases with its density. This contributes an increasing capacitance which cancels some of the decrease due to the net fall of mercury with compression.

Other effects caused by compression arise from the plastic insulators which are used on the penetrometer bulb base to prevent an electrical short circuit. Not only does the plastic compress almost as much as the oil, but it lags behind and only slowly assumes its final density. This is especially pronounced upon release of pressure where the plastic may continue to increase in dimensions for almost an hour. It also tends to increase the dielectric constant and capacitance with increasing pressure. The pressure vessel expands as the internal pressure is increased and, like the plastic, requires considerable time to stabilize. The resulting changes in spacing from the sample bulb to the walls and bottom causes a decrease in capacitance. Micromeritics has minimized this effect by making the initial spacings as large as is practical.

Another effect, and the one most difficult to predict, arises from the similarity of the penetrometer to a thermometer. This would not be troublesome if its temperature could be maintained constant, but compression of the surrounding oil causes a temperature rise of nearly 50 ºC in the oil and a smaller change in the glass and mercury. How quickly this heat is transferred to the mercury depends upon how rapidly the pressure is being increased, the relative amounts of oil and mercury present, and how recently the vessel has been previously cycled and the metal and oil warmed relative to the penetrometer. Release of the pressure causes the inverse effect, chilling the oil and setting up a reversal of the heat flow. The thermal gradient across the glass of the penetrometer may be considerable such that little benefit may be derived from precisely equalizing the temperature coefficients of the mercury and glass. As might be expected this problem is worst when the sample bulb is large and the capillary volume small. Choosing the right penetrometer helps minimize this effect. Make sure the sample nearly matches the size of the sample bulb and that the capillary volume is large enough to satisfy intrusion.

APPROACHES FOR ERROR COMPENSATION

Situations arise where the typical errors of about 1.0% of capillary volume are significant or where the errors exceed this level due to unfavorable sample characteristics. Most commonly, this happens when one of the following is encountered: 1) The amount of sample available is so limited that the intrusion volume is only a small fraction of the smallest diameter capillary; 2) adequate sample is available but the porosity is so low that a limited amount of the smallest capillary is used even though the largest sample bulb is filled; 3) the sample is of small or moderate porosity and its compressibility or thermal properties differ considerably from those of mercury; 4) accuracy and reproducibility specifications have been imposed at levels tighter than the typically expected levels for mercury porosimetry. In such cases “blank corrections” may be used to advantage.

Micromeritics’ AutoPore provides four different ways to apply blank corrections. The first, and simplest, is by use of stored formulas based upon averages of large numbers of blank runs on mercury-filled penetrometers under varying rates of pressure build and release. No provisions are made for entering compressibility data or thermal data since these numbers are seldom known and the formulas would become very complex. Typical examples of blank runs are shown in Figures 1, 3 and 7. Typical examples of formula blank correction of data are shown in Figures 2 and 6. It is very important that trial blank runs be made when applying these formulas to ensure that a reasonable degree of correction is actually attained under the running conditions being used.

The second technique is apt to be much more useful. It permits the user to run a blank run, store the results using the exact run conditions and penetrometer type to be used for the real sample, and subtract this result from other runs. Examples of correction by subtracting a blank run file are shown in Figures 4 and 8.

The third technique provides the highest degree of compensation possible and can be attained when the exact penetrometer to be used later is loaded with a nonporous sample of the same weight and material as the porous sample to be run later. When analyzed, the non-porous sample will expose all the aforementioned compressibility effects which can then be subtracted from the porous sample run. This third technique has the advantage of compensating for differences in compressibility and thermal effects between mercury and the sample material. Care should be exercised that the interval between runs, oil temperature, and penetrometer temperature, and any other initial conditions are made as nearly identical as possible. Figure 9 is a typical baseline run so obtained. Figure 10 is a subsequent blank run corrected using the Figure 9 data and shows the actual degree of correction attained.

Besides running blank runs, correction files may be created by manually entering the data. This fourth technique allows entry of the average of several blank runs, assuring a representative correction.

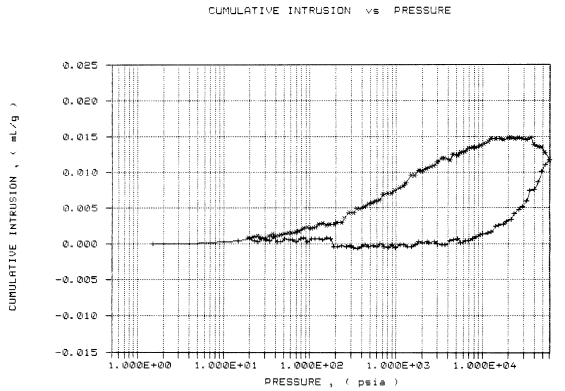


Figure 1

A blank run on a 5-mL powder penetrometer with a 1.1-mL stem volume. The rise in the initial depressurization data is primarily caused by thermal effects. As the hydraulic fluid is allowed to expand, it cools. this in turn cools the mercury in the penetrometer, causing it to contract and recede in the stem, giving the appearance of positive intrusion during depressurization.

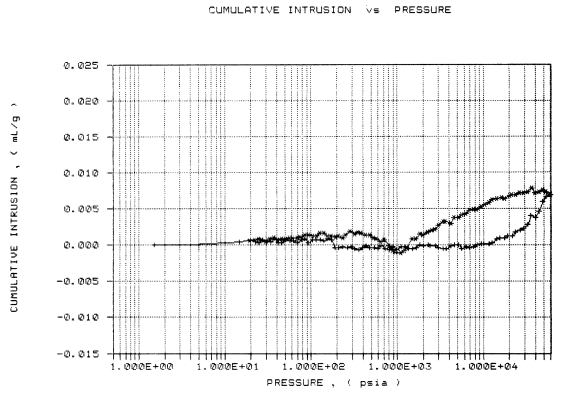


Figure 2

The difference between the blank data in Figure 1 and the formula blank correction for a run under the same conditions. The formula cancels some of the error, but does an imperfect job in this case.

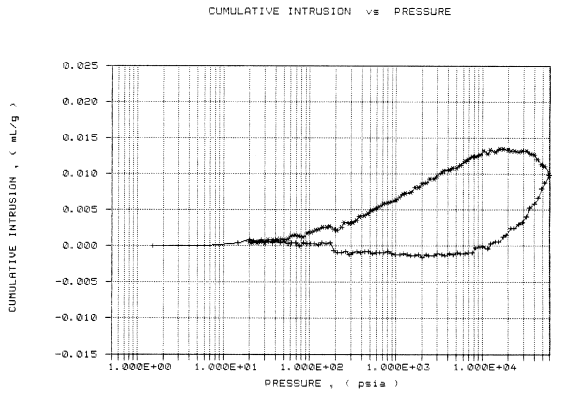


Figure 3

Another blank data set taken under identical conditions to those for Figure 1. The similarity between the two blank data sets is an indication of the excellent repeatability of blank runs.

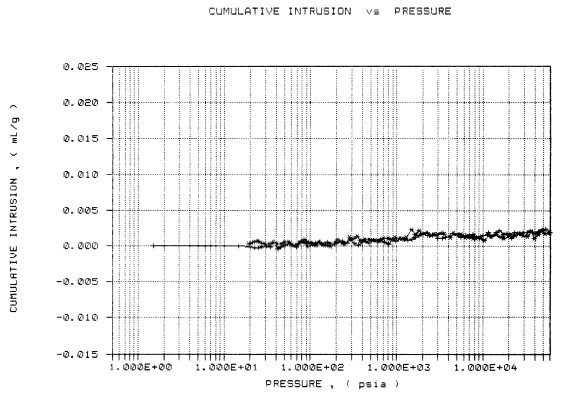


Figure 4

The difference between the blank data from Figure 1 and the blank data from Figure 3. This demonstrates that blank data collection and subtraction is a powerful method for accurately removing blank error from sample data.

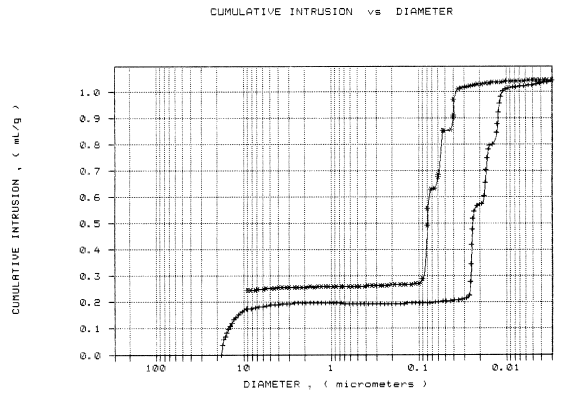


Figure 5

Uncorrected data from analysis of a sample of controlled pore glass made of a mixture of three pore sizes. Note the three distinct regions of intrusion between 0.03 and 0.01 micrometers on the pressurization curve, and the corresponding extrusion regions. The apparent intrusion at sizes above 10 micrometers is due to interparticle filling. The apparent intrusion between 0.01 and 0.003 micrometers, and the “loop” in the extrusion curve from 0.04 to 0.003 micrometers, are due to a combination of sample compression and blank error. There is no actual intrusion in this region.

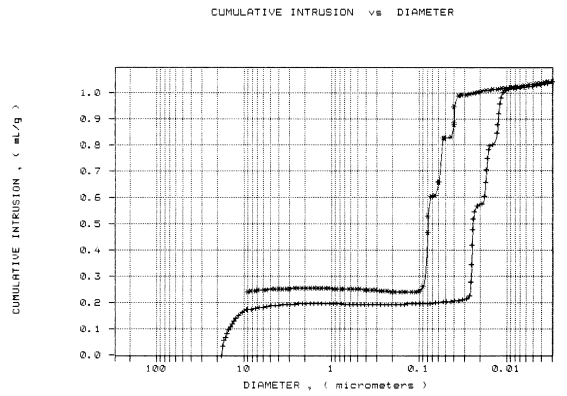


Figure 6

The data from Figure 5 with the formula blank correction applied. Note that the rise at the top due to blank error has been removed, but the apparent intrusion due to sample compression remains. This is because the formula makes no attempt to account for sample compression.

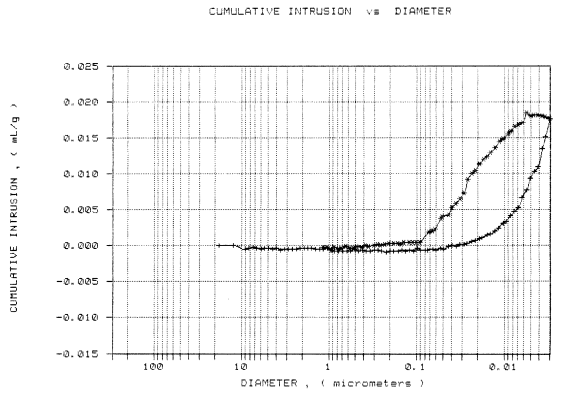


Figure 7

A blank run with the same type of penetrometer under the same conditions as the sample in Figure 5. It is dominated by the initial increase between pressurization and depressurization, primarily due to thermal effects.

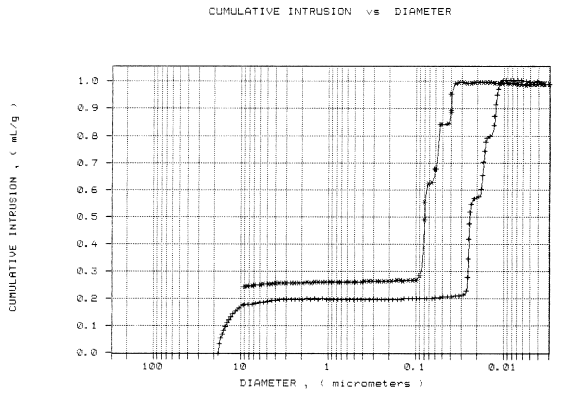


Figure 8

The sample data from Figure 5 corrected by subtracting the blank data from Figure 7. Note that practically all of the blank error and compression data have been removed, leaving only the filling curve and the actual intrusion. The sample compression is effectively cancelled because the compression coefficient of mercury is close to that of the controlled pore glass used as sample. Many solid materials have compression coefficient fairly close to that of mercury, making this a very effective means of blank correction in many cases.

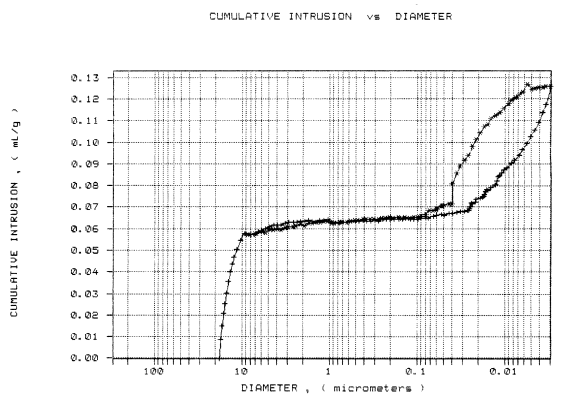


Figure 9

Uncorrected data from an essentially nonporous sample of the same type of glass shown in Figure 5. The weight of sample used was approximately equal to the weight of porous sample analyzed, so that the same volume was occupied. Note the filling curve and the blank error “loop.” The slight incline of the intermediate plateau and the angle of the “loop” are due to compression of the sample.

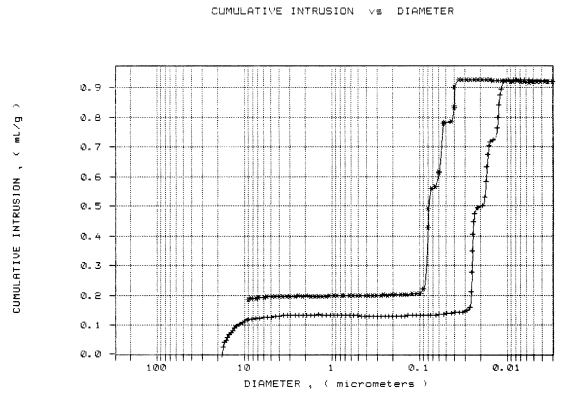


Figure 10

The difference between the porous sample data of Figure 5 and the nonporous sample data of Figure 9. Some of the filling curve has been removed, as well as all blank error and sample compression effect, leaving an accurate picture of the actual intrusion. This is the preferred method of blank correction, especially for materials with compression coefficients substantially different from that of mercury, and where maximum accuracy is desired.

15 Computing Volumetric Compressibility of a Sample Material

Ideally, you should choose a sample material that is completely non-porous; if this is not the case, then you should choose the pressure range over which the compressibility test is conducted such that no pore filling occurs within it. Closed pores may not always cause volume changes during testing but they may alter the results due to stress concentrations around them or because of their effects upon measured density. Closed pores may also abruptly fail and even become open during testing and cause invalid compressibility results to be reported. In some cases, such as the testing of plastic foams at low pressures, the presence of closed pores may be acceptable and expected.

The sample weight and sample density must be known and available to a resolution and accuracy at least three significant digits (preferably better) to permit accurate computation of the initial volume of the sample material. Alternatively, an accurate geometric volume of a material such as one containing closed pores (such as plastic foam) may be supplied. Before data reduction can be performed, you must have available a “blank run” file consisting (at least ideally) of a run made with the same penetrometer and accessory hardware that is to be used in the compressibility test and (again ideally) on the same instrument ports as will be used in the compressibility run. The pressure range of the blank run must, at a minimum, fully encompass the planned range to be used in the compressibility measurement and have a minimum of seven uniformly spaced (linear basis) data points inside the planned computation range and with the beginning and ending data points within 5% (pressure) of the planned computation range end points. It is also permissible for the “blank run” to consist of a manually entered data file.

The first and second order isostatic pressure coefficients of volumetric compressibility for mercury over the pressure range from zero psia to 60,000 psia must be known and available. All standard input information such as sample material identity, equilibration times, evacuation information, penetrometer constants, etc. that would be required for standard runs is required for a compressibility run. Note that sample volume, bulk volume/density, and skeletal volume/density as measured during the mercury porosimetry run are, in general, far too imprecise to yield good results if used in the compressibility computations. For this reason, you must enter very accurate material density and sample weight values to be used in computing an accurate initial sample volume or, alternatively, directly enter a measured initial sample material volume.

The pressure table entered must contain at least seven pressure points uniformly spaced (on a linear basis), with these points coinciding as closely as possible to those in the blank run which is to be used along with the data in the final computation. As indicated above, the pressure values of the end points achieved during the run must be within 5%

Blank Page

16 Use of the Maximum Intrusion Volume Option

Using the maximum intrusion volume option allows routine analyses with fewer points in a pressure table while maintaining good resolution. However, use of the maximum intrusion volume requires some knowledge of the total pore volume of the sample to be analyzed. You should use about 2% of the sample's total pore volume as the maximum intrusion volume. This would give about fifty points for the intrusion pore spectrum and should be adequate to completely characterize most samples. The AutoPore IV will automatically add a pore spectrum point any time it sees an increment of intrusion equal to the maximum intrusion volume specified.

Care should be taken not to use too small a maximum intrusion volume. Use of a value less than 0.4% of the total intrusion volume will cause too many points to be taken at lower pressures. The total of 1000 data points will be exhausted and the analysis will terminate prematurely.

Use of too small a maximum intrusion volume can also cause points to be taken too close together on the pressure axis. If this causes pressures to be taken within the target pressure tolerance of each other, an apparent pressure decrease may be reported during the intrusion sequence. A reported pressure drop greater than 10 psi or 0.5% of the target pressure will be interpreted as the end of the intrusion segment. Reported summary data (such as total intrusion volume) will be reported at this point, rather than at the maximum pressure as intended. Data for graphs other than cumulative intrusion volume will also be terminated at this point.

Blank Page

17 Pore Surface Area Computation

It is sometimes asserted that pore wall surface area computed on the basis of the work required to immerse a surface in mercury is superior to assuming the pores are cylindrical and calculating area from geometric relationships. What those who make the assertion fail to recognize is that mathematically and in practice, the two computations are identical as shown below.

Work

The reversible work dW required to immerse an area dA of a non-wetting object in mercury[1] is

$$dW = \gamma \cos \Theta dA \tag{1}$$

where γ is the surface tension of mercury and θ its contact angle with the object. In the case of mercury and pores, this work is supplied when the external pressure P forces a volume of mercury dV into pores. Equation 1, therefore, becomes

$$\gamma \cos \Theta dA = -PdV \tag{2}$$

Assuming that γ and θ do not vary with pressure, equation 2 can be written

$$A = -\frac{\int PdV}{\gamma \cos \theta} \tag{3}$$

which, expressed for evaluation from pressure-volume mercury penetration data, becomes

$$\Sigma \quad A = -\frac{\Sigma P \quad V}{\gamma \cos \theta} \tag{4}$$

[1] Rootare, H.M. and Prenzlow, C.F., "Surface Areas from Mercury Porosimeter Measurements," J. Phys. Chem., 71, 2733-6 (1967).

CYLINDRICAL GEOMETRY

The basic relationship describing the penetration of mercury into a cylindrical pore of diameter D derived from equating the applied pressure to the resisting surface tension[1] is

$$PD = -4\gamma\cos\Theta \tag{5}$$

The relationship among wall area, diameter, and volume for a cylinder is

$$A = \frac{4V}{D} \tag{6}$$

Combining equations 5 and 6, yields

$$A = \frac{PV}{\gamma\cos\Theta} \tag{7}$$

which, as before, when written for evaluation from pressure-volume mercury penetration data, becomes

$$\Sigma \quad A = -\frac{\Sigma P \quad V}{\gamma\cos\theta} \tag{8}$$

[1] Washburn, E.W., "Note on a Method of Determining the Distribution of Pore Sizes in a Porous Material," Proc. Nat. Acad. Sci., 7, 115-6 (1921).

18 DATA REDUCTION

Data for presentation in tabular and plot form is calculated in the following manner:

P_i	=	head-corrected pressure as stored
V_{ri}	=	intrusion volume as stored
q	=	user-entered contact angle
g	=	user-entered surface tension
W_s	=	user-entered sample mass
W_p	=	user-entered mass for penetrometer
W_{psm}	=	user-entered mass for penetrometer + sample + mercury
V_p	=	user-entered volume for penetrometer
V_c	=	user-entered volume for capillary (stem)
V_{bup}		bulk volume at the filling pressure
V_{bup}		bulk volume at the user-entered pressure
Y_m		user-entered density for mercury

$$\text{WASHCON=Washcon constant} = \frac{10^4 \mu m / cm}{68947.6 \ \ dynes / cm^2 - psia} = 0.145038$$

For all calculations requiring interpolation between collected data points, an Akima[1] method semi-spline is used.

Diameter for the ith point is:

$$D_i = \frac{WASHCON \ \ \gamma(-4\cos\theta)}{P_i}$$

Radius for the ithpoint is:

$$R_i = \frac{D_i}{2}$$

Cumulative specific intrusion volume for the ithpoint is:

$$I_i = \frac{V_i}{W_s}$$

1) "A New Method of Interpolation and Smooth Curve Fitting Based on Local Procedures," Journal of the Association of Computing Machinery, 17(4) 1970, 589-602.

Mean diameter for the ithpoint is:

$$Dm_i = \frac{D_i + D_{i-1}}{2}$$

Incremental specific intrusion volume for the thpoint is:

$$Ii_i = I_i - I_{i-1}$$

Incremental specific pore area for the thpoint is:

$$Ai_i = \frac{4 \times Ii_i}{Dm_i}$$

Cumulative specific pore area for the thpoint is:

$$A_i = Ai_i + Ai_i + \ldots + Ai_i$$

If more than 8 data points are available, differential and log differential specific intrusion volume are calculated as follows.

Differential and log differential data are the 1st derivative of the cumulative specific intrusion volume (all) data as a function of calculated log diameter, normalized by the diameter or log diameter interval. This derivation is comprised of four transformations.

1. Interpolation of cumulative specific intrusion volume vs. log diameter is made to get cumulative specific intrusion volume corresponding to evenly spaced log diameters.
2. The uniform cumulative specific intrusion volume data are then subjected to a 1st derivative calculation, using a 9-point smoothing method. This gives the desired differential data in terms of uniform intervals of collected data.
3. Log differential data are normalized by dividing by the log diameter interval between points. Since the points are evenly log spaced, this interval is the same for all points. Differential data are normalized by dividing by the diameter interval between points. Since the points are evenly log spaced, this interval is larger for larger diameters.
4. Interpolation of the differential or log differential data vs. log diameter is made to get data corresponding to collected data points.

If 8 or fewer data points are available, differential and log differential specific intrusion volume are calculated as:

Differential specific intrusion volume by diameter for the ith point is:

$$Id_i = \frac{-Ii_i}{D_i - D_{i-1}}$$

Log differential specific intrusion volume by diameter is:

$$I1d_i = \frac{-Ii_i}{\log D_i - \log D_{i-1}}$$

Differential specific intrusion volume by radius for the ithpoint is:

$$Ir_i = \frac{-Ii_i}{R_i - R_{i-1}}$$

Log differential specific intrusion volume by radius is:

$$I1r_i = \frac{-Ii_i}{R_i - R_{i-1}}$$

Total intrusion volume is:

$$V_{tot} = V_j$$

where the jthpoint is the first such that:

$$P_{j+1} \leq P_j - 10 \quad \text{and} \quad P_{j+1} \leq P_j \times 0.995$$

Total specific intrusion volume is:

$$I_{tot} = \frac{V_{tot}}{W_s}$$

Percent of total specific intrusion volume for the ith point is:

$$Ip_i = \frac{100 \times I_i}{I_{tot}}$$

Total specific pore area is:

$$A_{tot} = A_j$$

for point j as defined above.

Median diameter by volume is:

$$D_{mv} = D_k$$

where

$$I_k = \frac{I_{tot}}{2}$$

and P_k is interpolated from I_k and the collected data, and D_k is calculated from P_k

Median diameter by area is:

$$D_{ma} = D_k$$

where

$$I_k = \frac{I_{tot}}{2}$$

and P_k is interpolated from A_k and the collected data, and D_k is calculated from P_k.

Average diameter is:

$$D_{av} = \frac{4 \times I_{tot}}{A_{tot}}$$

BLANK CORRECTION BY FORMULA

For equilibration time 6 seconds: $X = \log\left(\frac{T}{6}\right)$

For equilibration time < 6 seconds: $X = 0.0$

$$A_i = \left[1.23 \times 10^{-7} + 2.67 \times 10^{-7} X\right] - V_p\left[1.78 \times 10^{-7} + 1.0 \times 10^{-8} X\right]$$
$$+ V_m\left[1.64 \times 10^{-7} + 2.4 \times 10^{-8}\right]$$

For intrusion,

$$B = A_1 P_i + A_2 P_i^2$$

For extrusion points ≥ 1000 psia,

$$B = A_1 P_i + A_2 P_i^2 + 8.85 \times 10^{-3}\left(1 \frac{P_i}{60000}\right)$$

For extrusion points < 1000 psia,

$$B = A_1 P_i + A_2 P_i^2 + 8.7 \times 10^{-6} P_i$$

Blank-corrected intrusion volume for the i_{th} point is:

$$V_i = Vr_i - B$$

where

T	=	equilibration time in seconds
	=	volume of mercury in penetrometer; where, volume of mercury =
V_m		$\frac{Wpsm - Ws - Wp}{Ym}$
P_i	=	pressure for this data point
V_i	=	corrected intrusion volume

Bulk volume is: $$V_b = V_p - V_m$$

Bulk density is: $$Y_b = \frac{W_s}{V_{bfp} - V_{bup}}$$

Skeletal volume is: $$V_s = V_b - V_{tot}$$

Skeletal density is: $$Y_s = \frac{W_s}{V_s}$$

Porosity % is: $$P_{pc} = \frac{100 \times V_{tot}}{V_b}$$

Percent capillary used is: $$V_{pc} = \frac{100 \times V_{tot}}{V_c}$$

COMPUTATION ALGORITHM FOR VOLUMETRIC PRESSURE COEFFICIENTS OF COMPRESSIBILITY

The data acquired during the AutoPore run is examined to determine that at least seven intrusion data points having progressively ascending pressures have been designated for use in the computation. Note that the intrusion values which will initially be referred to are not SPECIFIC values. They are " total" values never having been divided by the sample material weight. Later, it will be necessary to shift to specific values.

The specified blank data are examined to determine that at least seven blank intrusion data points having progressively increasing pressures are available to use with the specified pressure computation range. Of the seven, the pressure values of the two blank data end points must fall within 5 % (either above or below) of the two end points of the sample material run data.

Interpolation by spline curve polynomial or other suitable technique is to be applied to the blank data to allow computation of blank intrusion volumes at pressures which exactly match those in the experimentally acquired data i.e., take a pressure from the acquired data, enter the interpolation routine and find and save the blank intrusion volume which would correspond to that exact pressure. Repeat this for each pressure value in the acquired data set.

Pointwise at each experimental pressure value, subtract the blank intrusion values as interpolated above from the experimentally acquired data intrusion values to give a "blank-corrected acquired data intrusion values set" or more simply "blank corrected data" for short.

Assume that at each experimental pressure, Pn, the corresponding blank corrected intrusion, V(Pn), is computed using the second order polynomial expression

$$V\left(Pn\right) = V_0 + B^{*}Pn + C^{*}Pn^2$$

where

V_o	=	the exact volume of the sample material computed as the ratio of the sample weight and the sample density supplied by the user or, alternatively, supplied as the pre-measured sample volume by the user;
B	=	the linear pressure coefficient of volumetric compressibility must be a negative real number to avoid violation of fundamental physical laws; and
C	=	the quadratic pressure coefficient of volumetric compressibility.

Construct the summation of differences as follows and solve for the values of B and C which produces the least squared error:

$$\sum_{n=1}^{nN\max}\left[V\left(Pn\right)-\left(V_o+B*Pn+C*Pn^2\right)\right]=\text{minimum}$$

Where **Nmax** is the index of the uppermost blank corrected data point. Now it is necessary to stop using total values and change to the use of specific values; convert the total values **B** and **C** to specific values, **b** and **c**, by dividing them by the sample material volume.

The first and second order pressure coefficients of volumetric compressibility of mercury must be added to the computed first and second order coefficients yielded here. They are expressed in the same units. The resulting values are **b'** and **c'**. It is necessary to do this addition because the blank corrected experimental data actually is a measure of the sample material's differential compressibility compared to that of mercury. One may imagine that a repeat of the blank run could be considered as a test of a some unit volume of mercury itself immersed in the surrounding mercury. The result should be the same as the blank run since in reality nothing has (at least on purpose) been changed. The blank corrected data consists of all zero volume changes with pressure and the **b** and **c** computed from it will likewise both be zero.

Also one should consider the situation which we have experienced wherein a less compressible material such as stainless steel is tested. Since both mercury and glass compress more than does the steel, the mercury column actually must rise in the bore of the penetrometer as the pressure is increased. This is interpreted as a negative intrusion volume change with pressure and leads to the computation of values for b (positive) and c which are physically impossible. Only when they are interpreted as values relative to mercury can they be valid and, by addition of mercury's coefficients respectively, they can be expressed as absolute values.

The values of **b'** and **c'** produced by this calculation will likely be in units of *absolute milliliters per milliliter * psia* and *absolute milliliters per milliliter * psia squared* if internal AutoPore computations are, as expected, performed in these units. Reporting these in alternate units of measure will be required. The most useful alternate units will be *milliliters per milliliter* megaPascal* and *milliliters per milliliter* kpsia* and analogous second order units. Strictly speaking, convention requires that the duplicated fundamental units of measure in the numerator and denominator be eliminated. This results in expressing the first order coefficient as *meters squared per Newton.* This choice also is provided in spite of its less intuitive impression.

FRACTAL DIMENSIONS

Pore space in sedimentary rocks exhibit fractal characteristics. The fractal dimension of these materials has been shown to be an important petrophysical parameter partly because capillary pressure and other transport coefficients scale as power laws of fluid saturation. The scaling exponents often relate to the fractal dimension of the medium. Angulo[1)] , et al, show that fractal dimensions of a quantity related to pore space bulk can be determined by mercury intrusion porosimetry.

According to percolation theory (see reference to Katz and Thompson in Permeability section of this appendix), at some threshold pressure PT, the invading fluid first spans the entire sample, that is, the fluid percolates for the first time. This then produces a geometrical configuration of fluid known as the percolation backbone and pressures from the point of percolation to completion of the backbone are in the *backbone formation* region. At greater pressures, filling of pore cavities behind smaller pore throats continues but without the sudden influx of fluid as observed at the threshold pressure. The backbone is a fractal with fractal dimension DH, but at higher pressures, the geometry of the fluid cluster changes rapidly to another fractal with fractal dimension DV (>DH) of the supporting media.

MIP DATA REDUCTION

In order to calculate a fractal dimension, the threshold pressure, Pthresh, must be known. The threshold pressure is the pressure at which the intrusion volume vs.pressure curve is steepest. This is either a calculated value (if chosen) or the value entered on the Material Properties dialog. It is the same value used in permeability calculations.

If necessary, the value is calculated as follows. First set up an Akima spline for specific intrusion volume (Ii) vs. pressure (Pi) for all points on the first intrusion cycle. This is used to calculate the slope, (dI/dP)i, at each pressure. Use these values to set up another Akima spline for slopes vs. pressures. Finally, use the second Akima spline to find the value of pressure that gives the maximum slope. This is the threshold pressure, Pthresh. In addition, the user must specify the backbone formation and percolation pressure ranges over which calculations are to be performed.

The equation that defines fractal dimension is:

$$I = \alpha\left(P - p^{thresh}\right)^{(3-D)}$$

1) R.F. Angulo, V. Alvarado, and H. Gonzalez, “Fractal Dimensions from Mercury Instrusion Capillary Tests,” II LAPEC, Caracas, March 1992.

I	=	specific intrusion volume
P	=	pressure
P_{thresh}	=	threshold pressure
D	=	the fractal dimension
α	=	proportionality constant

This equation is transformed to the following to make it linear in the unknown parameters.

$$\log(I) = (d - D)\log(P - P_{thresh}) + \log\alpha$$

D and α are calculated by least squares fit to this equation, using all collected points (I_i, P_i) where P_i is in the user-selected range and above the threshold pressure.

MIP Data Reduction

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MATERIAL PERMEABILITY

BACKGROUND

Permeability is a basic permeable medium property that, unlike porosity, cannot be defined apart from fluid flow.

Permeability is the proportionality " constant" between the fluid flow rate and an applied pressure or potential gradient.

Hydrologists, petrologists, and other branches of geology need to measure the intrinsic properties of rock and soils to both store and transmit fluid. These are porosity, permeability, the hydraulic conductivity of Darcy's law, and specific storage.

BASIS OF DATA REDUCTION METHOD TO BE USED

1. A.J. Katz and A.H.Thompson, Quantitative prediction of permeability in porous rock: Physical Review, Series B, Vol. 34, pp. 8179-8191 (1986).
2. A.J. Katz and A.H. Thompson, "Prediction of Rock Electrical Conductivity From Mercury Injection Measurements," *Journal of Geophysical Research, Vol. 92*, No. B1, pp. 599-607, (1987).
3. E.J. Garboczi, "Mercury Porosimetry and Effective Networks for Permeability Calculations in Porous Materials," NIST.
4. Kelli Murbach, "Permeability in Cement Impedance Spectroscopy," Case Western Reserve University.
5. P.J. Tumidajski and B. Lin, "On the Validity of the Katz-Thompson Equation for Permeabilities in Concrete", pp. 643-647.
6. A.H. Thompson, A.J. Katz, and C.E. Krohn, "The microgeometry and transport properties of sedimentary rock," *Advances in Physics*, Vol. 36, No. 5, pp. 625-694 (1987).
7. A.H.Thompson, S.W. Sinton, S.L. Huff, A.J. Katz, R.A. Raschke, and G.A.Gist, "Deuterium magnetic resonance and permeability in porous media," *Journal of Applied Physics, Vol. 65*, pp. 3259-3263 (1989).

THEORY

In their 1986 paper, Katz and Thompson introduced a model for absolute permeability, the key relationship being

$$k = c\iota_c^2 \sigma / \sigma_o$$

where k is absolute permeability in terms of the rock conductivity σ and a characteristic length l_c. The constant c is of the order of (1/226 = 0.00442), and σ_o is the conductivity of the brine in the pore space. The characteristic length is determined experimentally from the threshold pressure in a mercury injection experiment. The equation follows from the percolation arguments of Ambegaokar, Halperin, and Langer (1971) and pertaining specifically to electron transport in amorphous semiconductors, but which are generally applicable to systems characterized by a broad distribution of conductances.

THE KATZ-THOMPSON METHOD OF DATA REDUCTION USING MERCURY POROSIMETRY

In order to calculate the permeability, the characteristic length, L_{char}, must be determined. This is determined from the threshold pressure, P_{thresh}, using the Washburn equation. The threshold pressure is the pressure at which the intrusion volume vs. pressure curve is steepest. This is either a calculated value (if chosen) or the value entered on the Material Properties window (see *"Fractal Dimensions" on page 18 - 8*). The specific volume intruded at pores larger than L_{char}, I_{thresh} , is also used. This is calculated by interpolating the specific intrusion volume vs. pore diameter curve at L_{char}.

If a conductivity formation factor (s/so), is entered, the permeability is calculated as:

Perm	=	$CL^2_{char}\sigma/\sigma_o$
C	=	user-entered permeability constant
σ/σ_o	=	user-entered conductivity formation factor

If the conductivity formation factor was not entered, calculations proceed using the length at which the conductance is maximum, L_{max}. The conductance is maximum when $(I-I_{thresh})D^3$ is maximum, where I is specific intrusion volume and D is diameter. To find the diameter at which this is the case, an Akima spline is set up for ($I_i > I_{thresh}$). The spline is then used to find the value of the diameter, L_{max} , at which this curve is maximized (not necessarily a node point). From this, the fractional volume of connected pore space involving pore widths of size L_{max} and larger, S_{Lmax}, can be calculated by interpolating the specific intrusion volume vs. pore size curve to L_{max} and dividing by the total specific intrusion volume I_{tot}.

With this in hand, the permeability and conductivity formation factor can be calculated as:

$$Perm = \frac{1}{89} L_{\max} \quad \frac{2L_{\max}}{L_{char}} \cdot I_{tot} \cdot Y_b \cdot S_{L\max}$$

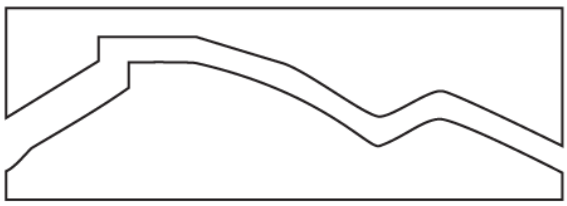
where

C	=	user-entered permeability constant
Y_b		bulk density, either calculated (if chosen) or user-entered on the *Material Properties* window

TORTUOSITY

The terms tortuosity and tortuosity factor are often used interchangeably. Tortuosity is the ratio of actual distance traveled between two points to the minimum distance between two points.

$$\zeta = tortuosity = \frac{\text{Actual distance traveled}}{\text{shortest distance}} = \frac{1_e}{I} \tag{1}$$



Required parameters (units specified as mass, volume, length, and area):

ρ	=	density (mass/volume) – from pycnometry
V_{tot}	=	total pore volume (volume/mass)
K	=	permeability (area)

DIFFERENTIAL INTRUSION FROM HG POROSIMETRY

The tortuosity can be calculated from the following expression:

$$\zeta = \sqrt{\frac{\rho}{24K(1+\rho V_{tot})}} \int_{\eta = rc,\min}^{\eta = rc,\max} \eta \int v(\eta) \tag{2}$$

where

$$-\int v(r_c) = \frac{dV(r_c)}{dr_c}, \text{ from MIP} \tag{3}$$

In order to calculate the tortuosity, the weighted average pore size, D_{avg}, must be found. This is accomplished as:

$$D_{avg}^2 = Y_s = \left[\frac{1}{2}1_1 O_i^2 + \Sigma I_i D_i^2 + \frac{1}{2} I_n O_n^2\right]$$

Y_s	=	skeletal or true density, either calculated (if chosen) or user-entered on the *Material Properties* window
D_i	=	pore diameter for the ith point
I_i	=	specific intrusion volume for the ith point

Given this value, the tortuosity is calculated as:

$$\xi = \sqrt{\frac{D_{avg}^2}{4 \cdot 24 Perm(1 - YI_{tot})}}$$

where

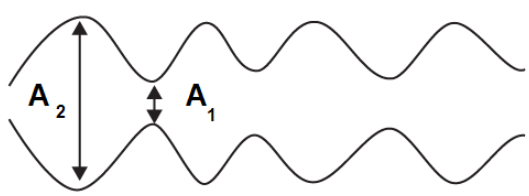
Perm	=	C L^2 char s/so
I_{tot}	=	total specific intrusion volume

CALCULATING TORTUOSITY FACTOR

Tortuosity factor is commonly used in the area of heterogeneous catalysis and is the ratio of tortuosity to constriction.

$$\tau = \frac{D_{eff}}{D\theta_c} \tag{4}$$

$$\tau = \frac{\xi}{\sigma} \tag{5}$$



$\beta = \frac{A_2}{B_2}$, area ratio

$\sigma = f(\beta)$, constriction factor

Carniglia has derived a simple expression for calculating the Tortuosity Factor of porous media. While this expression was derived using Fick's first law of diffusion and is convenient to calculate, the use of this correlation is severely limited by the data required to calculate the tortuosity factor.

V_{tot}	=	total pore volume
ρ_b	=	bulk density
S	=	total BET surface area
ΔV_i	=	change in pore volume within a pore size interval
d_i	=	average diameter within a pore size interval

For non-intersecting cylindrical pores the following simple correlation may be used:

$$\tau = 2.23 - 1.13 V_{totrb}$$

where

$$0.05 \leq V_{totpb} \leq 1.13 V_{totrb}$$

This correlation is limited to values of τ ranging from 1 to 2.2.

A generalized correlation has also been developed, however the generalized method requires diffusivity data for the system and conditions of interest (temperature and pressure). It is worth noting that if this diffusivity data is available, tortuosity factor can be calculated directly from equation 4.

$$\tau = \left(2.23 - 1.3 V_{rotpb}\right)\left(0.92\left(\frac{4}{S}\Sigma\frac{V_i}{D_i}\right)^{1+\epsilon}\right)$$

where

ε	=	pore shape exponent, Carniglia has assigned a value of 1 for cylinders.

The tortuosity factor is calculated as:

$$TF = 0.92\left[\left(\Sigma\frac{\iota_i}{D_i}\right)\frac{4}{S}\right]$$

ΔI_i	=	difference in specific intrusion volume for two adjacent points I_i - I_{i-1}
$\overline{D}$	=	average pore size for the interval between adjacent points 0.5 (D_i + D_{i-1})
S	=	user-entered BET surface area
I_i	=	specific intrusion volume for the ith point
D_i	=	pore diameter for the ith point
I_{tot}	=	total specific intrusion volume
Y_b	=	envelope density, either calculated (if chosen) or user-entered on the *Material Properties* window

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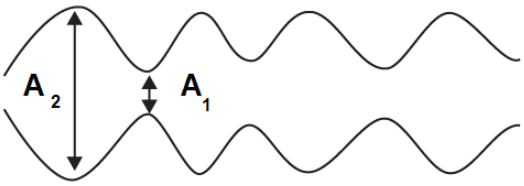

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19 Pore Surface Area Computation

It is sometimes asserted that pore wall surface area computed on the basis of the work required to immerse a surface in mercury is superior to assuming the pores are cylindrical and calculating area from geometric relationships. What those who make the assertion fail to recognize is that mathematically and in practice, the two computations are identical as shown below.

Work

The reversible work dW required to immerse an area dA of a non-wetting object in mercury[1] is

$$dW = \gamma \cos \Theta dA \tag{1}$$

where γ is the surface tension of mercury and θ its contact angle with the object. In the case of mercury and pores, this work is supplied when the external pressure P forces a volume of mercury dV into pores. Equation 1, therefore, becomes

$$\gamma \cos \Theta dA = -PdV \tag{2}$$

Assuming that γ and θ do not vary with pressure, equation 2 can be written

$$A = -\frac{\int PdV}{\gamma \cos \theta} \tag{3}$$

which, expressed for evaluation from pressure-volume mercury penetration data, becomes

$$\Sigma \quad A = -\frac{\Sigma P \quad V}{\gamma \cos \theta} \tag{4}$$

[1] Rootare, H.M. and Prenzlow, C.F., "Surface Areas from Mercury Porosimeter Measurements," J. Phys. Chem., 71, 2733-6 (1967).

CYLINDRICAL GEOMETRY

The basic relationship describing the penetration of mercury into a cylindrical pore of diameter D derived from equating the applied pressure to the resisting surface tension[1] is

$$PD = -4\gamma\cos\Theta \tag{5}$$

The relationship among wall area, diameter, and volume for a cylinder is

$$A = \frac{4V}{D} \tag{6}$$

Combining equations 5 and 6, yields

$$A = \frac{PV}{\gamma\cos\Theta} \tag{7}$$

which, as before, when written for evaluation from pressure-volume mercury penetration data, becomes

$$\Sigma \quad A = -\frac{\Sigma P \quad V}{\gamma\cos\theta} \tag{8}$$

[1] Washburn, E.W., "Note on a Method of Determining the Distribution of Pore Sizes in a Porous Material," Proc. Nat. Acad. Sci., 7, 115-6 (1921).

20 THEORY

Mercury porosimetry is based on the capillary law governing liquid penetration into small pores. This law, in the case of a non-wetting liquid like mercury and cylindrical pores, is expressed by the Washburn equation

$$D = -\left(\frac{1}{p}\right)4\gamma\cos\phi$$

where D is pore diameter, P the applied pressure, γ the surface tension, and ϕ the contact angle, all in consistent units. The volume of mercury V penetrating the pores is measured directly as a function of applied pressure. This P-V information serves as a unique characterization of pore structure.

Pores are rarely cylindrical, hence the above equation constitutes a special model. Such a model may not best represent pores in actual materials, but its use is generally accepted as the practical means for treating what, otherwise, would be a most complex problem.

The surface tension of mercury varies with purity; its usually accepted value and the value recommended here is 485 dynes/cm. The contact angle between mercury and the solid containing the pores varies somewhat with solid composition. A value of 130 degrees is recommended in the absence of specific information to the contrary.

Mercury extruding from pores upon reduction of pressure is in general accord with the above equation, but indicated pore diameters are always offset toward larger diameters. This results from equivalent volumes of mercury extruding at pressures lower than those at which the pores were intruded. It is also commonly observed that actual pores always trap mercury. The first phenomena is usually attributed to receding contact angles being less than advancing ones. The second is likely due to pore irregularities giving rise to enlarged chambers and “inkwell” structures. These phenomena give rise to hysteresis phenomena, i.e., distinct intrusion and extrusion P-V curves. See ***"Pore Surface Area Computation" on page 19 - 1*** for a discussion of surface area calculations.

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A ERROR MESSAGES

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

2401 | FATAL ERROR.

Cause: An internal processing and / or hardware error has occurred during communication with the analyzer.
Action: Contact your Micromeritics service representative.

2430 | Error accessing file [*n*], error code = [*n*].

Cause A: Media may be damaged.
Action A: Clean the media drive. If this does not eliminate the problem, attempt operation using a backup copy of the file.
Cause B: Hard disk may be damaged.
Cause B: Contact your Micromeritics service representative.
Cause C: A software error occurred when the file was accessed.
Cause C: Contact your Micromeritics service representative.
Cause D: The file name specified contains one or more invalid characters.
Cause D: Enter a valid file name. Do not use characters such as * or ?. Refer to the operating systems manual.

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

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

2432 | Invalid response from MMI 'FILE_READ' request.

Cause: An internal processing and/or hardware error has occurred.
Action: Contact a Micromeritics service representative if this error message continues.

2433 | New entries have been found in this directory. Refresh the directory information?

Cause: Several analyzer files (sample information, analysis conditions, adsorptive properties, or report options) have been added to this directory by some function other than the analyzer program.
Action: Click **Yes** to update the directory information with data from each new file. This operation may take a minute.

Click **No** to locate the file manually. This option may be feasible if a large number of files have been copied into the directory and the file name is known.

2434 | File [*n*] — Subset [*n*] wrote wrong [*n*] of data, expected [*n*] bytes.

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

2436 | Path specification [*n*] is invalid.

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

2437 | Overlay file [*n*] does not exist.

Cause: The entered file specification does not exist.
Action: Enter an existing file specification, or select a file name from the list box.

2439 | Could not register file.

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

2440 | Subset not found.

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

2441 | Seek within file failed.

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

2442 | Bad header in subset file.

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

2443 | Subset owner denied access.

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

2444 | Not a valid file format.

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

2445 | Subset wrote the wrong amount of data.

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

2446 | Error reading data.

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

2447 | Error writing data.

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

2448 | 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 | Deleted entries have been found in this directory. Refreshing the directory information.

Cause: Informational message only indicating the system is looking for directory entries that cannot be found.

Action: Wait a few moments for the system to finish refreshing, then retry the operation again.

2452 | Attempt to write MICATTR.DIR in read only mode. [*n*]

Cause: The *Read-Only* attribute is turned on in the application's MICATTR.DIR file (this file exists in each folder containing sample or parameter files).

Action: Use Windows Explorer to access the folder containing the MICATTR.DIR file, then disable the *Read-Only* option.

2453 | Attempt to append MICATTR.DIR in read only mode. [file name]

Cause: The *Read-Only* attribute is turned on in the application's MICATTR.DIR file (this file exists in each folder containing sample or parameter files).

Action: Use Windows Explorer to access the folder containing the MICATTR.DIR file, then disable the *Read-Only* option.

2454 | Too many selections for a print-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

Cause: Too many files were selected for this operation.

Action: Select only the number of files specified in the message.

2455 | Too many selections for an export-to-file operation. Only the first (number) selections will be processed. Please reselect the remainder.

Cause: Too many files were selected for this operation.

Action: Select only the number of files specified in the message.

2456 | Insufficient file handles available. Application cannot continue.

Cause: More than 50 files are open at the same time.

Action: Refer to an operating system manual then set the limit for open files to 50 or greater.

2457 | Results cannot be displayed. More than [*n*] windows are currently displaying or printing results.

Cause: Too many windows are open in the application.

Action: Close some of the open windows.

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.

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).

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.

2486 | Could not construct [*n*] report type. Program will terminate.

Cause A: Full rights to the application's folders and files is required.
Action A: Contact a system administrator to have full rights granted.
Cause B: An internal processing and / or hardware error has occurred.
Action B: Contact your Micromeritics service representative.

2487 | Could not start report generator. Error code [*n*]. Program will terminate.

Cause A: Full rights to the application's folders and files is required.
Action A: Contact a system administrator to have full rights granted.
Cause B: An internal processing and / or hardware error has occurred.
Action B: Contact your Micromeritics service representative.

2488 | File [*n*] cannot be opened for editing.

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

2489 | File [*n*] cannot be opened for writing.

Cause: The specified file in a *Save As* operation is already open for edit.
Action: Select a different file for the *Save As* operation.

2490 | No '.INI' file present. Application will terminate.

Cause: The ASCII .INI file containing initialization information and system options information used during program startup does not exist.
Action: Run the analyzer *Setup* program (located on the applications CD), select *Change analyzer setup* and enter the pertinent information.

2491 | Highlighted fields contain errors. Please correct the errors before closing.

Cause: The highlighted fields contain invalid entries. The window cannot be closed until

all errors are corrected.

Action: Check the entries, correct the errors, then close the window.

2492 | This field's entry is invalid.

Cause: The highlighted field contains an invalid entry.

Action: Check the entry and correct the error.

2493 | An entry is required for this field.

Cause: This field requires a valid entry to proceed.

Action: Enter or select an appropriate value.

2494 | Value is out of the valid range.

Cause: The entered value in the highlighted field is outside the valid range of values.

Action: Check the entry, then either enter or select an appropriate value.

2495 | Enter a value between [*n*] and [*n*].

Cause: The entered value in the highlighted field is outside the valid range of values.

Action: Check the entry, then either enter or select an appropriate value.

2496 | Invalid number.

Cause: An invalid number was entered in the highlighted field.

Action: Check the entry, then either enter or select a valid number.

2497 | This field contains an invalid character.

Cause: An invalid character was entered in the highlighted field.

Action: Check the entry, then enter valid characters.

2498 | The requested change to the Sample's status is invalid at this time.

Cause: A request to change the file's status, for example, from *automatically collected* to *manually entered* could not be done.

Action: Contact your Micromeritics service representative. Record the name of the sample file in which the problem occurred.

2499 | Sequence number must contain at least 3 digits.

Cause: An attempt was made to enter a sequence number that did not contain at least three digits.

Action: Enter a sequence number that contains at least three digits.

2500 | All sample file names that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause: No more sample information files can be created using the currently entered file name sequence number.
Action: Go to ***Options > Default Method***, then enter another sequence number.

2501 | System resources have reached a dangerously low level. Please close some windows to avoid the loss of data.

Cause: A large number of windows are open and consuming the system resources available to all applications.
Action: Close one or more windows. Contact your Micromeritics service representative.

2505 | Error logger cannot be initialized. Error code [*n*]. Program will exit.

Cause: An internal processing error has occurred.
Action: Contact your Micromeritics service representative.

2507 | The sample has an invalid status and cannot be used for degassing.

Cause: A sample file has been selected which does not have a *No Analysis* or *Prepared* status.
Action: Select a different file with a status of *No Analysis* or *Prepared*.

2508 | Overlay [*n*] was not found. It will not be included in the reports.

Cause: The specified overlay file could not be found.
Action: Ensure the file specified as an overlay exists.

2509 | Error opening file [*n*]. Reports cannot be produced.

Cause: An error occurred while the program was opening a file necessary to the report operation.
Action: Use the name given in the error message to investigate. Contact your Micromeritics service representative.

2510 | Error parsing reports from file [name]. Reports cannot be produced.

Cause A: One or more data entry fields in the sample file may contain an invalid character (such as a single quote or double quotes).
Action A: Review the data entry fields (for example, the *Sample* field), then remove the invalid character.
Cause B: The system was unable to create the usual temporary files during the report, possibly due to insufficient disk space.
Action B: Check the space available on the hard disk.
Cause C: An internal processing error occurred.
Action C: Contact your Micromeritics service representative.

2511 | Print job [*n*] has been canceled due to insufficient disk space. Delete unnecessary

files and restart the report.

Cause: The disk drive does not have required space for the temporary file.
Action: Delete unnecessary files from the disk. At least five megabytes of free space is required for normal operation.

2512 | Print job [*n*] canceled.

Cause: The print job was canceled by the operator.
Action: None required.

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.

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.

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.

2553 | Dialog ID [*n*] can not be created!

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

2556 | Directory database [*n*] error [*n*].

Cause: The sample file is currently selected and is undergoing a critical operation.
Action: Open the sample file after the critical operation has completed.

2557 | Cannot access web page.

Cause: The Micromeritics web page for DFT models cannot be accessed. This could be caused by an ISP problem of high internet traffic.
Action: Try the operation later.

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.

2560 | File [*n*] cannot be created. 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.

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.

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.

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.

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.

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.

2599 | The selected file has an extension that is not supported by this operation

Cause: The selected file does not have a supported file extension.

Action: Open the adsorptive properties file. Open the FPI file selector and select another file with a supported file extension.

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.

4021 | The entered [*n*] value ([*n*] *and Temperature Options of the Analysis Conditions*) is outside the range of the pressures listed in the Psat vs Temperature Table (*Adsorptive Properties*).

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

4022 | The entered bath temperature value ([*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*.

4026 | Cannot calculate Dubinin-Astkahov: bad least squares data.

Cause: Less than two selected data points are within the fitted pressure range.
Action: Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4027 | Fewer than two sample files have data suitable for heat of adsorption reports.

Cause: Less than two of the selected sample files for heat of adsorption reports contain appropriate data.

Action: Edit the *Quantity Adsorbed* table, or select other sample files.

4028 | Dubinin calculations cannot be performed because the affinity coefficient of the analysis gas is zero.

Cause: Dubinin values could not be calculated because the affinity coefficient of the analysis gas is zero.

Action: Access the *Dubinin Report Adsorptive* options in the sample file, then enter an appropriate value for the analysis gas.

4029 | At least two fitted data points are needed for Alpha-S calculations.

Cause: Fewer than two data points fall within the selected Alpha-s range.

Action: Edit either the calculation pressure in the fitted Alpha-s range, or use a different reference curve.

4030 | Preparations failed in primary data.

Cause: Appropriate data were not available to generate the report.

Action: This message was preceded by a different error message. Refer to the cause/action of the preceding message.

4031 | Not enough points with a relative pressure in the range [*n,n*].

Cause: Fewer than two data points selected for the Dubinin report fall within the selected relative pressure range.

Action: Edit the calculation pressure range or the fitted relative pressure range.

4033 | Not enough points to generate Dubinin Tabular Report.

Cause: There are fewer than two valid data points available for the Dubinin tabular reports.

Action: At least two micropore pressures must be selected for inclusion in the Dubinin report. Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4034 | Fewer than 2 points available for Dubinin calculations.

Cause: There are fewer than two valid data points available for Dubinin reports in one of the sample files selected for overlaying.

Action: At least two micropore pressures must be selected for inclusion in the Dubinin report. Edit the selection of data points on the Dubinin interactive editor or on the *Dubinin Pressures* window.

4035 | Cannot calculate optimized Astakhov exponent.

Cause: There are fewer than two valid data points in the relative pressure range specified. Astakhov reports will not be produced.

Action: At least two pressures must be selected for inclusion in the Astakhov report. Edit the selection of data points on the Astakhov interactive editor or on the *Astakhov Pressures* window.

4036 | Fewer than 2 points available for Horvath-Kawazoe calculations.

Cause: At least two data points must be selected for inclusion in the Horvath-Kawazoe analysis. No report will be produced.

Action: Edit the selection of points on the Horvath-Kawazoe interactive editor or on the *Horvath-Kawazoe* window.

4037 | Computations failed while processing the primary data set. No reports will be produced.

Cause: The preparation of data for reporting could not be successfully completed. No Horvath-Kawazoe reports will be produced. This message will always be preceded with another one containing additional information.

Action: Refer to the error message number which preceded this one for an explanation.

4038 | Fewer than 2 points available for the Langmuir Qm computation.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm must include at least two points above 0.02 relative pressure for the Langmuir equation to be applied.

Action: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the *Horvath-Kawazoe Report Options* window to prevent this message from appearing on future reports.

4039 | The isotherm does not meet the constraints of the Cheng/Yang assumption.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm does not correlate to the Langmuir assumption with a coefficient of 0.98 or more. The correction is not applicable to this isotherm or to the range of the data points selected.

Action A: The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the *Horvath-Kawazoe Report Options* window to prevent this message from appearing on future reports.

Action B: Generate the Langmuir report for the same data points selected for the Horvath-Kawazoe report. If the Langmuir correlation coefficient can be brought above 0.98 by removing some points at high relative pressure, remove them, then reproduce the Horvath-Kawazoe reports.

4040 | The value of Qm computed from the Langmuir equation is too low.

Cause: The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The computed value is less than the volume adsorbed at the largest relative pressure included in the analysis. The correction is not applicable to this isotherm or to the range of the data points selected.

Action: The analysis will be performed and the Cheng/Yang correction will be applied to all points with a volume adsorbed less than the value of Vm. The pore size will not be calculated for data points with an invalid volume adsorbed. Deselect Apply Cheng/Yang correction on the *Horvath-Kawazoe Report Options* window to clear this message.

4041 | Cheng/Yang correction is inappropriate for some [*n*] .

Cause: The Cheng/Yang correction is usually inappropriate for any P/P_o 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_o) in the analysis, the computed pore sizes for these pressures are usually meaningless.

Action: Change the data points selected for the Horvath-Kawazoe report to include only relative pressures at or below the knee of the isotherm, or change the Horvath-Kawazoe report options so that the Cheng/Yang correction is not applied.

4042 | 0.0 cannot be a starting or ending pressure for a geometric progression from low pressure.

Cause: An attempt was made to generate a pressure table from a geometrically progressing range.

Action: Change the 0.0 entered value.

4043 | 1.0 cannot be a starting or ending pressure for a geometric progression toward saturation.

Cause: An attempt was made to generate a pressure table from a geometrically progressing range.

Action: Change the 1.0 entered value.

4044 | Points in the Langmuir report pressure table lie outside the collected data.

Cause: Calculation pressure range is not being used. More than one of the report pressure table points is above the range of the collected data and more than one is below.

Action: Change the report pressure table to be more consistent with the collected data.

4045 | Points in the report pressure table lie outside the collected data.

Cause: Calculation range is not being used. More than one of the report pressure table points is above the range of the collected data and more than one is below.

Action: Change the report pressure table to be more consistent with the collected data.

4046 | [*n*] could not be opened for reading.

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

4047 | Warning: An error occurred while reading [*n*].

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

4048 | Warning: An error occurred while restoring the heat of adsorption report editor.

Cause: The state of the heat of adsorption report editor could not be restored. Default settings will be used.
Action: No action.

4049 | The sample [*n*] does not have enough data. A minimum of two adsorption points is required.

Cause: A sample file has been included in the Heat of Adsorption report that does not have enough data.
Action: Remove the file from the selected file list.

4050 | None of the requested quantities adsorbed is within the range of the collected data of more than one sample file.

Cause: The *Heat of Adsorption* report failed because the specified quantities are not within the range of the collected data.
Action: Edit the quantities adsorbed so that they are within the range of the collected data, or select other sample files.

4051 | The sample [*n*] does not have any data in the range of the requested quantities adsorbed.

Cause: The sample's data cannot be interpolated to any of the quantities adsorbed.
Action: Edit the quantities adsorbed so that they are within the range of the collected data.

4052 | Fewer than two points are selected for this report.

Cause: At least two points are required for the BET calculations.
Action: Edit the calculation range in the BET report.

4053 | At least two data points must be selected for t-Plot calculations.

Cause: At least two points are required for the t-Plot calculations.
Action: Edit the calculation range for the t-Plot report.

4054 | Fewer than two data points are inside the fitted thickness range.

Cause: At least two points must be within the fitted thickness range for the t-Plot calculations.
Action A: Edit the calculation range for the t-Plot report.
Action B: Edit the fitted thickness range in the t-Plot report editor.

4055 | A positive BET surface area was not calculated. Please check your BET Report.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.
Action A: Assign more points to the surface area calculation.
Action B: Select a different surface area in the t-Plot report editor.

4056 | A positive Langmuir surface area was not calculated. Please check your Langmuir report.

Cause: Fewer than two points were assigned to the requested surface area calculation in the collected data table.
Action A: Assign more points to the surface area calculation.
Action B: Select a different surface area in the t-Plot report editor.

4057 | At least two data points are needed for Freundlich calculations.

Cause: Less than two data points have been selected for the Freundlich report; at least two are required.
Action: Edit the selection of points on the Freundlich interactive editor or on the *Freundlich Pressures* window.

4058 | At least two data points are needed for Temkin calculations.

Cause: Less than two data points have been selected for the Temkin report; at least two are required.
Action: Edit the selection of points on the Temkin interactive editor or on the *Temkin Pressures* window.

4059 | Fewer than 2 points available for MP-Method calculations.

Cause: At least two points are required for the MP-Method calculations.
Action: Edit the calculation range for the MP-Method report.

4060 | Sample [*n*] contains no data points.

Cause: An attempt was made to save a sample without collected data as a t-curve or alpha-S curve.
Action: Repeat the *Save As t-curve* or *Save As alpha-S* operation after opening a sample that has collected data.

4061 | The t-curve must contain at least 2 points.

Cause: At least two points are required in a thickness curve definition.
Action: Edit the thickness curve.

4062 | Error during report preparation.

Cause: An internal processing error has occurred.
Action: Contact your Micromeritics service representative.

4063 | The data requested on this report are not available. No subreports selected.

Cause: There is no information in the sample log to report.
Action: A sample file was selected of which no instrument operations were used. Select a sample file with a status of *Prepared*, *Preparing*, *Analyzing*, or *Complete* to obtain a valid sample log report.

4067 | No data points are within the range of pressures in the reference isotherm.

Cause: There are no collected data points within the range of pressures in the reference isotherm.
Action: Select data points in the range of the reference isotherm, or select a more appropriate reference isotherm.

4068 | No points were selected for the f-Ratio report.

Cause: The f-Ratio report does not have any points selected.
Action: Edit the selection of data points on the *f-Ratio* window.

4070 | Unable to load deconvolution model [*n*].

Cause: The list of available models was corrupted; therefore, the model selected could not be loaded for the deconvolution.
Action: Exit the application. Reinstall the software, then try again.

4071 | The selected pressures points do not form a valid set for deconvolution.

Cause: The data points selected for analysis do not contain enough information to allow a DFT data reduction.
Action: At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction. Edit the selection of data points on the DFT interactive editor or on the *DFT Pressures* window.

4072 | The range of pressures selected is too small to deconvolute using this model.

Cause: A null result was found using the selected model.
Action: At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction. Edit the selection of data points on the DFT interactive editor or on the *DFT Pressures* window.

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.

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

Cause: The Langmuir report cannot be generated from the selected points.
Action: Edit the calculation pressure range in the Langmuir report pressure window.

4112 | Hard-sphere diameter, molecular weight, and mass flow constant have been updated from the fluid property information.

Cause: A new fluid property information file was loaded. The indicated fields have been updated with values from the file.
Action: This message is informational; no action is required.

4135 | HOA file [*n*] does not exist.

Cause: The sample file in the *Heat of Adsorption* report list does not exist.
Action: Go to ***Report > Heat of Adsorption***. Click **Add Samples**, then select the sample file.

4136 | HOA file [*n*] is corrupt.

Cause: The sample file in the *Heat of Adsorption* report list is corrupt.
Action: Go to ***Report > Heat of Adsorption***. Select the corrupt sample file, then click **Remove Sample**. Rerun the *Heat of Adsorption* report.

6000 | An error occurred while loading the application control information. Data entry cannot be performed. (Code [*n*]).

Cause: An error occurred accessing the control information disk file required by this application.
Action: The disk drive may have failed or be corrupt. Run diagnostics on the disk drive.

6002 | File cannot be opened for writing.

Cause: An attempt to save a file marked as "read-only" was made. Files can be marked automatically as read-only when they are transferred from a CD to the application directory.
Action: Use Windows Explorer to access the folder containing the file, then disable the Read-Only option.
Action: Enter a different name to save the file.

6008 | File cannot be opened for writing. It is already in use.

Cause: You attempted a *Save As* operation to a file which is already in use. The save could not be completed.
Action: Wait until the selected file is no longer being used or select a new target name for the *Save As* operation.

6008 | At least one sample must be selected to proceed.

Cause: An attempt was made to start an analysis without selecting any sample files.
Action: Select at least one file, then start the analysis.

6009 | 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.

6012 | Cannot read the analysis conditions parameter file.

Cause: The parameter file is either corrupt or has been deleted.
Action A: If this is a file created in a lab, recreate the file.
Action B: If this is a default file created during application installation, re-install the software.

6013 | Cannot read the adsorptive properties parameter file.

Cause: The parameter file is either corrupt or has been deleted.
Action A: If this is a file created in a lab, recreate the file.
Action B: If this is a default file created during application installation, re-install the software.

6014 | Cannot read the report options parameter file.

Cause: The parameter file is either corrupt or has been deleted.
Action A: If this is a file created in a lab, recreate the file.
Action B: If this is a default file created during application installation, re-install the software.

6015 | Cannot read the sample tube properties parameter file.

Cause: The file selected as a sample tube properties file is not valid.
Action: Select a different file.

6032 | Template file [*n*] for the selected analysis type does not exist. Select another analysis type.

Cause: A program piece required to run the PCP analysis is missing. Applies when in *Service Test Mode*.

Action: Re-install the software.

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