micromeritics®

MicroActive for TriStar® II Plus

Operator Manual

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Purchaser agrees to hold MICROMERITICS harmless from any patent infringement action brought against MICROMERITICS if, at the request of the purchaser, MICROMERITICS modifies a standard product or manufactures a special product to the purchaser's specifications.

MICROMERITICS shall not be liable for consequential or other type damages resulting from the use of any of its products other than the liability stated above. This warranty is in lieu of all other warranties, express or implied, including, but not limited to, the implied warranties of merchantability or fitness for use.

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1. USING THE ANALYZER

Introduction

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

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

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

Organization of the Operator's Manual

This operator's manual is organized as follows:

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

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

Conventions

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

Notes contain important information pertinent to the subject matter.

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

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

Equipment Description

The Micromeritics' TriStar II Plus is a fully automated, three-station, surface area and porosity analyzer capable of increasing the speed and efficiency of routine quality control analyses. It has a unique stainless steel manifold which is corrosive resistant and designed for highly accurate gas management. The analyzer allows up to three samples to be analyzed simultaneously. It also features a dedicated port for measuring the saturation pressure (Po) on a continuous basis. Surface areas as low as 0.01 m^2/g can be measured using nitrogen and as low as 0.001 m^2/g with the Krypton option. Up to 1000 data points can be collected allowing the observation of minute details of the isotherm.

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

The system includes Isothermal Jackets, which were originally patented by Micromeritics, for the sample tube. The Isothermal Jacket maintains a stable thermal profile along the full length of the sample and Psat tubes.

For ordering information on the Krypton, vacuum pump, or degasser options, refer to ORDERING INFORMATION, page 10-1.

Krypton Option

The Krypton option extends the measurable surface area to as low as 0.001 m^2/g using krypton as the adsorptive. The analyzer must be equipped with a 10 mmHg transducer and a high-vacuum pump to provide this capability.

Vacuum Pump

The analyzer requires a vacuum pump for sample analysis. Vacuum pumps used must meet the following criteria:

- achieve vacuum levels of 20 µmHg at the instrument inlet
- contain an anti-suckback valve to prevent vacuum pump oil from back streaming into the analyzer in the event of a vacuum pump failure
- contain an NW16 inlet port for connection to the analyzer.

A device to reduce oil vapor backstreaming is recommended.

The vacuum pump must have an anti-suckback valve to prevent oil from being admitted to the instrument should the power fail while the system is under vacuum. Pumps available from Micromeritics are equipped with an anti-suckback valve.

An oil-based or oil-free vacuum pump can be used with the analyzer (an oil-free pump is *required* if the Krypton option is installed). Appropriate vacuum pumps are available from Micromeritics. Refer to **ORDERING INFORMATION**, page **10-1**.

Degasser Options

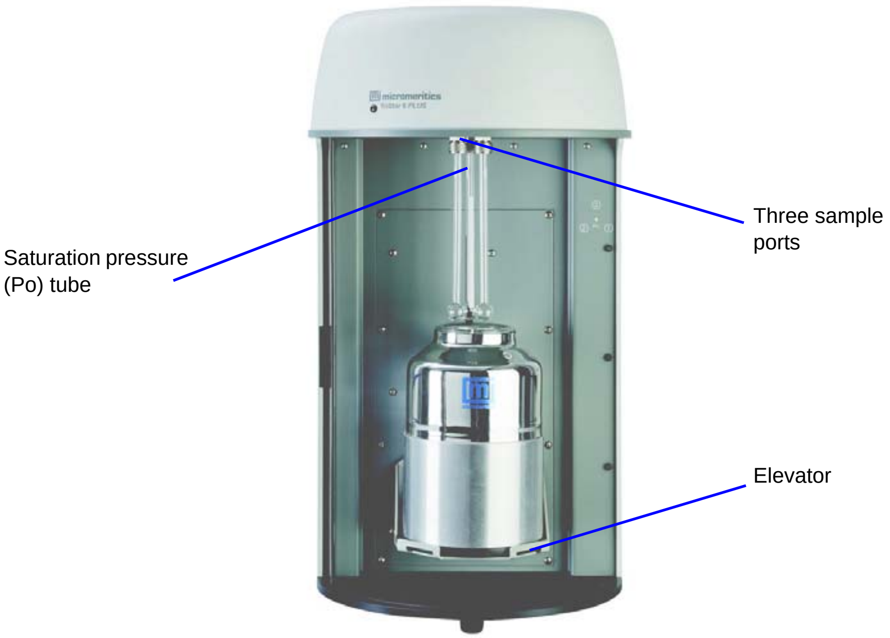
- **SmartPrep 065**

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

  The SmartPrep is the recommended degassing unit for the instrument.

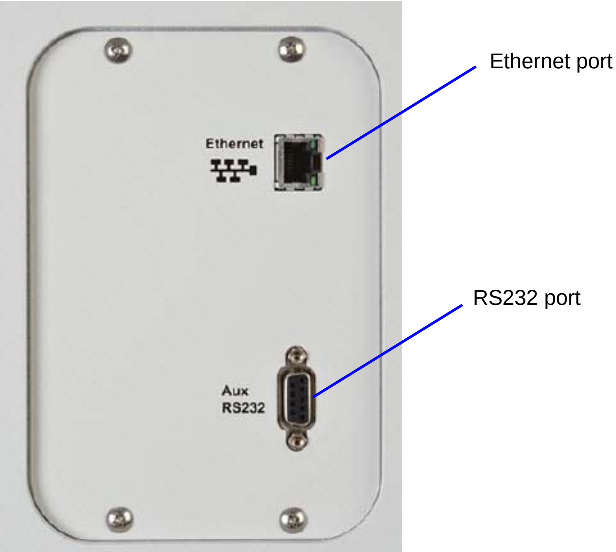
Instrument Components and Connectors

Sample Compartment



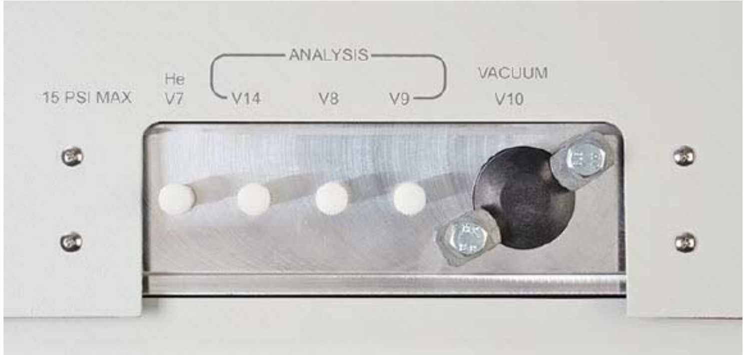
Component	Description
Sample ports	For installing up to three sample tubes.
Po tube	For measuring the saturation pressure.
Elevator	Allows placement of the dewar around the sample and Po tubes. The elevator is raised automatically when the analysis is started and lowers automatically upon completion.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

Side Panel



Connector	Description
Ethernet port	Port for an Ethernet cable allowing communication between the analyzer and the computer.
RS-232 port	Used to connect the SmartPrep.

Rear Panel



Component	Description
Gas inlet valves	Inlet valves for analysis gases.
Power cord connector	For setting the power voltage and connecting the analyzer to the power supply.

Component	Description *(continued)*
On/Off switch	For turning the analyzer on and off.

Turning the Analyzer On and Off

Turning the Analyzer On

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

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

Turning the Analyzer Off

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

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

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

   If an analysis is in progress, the following message is displayed:

   2459- An Instrument is busy. A delay in restarting this application could result in loss of new data. Continue program exit?

   Yes **No**

   Select **Yes** and the analysis program closes. The analysis continues and data continue to be collected. Queued reports under the printer will print. If a power failure occurs and an uninterruptible power supply (UPS) is not attached to the analyzer, the data collected *after exiting* the analysis program are lost.

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

2. Shut down the computer using standard Windows procedure.

3. Place the monitor and printer ON/OFF switches in the OFF position.
4. Place the analyzer ON/OFF switch in the OFF position.
5. Turn off the external vacuum pump.

Software

Some screen shots in this manual may differ slightly from those displayed on your screen.

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

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

- User-defined reports
- MicroActive reports

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

MicroActive Reports

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

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

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

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

Report Features

- After analysis, reports can be viewed, printed, and/or copied and pasted into other documents.
- The report zoom feature provides the viewing of fine graph details and the ability to shift the axes.
- Reports can be customized with a choice of fonts and a logo can be added to the report header.

Using the Software

The analysis program operates in the Windows environment and requires familiarity with standard Windows operations, for example, using the mouse, menus and windows. While this manual provides brief instructions for such standard operations, if necessary, refer to Windows documentation or its online help system to clarify any Windows functions. The application uses standard Windows features for file selection and supports Windows libraries for organizing and consolidating file locations. Refer to **Libraries**, page **1-14** for details on the File Selector.

Shortcut Menus

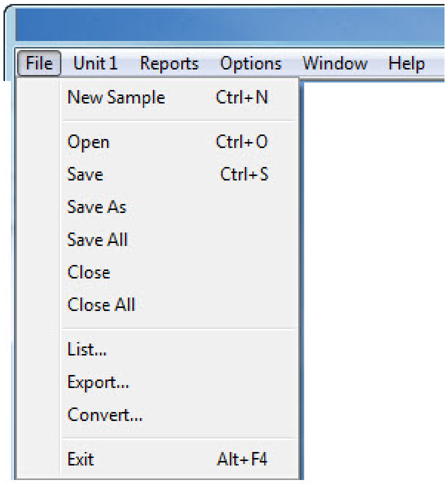
Shortcut menus are available for:

- The instrument schematic when Manual Control is enabled. Refer to **Enable Manual Control**, page **4-10**.

- On-screen graphs and tabular reports. Refer to **Report Shortcut Menus**, page **5-20**.

Shortcut Keys

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



Another shortcut method of accessing a menu or function is to use the **Alt** key plus the underlined letter in the menu command. For example, to access the **File** menu, press **Alt**+**F**. Then press the underlined letter on the submenu.

If the underscored letters do not display menus and windows, press the Alt key on the keyboard.

Shortcut Key(s)	Function
F1	Accesses the online operator’s manual.
F2	Displays the File Selector screen.
F3	When in the File Selector screen, displays the file search box.
F4	When in the File Selector screen, opens the address bar.
F6	Cascades open windows.
F7	Tiles open windows.
F8	Opens the File Selector to start a report from a selected .SMP file.
F9	Closes all open reports.

Shortcut Key(s)	Function *(continued)*
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Shift + F9	Accesses shortcut menu of (1) selected component on instrument schematic, when manual control is enabled or (2) onscreen reports.

Files

File Status and Description

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

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

File Name Conventions

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

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

File Type	Default File Name Extension
Alpha-s curve	.ALS
Adsorptive properties	.ADP
Analysis conditions	.ANC
Degas conditions	.DEG
Heat of Adsorption Report	.HOA
Methods	.MTH
Report options	.RPO
Sample information	.SMP
Sample tube properties	.STB
Thickness curve	.THK

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

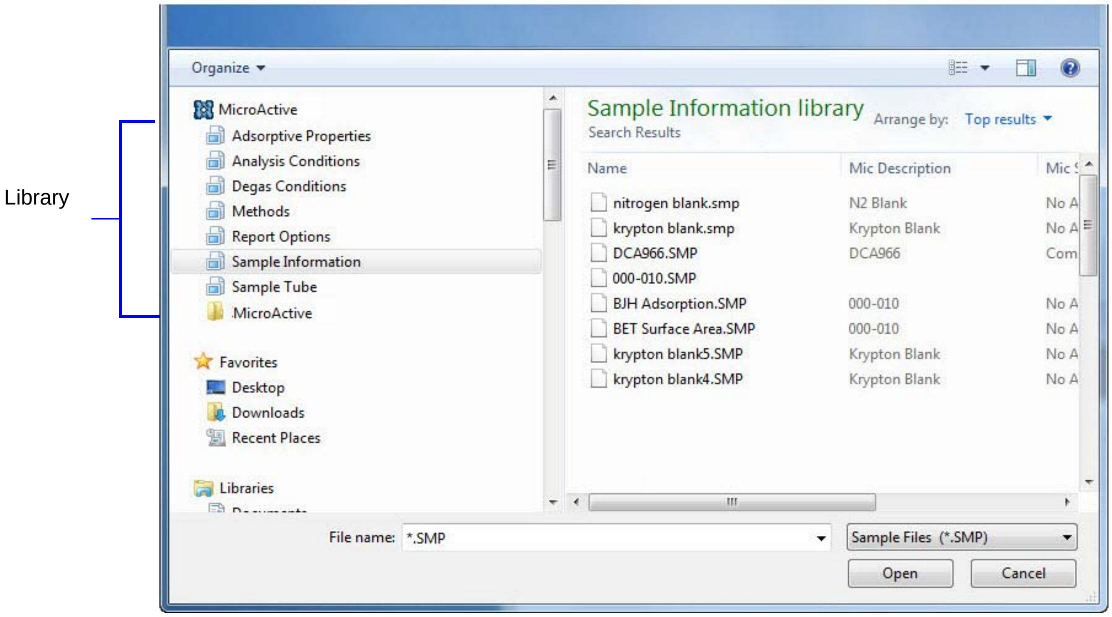
Report	.REP
Spreadsheet	.XLS
ASCII	.TXT

Libraries

Libraries provide a means for organizing files by acting as user-defined virtual folders. In a library, folders and files from different folders on the same or different drives are consolidated to appear in a single location. The folders and files in a library may even be on different computers.

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

The application is installed with libraries containing the default locations for Micromeritics application specific files. Refer to **Manage Libraries**, page **6-3** for information on adding other locations to your libraries.



Name	Mic Description	Mic S
nitrogen blank.smp	N2 Blank	No A
krypton blank.smp	Krypton Blank	No A
DCA966.SMP	DCA966	Com
000-010.SMP		
BJH Adsorption.SMP	000-010	No A
BET Surface Area.SMP	000-010	No A
krypton blank5.SMP	Krypton Blank	No A
krypton blank4.SMP	Krypton Blank	No A

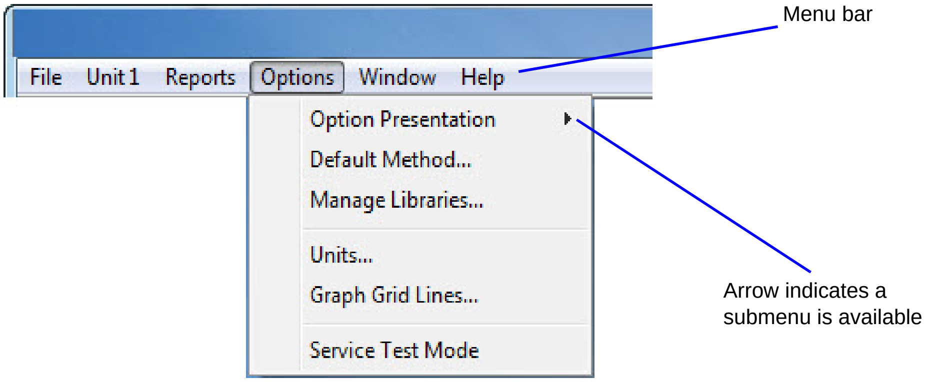
File name: *.SMP

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

Library Name	Default Directory *(continued)*	File extension
Sample Tube	...\Param	.STB

Menu Structure

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



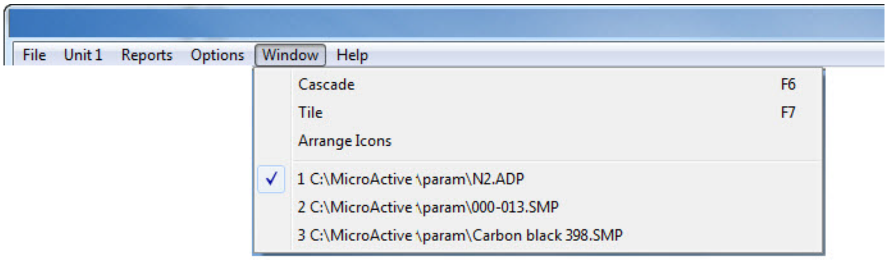
Main Menu Bar

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

Menu Item	Description
File	Use to manage files. Refer to **FILE MENU**, page **3-1**.
Unit [*n*]	Use to perform analyses, calibrations and other instrument operations. Refer to **UNIT MENU**, page **4-1**.
Reports	Use to run reports and view the results. Refer to **REPORTS MENU**, page **5-1**.
Options	Use to edit default method, specify system configuration and data presentation formats. Refer to **OPTIONS MENU**, page **6-1**.
Window	Use to arrange open windows and display a list of open windows. Refer to **Window Menu**, page **1-16**.
Help	Use to access this operator’s manual, Micromeritics web page, and tutorials. Refer to **Help Menu**, page **1-17**.

Window Menu

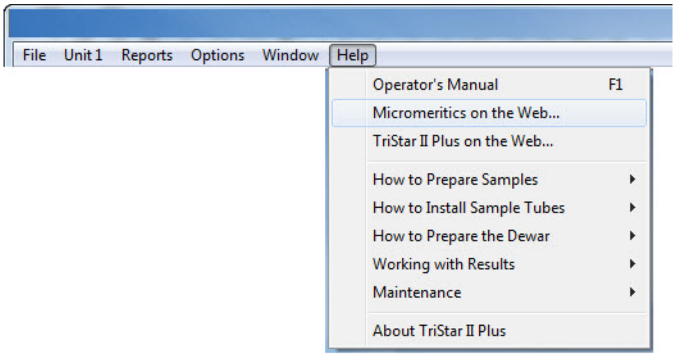
The **Window** menu lists open files and provides the ability to rearrange the way open windows display. A check mark appears to the left of the active window.



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

Help Menu

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



Help Menu Option	Description
Operator’s Manual	Provides access to the online operator’s manual.
Micromeritics on the Web	Provides a link to the Micromeritics web page: **www.Micromeritics.com**
TriStar II Plus on the Web	Provides an Internet link to additional analyzer information: **TriStar II Plus on the Web**
Available Videos	**How to Prepare Samples** • Selecting the Amount of Sample to Use for Analysis • Choosing Sample Tube Sets • Cleaning Sample Tubes • Measuring Samples Mass **How to Install Sample Tubes** **How to Prepare the Dewar** **Working with Results** • Working with Interactive Reports • Evaluating Data **Maintenance** • Connecting Gases • Cleaning and Verifying Gas Lines • Specifying Gas Ports • Maintaining the Vacuum Pump
About TriStar II Plus	Provides the analysis program version number.

Setup Program

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

- Reinstall the application
- Add an analyzer
- Move an analyzer
- Remove an analyzer
- Change analyzer setup
- Reinstall calibration files for an analyzer
- Import an analyzer from a previous installation on this PC

The IP address of the Ethernet interface on the computer connected to the instrument must not change after it has been selected in the analyzer setup program. Connectivity with the instrument may be disrupted if the analyzer program is not correctly configured with the IP address of the computer Ethernet interface. Check with your system administrator on how to change the IP address for your computer."

2. OPERATIONAL PROCEDURES

Introduction

This chapter contains information on:

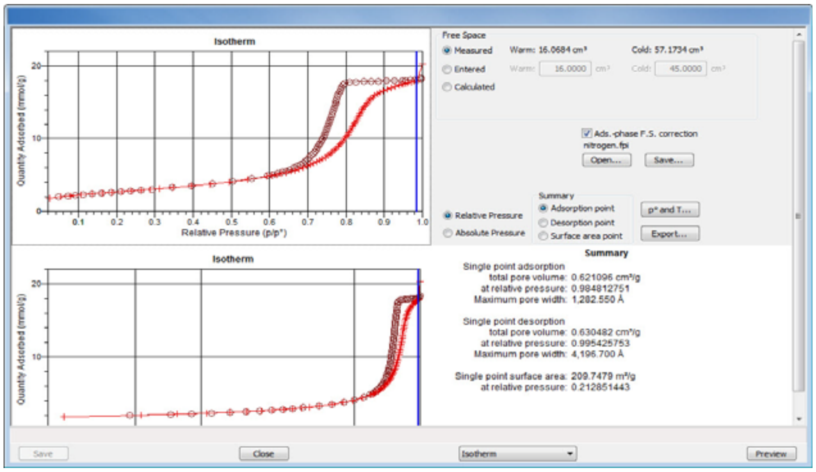
- working with Interactive Reports
- working with NLDFT Advanced PSD Reports
- specifying method defaults
- creating sample files in advanced, basic, and restricted formats
- defining parameter files
- manually entering isotherm data
- preparing samples and performing an analysis
- generating reports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

Working with Interactive Reports

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

- click the following link: Working with Interactive Reports
- go to ***Help*** > ***Working with Results*** > ***Working with Interactive Reports***

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



Free Space
● Measured Warm: 16.0684 cm³ Cold: 57.1734 cm³
○ Entered Warm: 16.0000 cm³ Cold: 45.0000 cm³
○ Calculated

☑ Ads.-phase F.S. correction
nitrogen.fpi

● Relative Pressure
○ Absolute Pressure

Summary
● Adsorption point
○ Desorption point
○ Surface area point

Summary

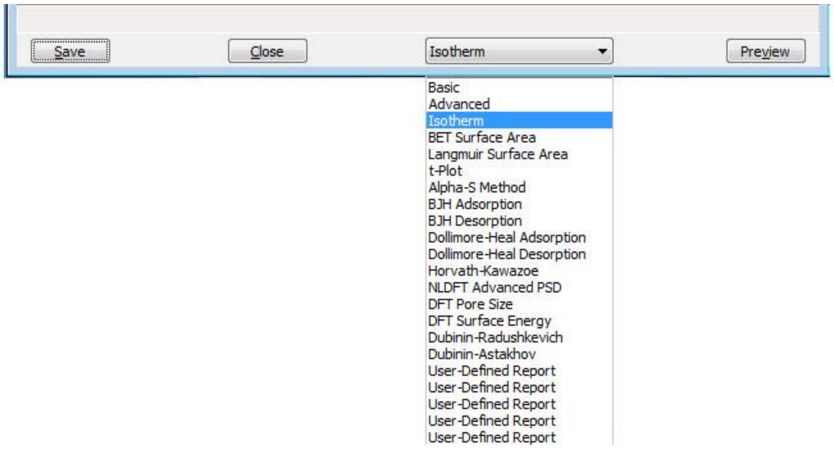
Single point adsorption
total pore volume: 0.621096 cm³/g
at relative pressure: 0.984812751
Maximum pore width: 1,282.550 Å

Single point desorption
total pore volume: 0.630482 cm³/g
at relative pressure: 0.995425753
Maximum pore width: 4,196.700 Å

Single point surface area: 209.7479 m²/g
at relative pressure: 0.212851443

2. To view the plots in either **Relative** or **Absolute** pressure, click the **Relative Pressure** or **Absolute Pressure** option.

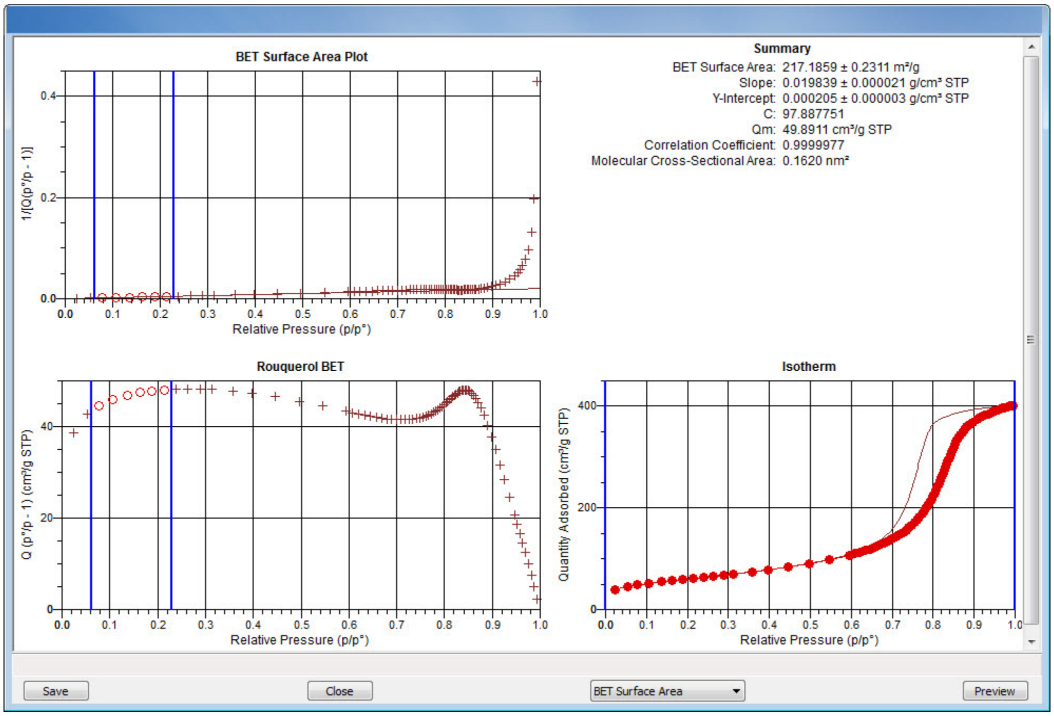
3. To view another report, click the **Isotherm** drop-down arrow and make another selection.



The choices in this list allow you to:

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

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



Summary

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

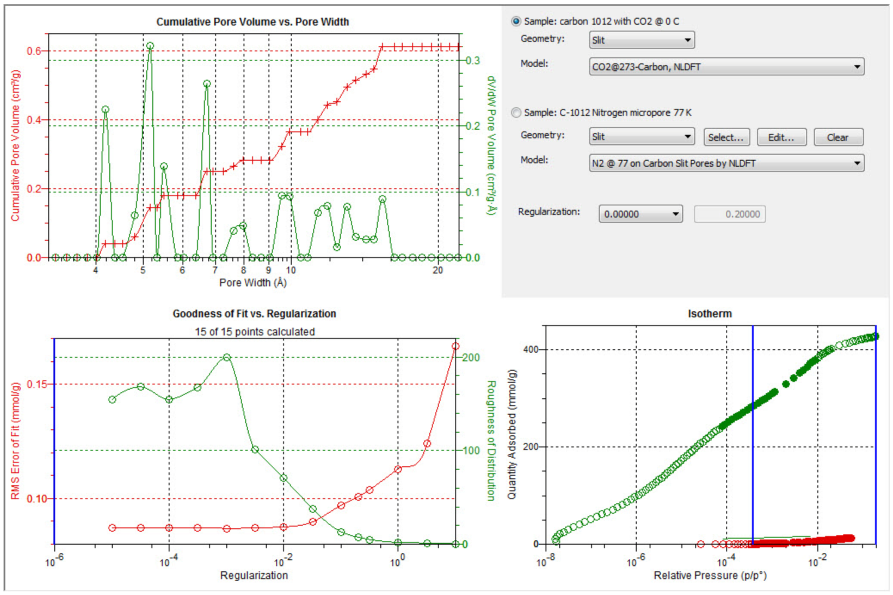
When ranges are edited, the changes are reflected immediately in the plots and the summary data displayed in the window. Some editing options are:

- Drag the blue bars to increase or decrease the range of data included in the plot.
- Press **CTRL**, then left-click the mouse on a data point in the Isotherm Linear Plot to include or omit the data point from the BET plot.
- Right-click the mouse to display a popup menu to include reports, enable or select overlays, edit curves, axes, legends, titles, and copy and paste the data in a graph or in tabular format.

4. After editing the report, save the changes in the sample information file by clicking **Save**.

Working with NLDFT Advanced PSD Report Option

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 dropdown list.



● Sample: carbon 1012 with CO2 @ 0 C
Geometry: Slit
Model: CO2@273-Carbon, NLDFT

○ Sample: C-1012 Nitrogen micropore 77 K
Geometry: Slit
Model: N2 @ 77 on Carbon Slit Pores by NLDFT

Regularization: 0.00000 0.20000

The model curve fit is shown in the lower right quadrant along with the adsorption isotherm. This curve fit is updated every 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 except using a different analysis gas and temperature.

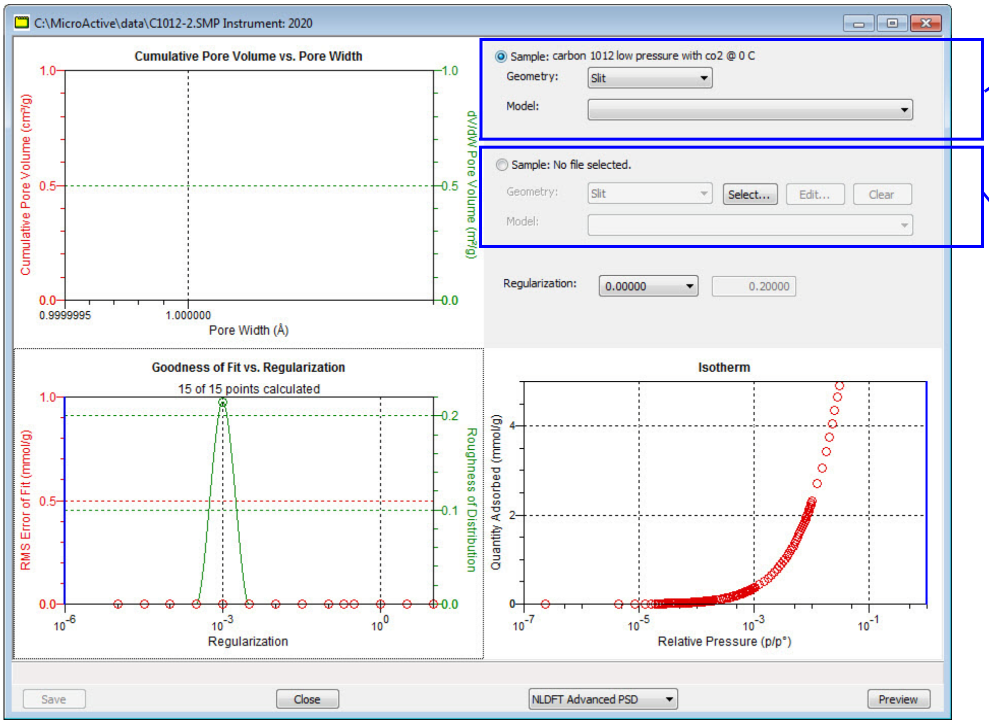
The isotherm for this second sample will in general be different than the first sample. The Advanced DFT calculation takes the data from both sample files and combines all this data into a more accurate calculation of the pore size distribution. More accurate means getting the pore distribution at smaller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

* In order to make a successful "advanced" calculation, the user must select a second sample file using the select button and also select a model for this second sample file. The user can use the radio buttons next to the two sample file names to select the isotherm data points for each sample (after changing the radio selection, 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 the isotherm plot (lower right) as well as a second curve-fit. As you adjust the selection of points, then 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 and click **Open**.

2. In the dropdown list at the bottom of the screen, 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 screen.


C:\MicroActive\data\C1012-2.SMP Instrument: 2020

● Sample: carbon 1012 low pressure with co2 @ 0 C
Geometry: Slit
Model:

○ Sample: No file selected.
Geometry: Slit
Model:

Regularization: 0.00000 0.20000

NLDFT Advanced PSD

First selected sample file

Second selected sample file

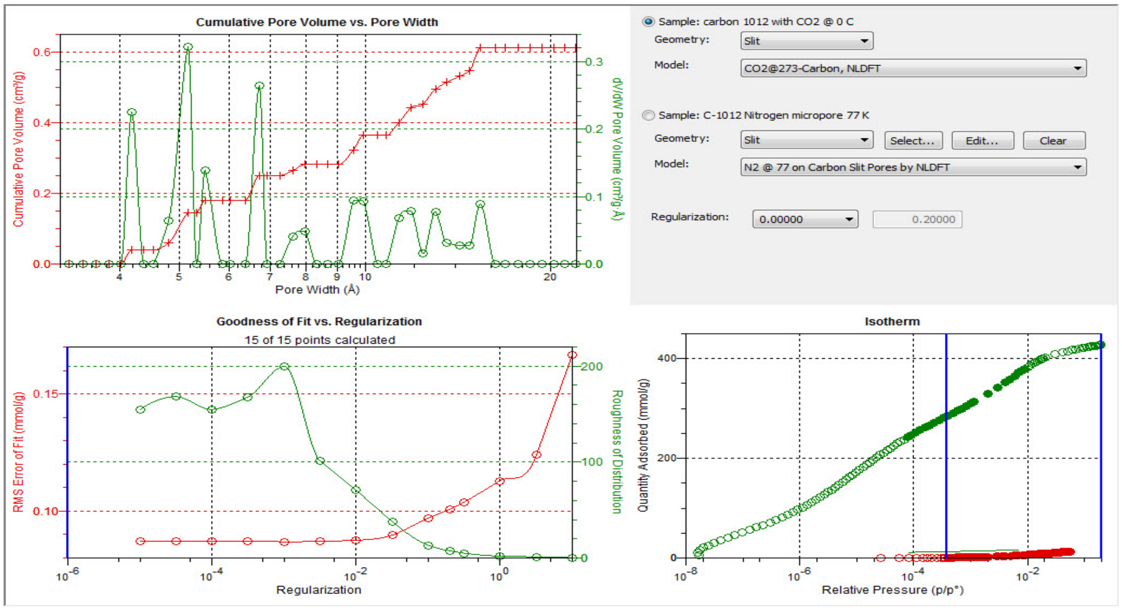
a.) Select the **Geometry** and **Model** from the dropdown lists for the first sample file.

b.) To select isotherm data points for calculation for the first sample file, ensure the radio button 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 screen.

a.) Select the **Geometry** and **Model** from the dropdown lists for the second sample file.

b.) To select isotherm data points for calculation for the second sample file, ensure the radio button 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.



● Sample: carbon 1012 with CO2 @ 0 C
Geometry: Slit
Model: CO2@273-Carbon, NLDFT

○ Sample: C-1012 Nitrogen micropore 77 K
Geometry: Slit
Model: N2 @ 77 on Carbon Slit Pores by NLDFT

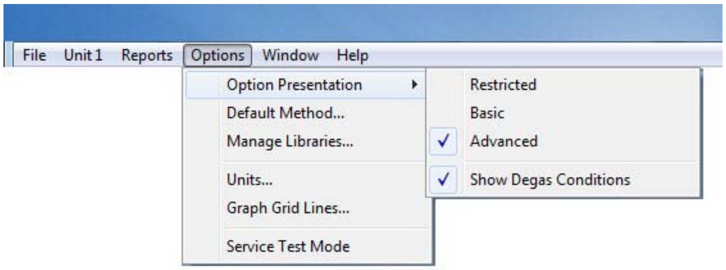
Regularization: 0.00000 0.20000

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

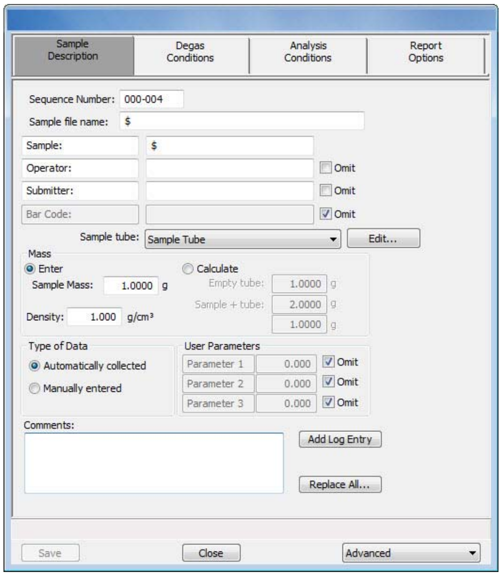
Editing the Default Method

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

1. Go to ***Options*** > ***Option Presentation*** >**Advanced**. Ensure a checkmark appears to the left of **Advanced**.



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



Sequence Number: 000-004
Sample file name: $
Sample: $
Operator: ☐ Omit
Submitter: ☐ Omit
Bar Code: ☑ Omit
Sample tube: Sample Tube
Mass
● Enter
Sample Mass: 1.0000 g
Density: 1.000 g/cm³
○ Calculate
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Type of Data
● Automatically collected
○ Manually entered
User Parameters
Parameter 1 0.000 ☑ Omit
Parameter 2 0.000 ☑ Omit
Parameter 3 0.000 ☑ Omit
Comments:
Advanced

3. In the **Sequence Number** text box, specify an optional default alphanumeric file sequence string. This field must contain a minimum of 3 numbers. As files are created, this number is incrementally sequenced as a part of the file name and will also display in the **Sample** text box when a sample file is created.

4. In the **Sample** text box, enter an optional default file name. This information will be appended to the sequence number as a part of the file name and will also display in the **Sample** text box when a sample file is created. The **$** character must remain in this field.

5. In the text box to the right of the **Sample** field, enter a format for the default sample identification. Include the **$** character to automatically include the contents of the **Sequence Number** field and **Sample file name** field as part of the sample identification.

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

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

7. To specify default sample tube parameters click **Edit** to the right of the **Sample tube** dropdown list. Refer to **Sample Tube**, page **2-17** for instructions on editing and saving the sample tube parameters to make them available in the **Sample tube** dropdown list.
8. In the **Mass** group box, indicate if mass is to be manually entered by the operator (**Enter**) or calculated by the system (**Calculate**). Refer to **Mass group box**, page **3-12**.
9. In the **Type of Data** group box, indicate if the data is to be automatically collected by the system or manually entered by the operator.
10. The optional user-defined fields in the **User Parameters** group box may be used to enter and track information from another instrument or source, along with other statistical process control (SPC) data. Refer to **Sample Information Files**, page **3-9**.

The labels for the User Parameter **fields can be modified by overwriting the default labels. These fields can also be omitted from a sample file by selecting the** Omit **checkbox.**

11. Use the **Comments** text box to enter notes about the **Method**.
12. After completing the **Sample Description** window, select the parameter tabs to edit other sample information file parameters. The saved parameter defaults become the defaults for new sample files. Refer to **Defining Parameter Files**, page **2-17**.
13. Click **Save**, then click **Close**.

Defining Sample Information Files

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

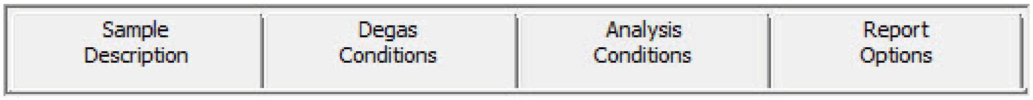
A sample information file may be created in Advanced, Basic, or Restricted presentation format.

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

Creating Sample Files in Advanced Format

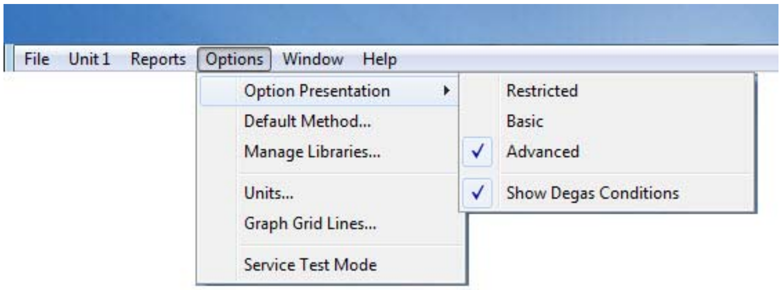
The values specified in the parameter portions of the default sample file (Degas Conditions, Analysis Conditions, and Report Options) are saved as the defaults for new sample files. To navigate from one set of parameters to another, select the parameter tab at the top of the window.

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

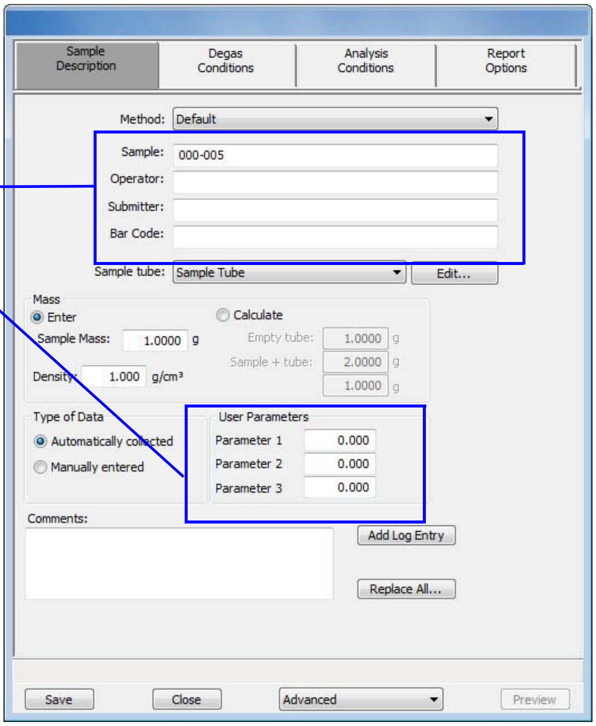


The Degas Conditions tab displays only if enabled in *Options > Option Presentation > Show Degas Conditions***.**

1. Go to ***Options*** > ***Option presentation*** > ***Advanced***. Ensure a checkmark appears to the left of **Advanced**.



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



Method: Default

Sample: 000-005

Operator:

Submitter:

Bar Code:

Sample tube: Sample Tube

Mass

● Enter

○ Calculate

Sample Mass: 1.0000 g

Empty tube: 1.0000 g

Sample + tube: 2.0000 g

1.0000 g

Density: 1.000 g/cm³

Type of Data

● Automatically collected

○ Manually entered

User Parameters

Parameter 1 0.000

Parameter 2 0.000

Parameter 3 0.000

Comments:

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

3. Select a **Method** from the **Method** dropdown list.

4. Enter a sample description in the **Sample** text box.

5. Enter **Operator**, **Submitter**, and **Bar Code** identification information in the respective text boxes. Some text box fields may have been renamed or may not display if modified in ***Options*** > ***Default Method***.

6. In the **Sample Tube** dropdown list, select a sample tube from the list or click **Edit** to specify a sample tube description, empty tube properties for calculated free space, isothermal jacket, filler rod, and vacuum seal type. The text entered into the **Description** field will display in the **Sample Tube** dropdown list of the previous window. Click **OK** to return to the previous window. Refer to **Sample Tube**, page **2-17**.

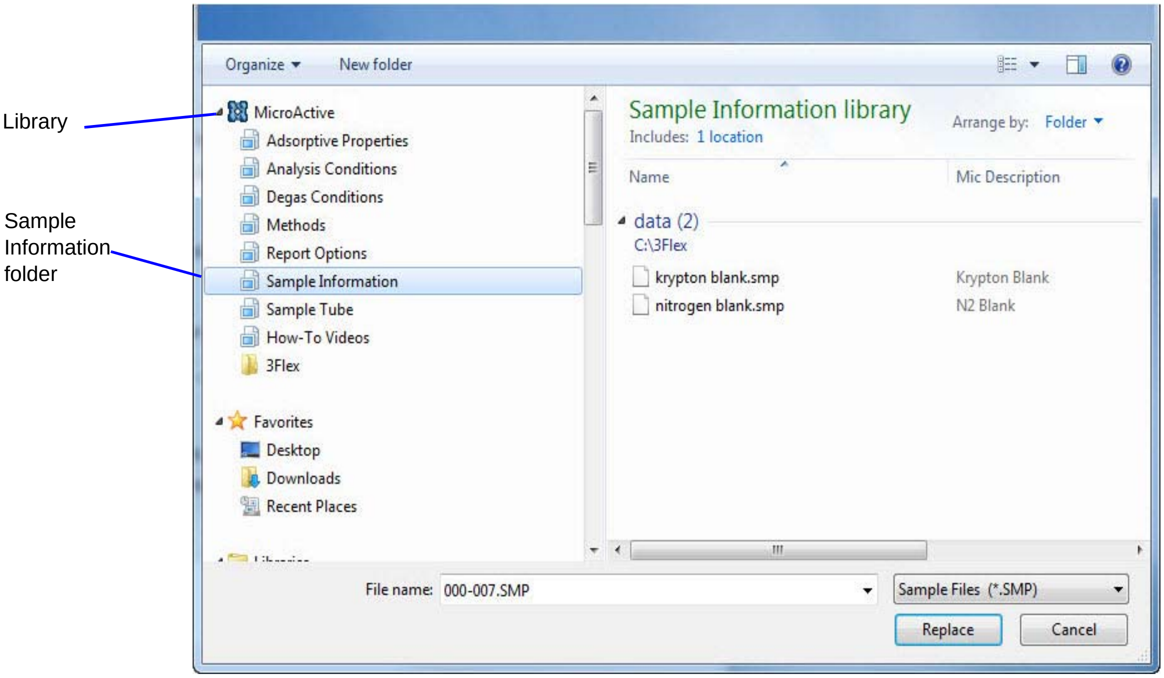
7. In the **Mass** group box, indicate if mass is to be manually entered by the operator (**Enter**) or calculated by the system (**Calculate**). Refer to **Mass group box**, page **3-12**.

8. In the **Type of Data** group box, indicate if the data is to be automatically collected by the system or manually entered by the operator. Select A**utomatically collected** for all sample runs where the data are collected. Select **Manually entered** when another sample has been run on a different instrument or different model instrument so that data can be analyzed or used for comparison. If **Manually entered** is selected, the data are entered in the isotherm interactive report. Refer to **Manually Entering Isotherm Data in a Sample File**, page **2-27**.

9. If SPC (Statistical Process Control) information is to be reported, enter appropriate information in the **User Parameters** group box. These are user-definable parameters that can be entered and

tracked along with other statistical process control data. Some text box fields may have been renamed or may not display if modified in ***Options*** > ***Default Method***.

10. Enter any pertinent information about the sample information file in the **Comments** text box. Comments will display in the report header.
11. Click the **Add Log Entry** button to enter notes for the instrument log report. Create entries that cannot be recorded automatically through the application software. For example, record that the port filter was changed.
12. To auto-populate fields from another .SMP file, click the **Replace All** button and select a .SMP file that contains the desired parameters. Select the file and click **Replace**.
13. After completing the **Sample Description** window, click the parameter tabs to edit other sample information file parameters. Refer to **Defining Parameter Files**, page **2-17**.
14. Click **Save**, then click **Close** to save the file with the default file name. To save as a different file name, go to ***File*** > ***Save As*** and enter a new file name. The file can later be retrieved from the Sample Information folder in the library.

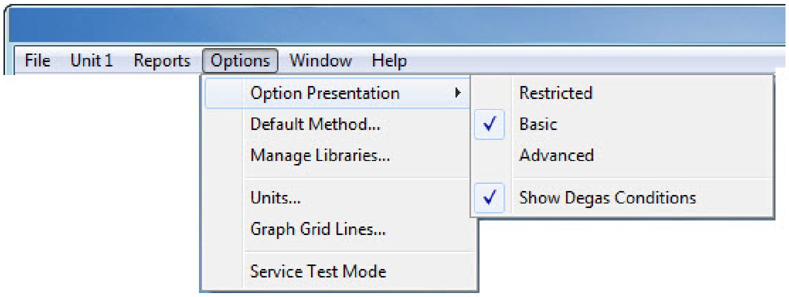


Creating Sample Files in Basic or Restricted Format

The Basic and Restricted formats use predefined parameter files to create a sample information file. Refer to **Sample Information Files**, page **3-9**.

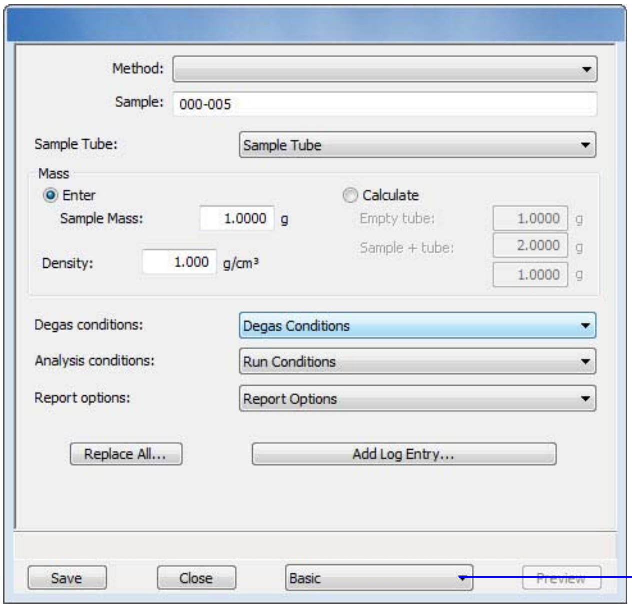
- When using the Basic presentation format, switch to the Advanced presentation format to edit parameter file values.
- When using the Restricted presentation format, parameter files cannot be edited.

1. Go to ***Options*** > ***Option presentation*** > ***Basic***. Ensure a checkmark appears to the left of **Basic**.



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

3. Select a Method from the **Method** dropdown list. Refer to **Editing the Default Method**, page **2-7**.



Method:
Sample: 000-005
Sample Tube: Sample Tube
Mass
● Enter
Sample Mass: 1.0000 g
○ Calculate
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Density: 1.000 g/cm³
Degas conditions: Degas Conditions
Analysis conditions: Run Conditions
Report options: Report Options

The Restricted presentation format does not show an Advanced option.

4. In the **Sample** field, enter a sample description.

5. Select a sample tube from the **Sample Tube** dropdown list.

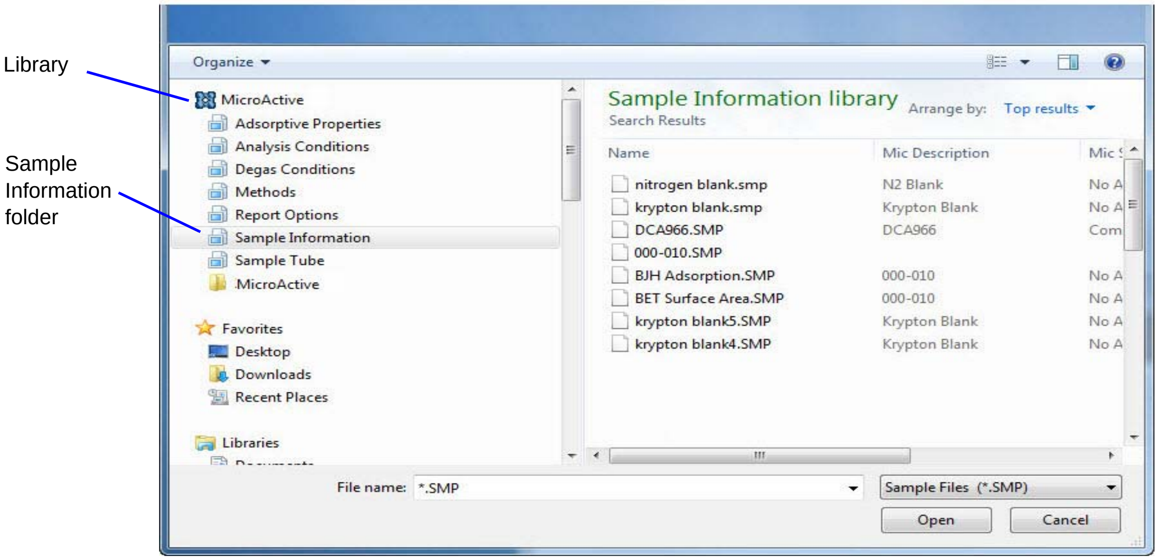
6. In the **Mass** group box, indicate if mass is to be manually entered by the operator (**Enter**) or calculated by the system (**Calculate**). Refer to **Mass group box**, page **4-10**.

7. Click the dropdown arrows to select default parameter files for **Degas conditions**, **Analysis conditions**, and **Report options**.

8. To auto-populate fields from another .SMP file, click the **Replace All** button and select a .SMP file that contains the desired parameters. Select the file and click **Replace**.

9. Click the **Add Log Entry** button to enter notes for the instrument log report. Create entries that cannot be recorded automatically through the application software. For example, record that the port filter was changed.

10. Click **Save**, then click **Close**. The file can be retrieved later from the Sample Information folder in the library.

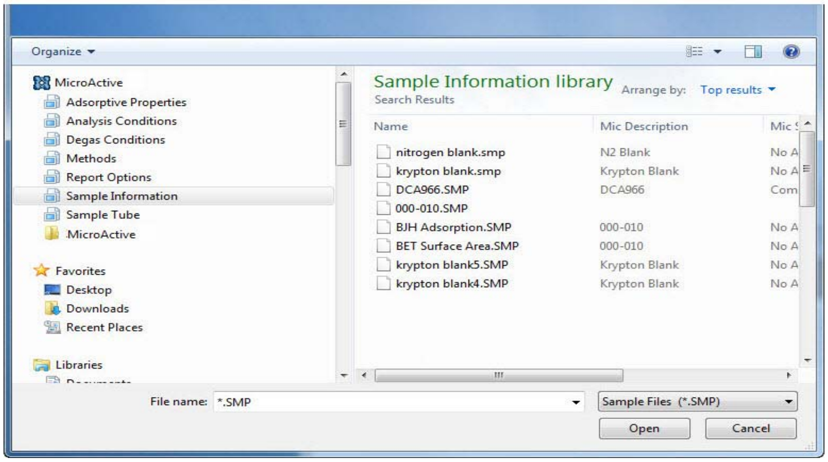


Sample Information library
Search Results

Name	Mic Description	Mic S
nitrogen blank.smp	N2 Blank	No A
krypton blank.smp	Krypton Blank	No A
DCA966.SMP	DCA966	Com
000-010.SMP		
BJH Adsorption.SMP	000-010	No A
BET Surface Area.SMP	000-010	No A
krypton blank5.SMP	Krypton Blank	No A
krypton blank4.SMP	Krypton Blank	No A

File name: *.SMP
Sample Files (*.SMP)

Opening Sample Information Files



Name	Mic Description	Mic S
nitrogen blank.smp	N2 Blank	No A
krypton blank.smp	Krypton Blank	No A
DCA966.SMP	DCA966	Com
000-010.SMP		
BJH Adsorption.SMP	000-010	No A
BET Surface Area.SMP	000-010	No A
krypton blank5.SMP	Krypton Blank	No A
krypton blank4.SMP	Krypton Blank	No A

File name: *.SMP

Sample Files (*.SMP)

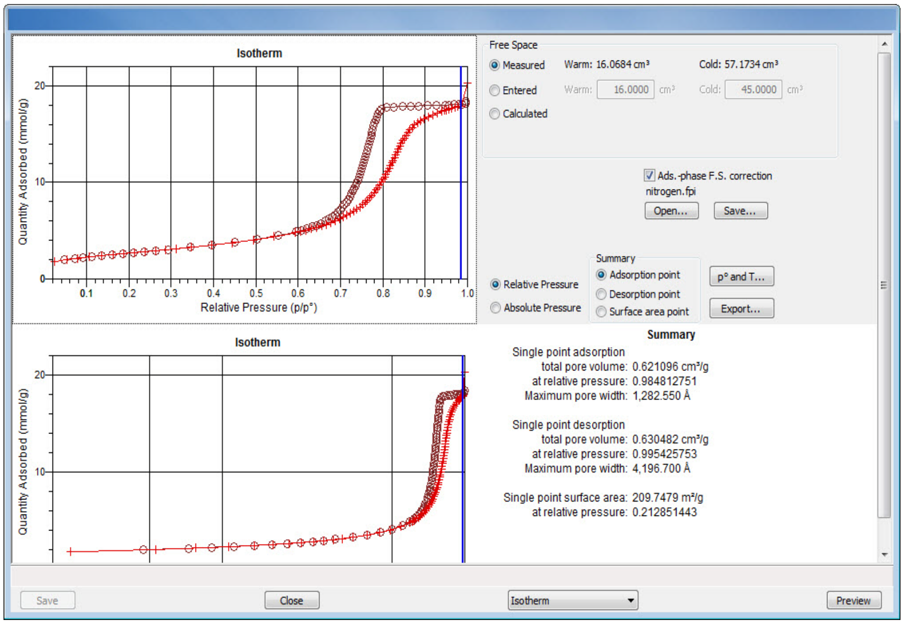
When working with an existing file, it is recommended that a copy of the file be used rather than the original.

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

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

1. Go to ***File*** > ***Open*** or use the **F2** keyboard shortcut.

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



Free Space
● Measured Warm: 16.0684 cm³ Cold: 57.1734 cm³
○ Entered Warm: 16.0000 cm³ Cold: 45.0000 cm³
○ Calculated

☑ Ads.-phase F.S. correction
nitrogen.fpi

● Relative Pressure
○ Absolute Pressure

Summary
● Adsorption point
○ Desorption point
○ Surface area point

Summary

Single point adsorption
total pore volume: 0.621096 cm³/g
at relative pressure: 0.984812751
Maximum pore width: 1,282.550 Å

Single point desorption
total pore volume: 0.630482 cm³/g
at relative pressure: 0.995425753
Maximum pore width: 4,196.700 Å

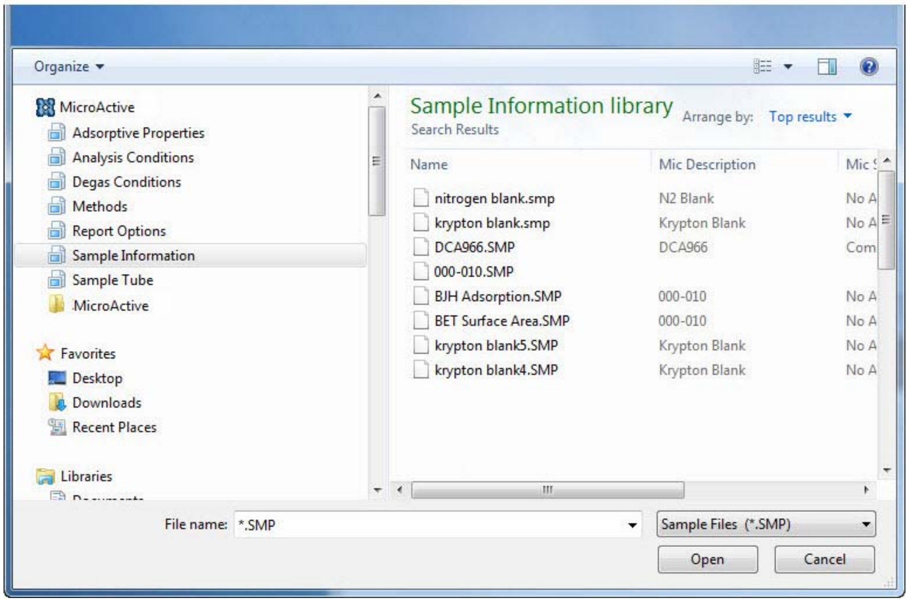
Single point surface area: 209.7479 m²/g
at relative pressure: 0.212851443

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

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

1. Go to ***File*** > ***Open*** or use the **F2** keyboard shortcut.

2. From the Sample Information library folder, select a .SMP file with a status of *Preparing*, *Prepared*, or *No Analysis* and click **Open** (or double click the file name).

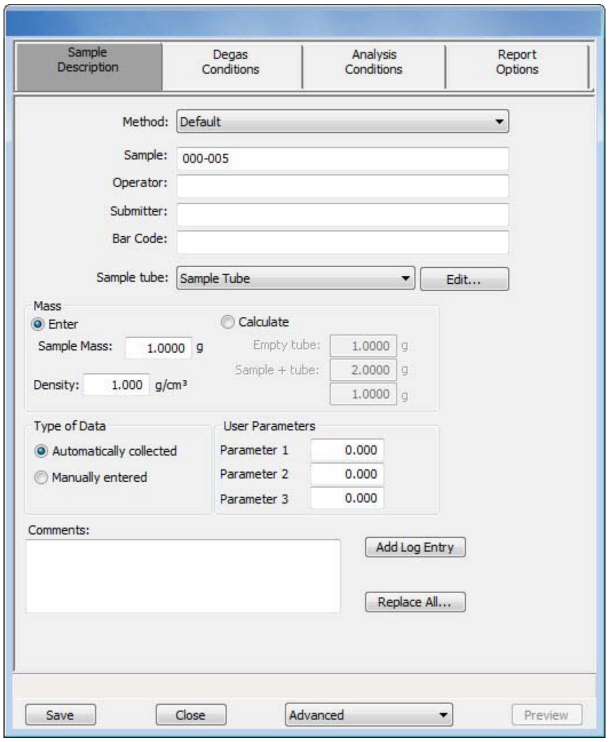


Name	Mic Description	Mic S
nitrogen blank.smp	N2 Blank	No A
krypton blank.smp	Krypton Blank	No A
DCA966.SMP	DCA966	Com
000-010.SMP		
BJH Adsorption.SMP	000-010	No A
BET Surface Area.SMP	000-010	No A
krypton blank5.SMP	Krypton Blank	No A
krypton blank4.SMP	Krypton Blank	No A

File name: *.SMP

Sample Files (*.SMP)

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



Method: Default

Sample: 000-005

Operator:

Submitter:

Bar Code:

Sample tube: Sample Tube

Mass

● Enter

Sample Mass: 1.0000 g

Density: 1.000 g/cm³

○ Calculate

Empty tube: 1.0000 g

Sample + tube: 2.0000 g

1.0000 g

Type of Data

● Automatically collected

○ Manually entered

User Parameters

Parameter 1 0.000

Parameter 2 0.000

Parameter 3 0.000

Comments:

Advanced

Defining Parameter Files

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

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

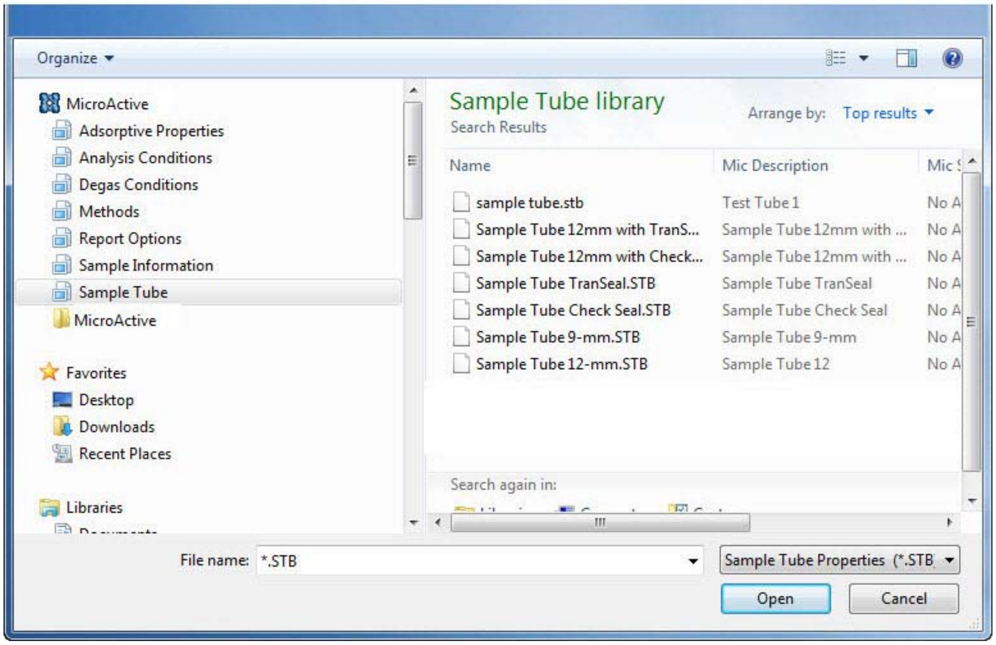
Default parameter files can be used for multiple analyses without having to re-enter the values each time an analysis is performed.

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

Sample Tube

Sample Tube files specify information about the sample tube.

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



Name	Mic Description	Mic S
sample tube.stb	Test Tube 1	No A
Sample Tube 12mm with TranS...	Sample Tube 12mm with ...	No A
Sample Tube 12mm with Check...	Sample Tube 12mm with ...	No A
Sample Tube TranSeal.STB	Sample Tube TranSeal	No A
Sample Tube Check Seal.STB	Sample Tube Check Seal	No A
Sample Tube 9-mm.STB	Sample Tube 9-mm	No A
Sample Tube 12-mm.STB	Sample Tube 12	No A

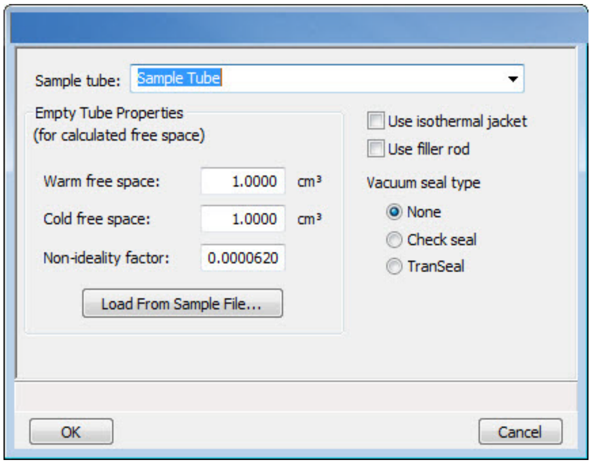
File name: *.STB

Sample Tube Properties (*.STB

2. Click **Open**.

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

4. Enter a description of the sample tube in the **Description** field. This description displays in the **Sample tube** dropdown list on the **Sample Description** tab.



Sample tube: Sample Tube

Empty Tube Properties
(for calculated free space)

Warm free space: 1.0000 cm^3

Cold free space: 1.0000 cm^3

Non-ideality factor: 0.0000620

☐ Use isothermal jacket

☐ Use filler rod

Vacuum seal type

● None

○ Check seal

○ TranSeal

5. If using calculated free space, enter the warm free space, cold free space and non-ideality factor; or click **Load from Sample File** to import the information from an existing sample information file.

6. Indicate if an isothermal jacket and/or filler rod will be used by selecting the **Use isothermal jacket** checkbox and/or **User filler rod** checkbox. Filler rods help to ensure accuracy in samples with lower total surface areas by reducing the free-space volume. It is generally a good practice to use filler rods for samples having less than 100 square meters of total surface area. Filler rods are unnecessary for samples with total surface areas greater than 100 square meters.

7. Select the vacuum seal type to be used.

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

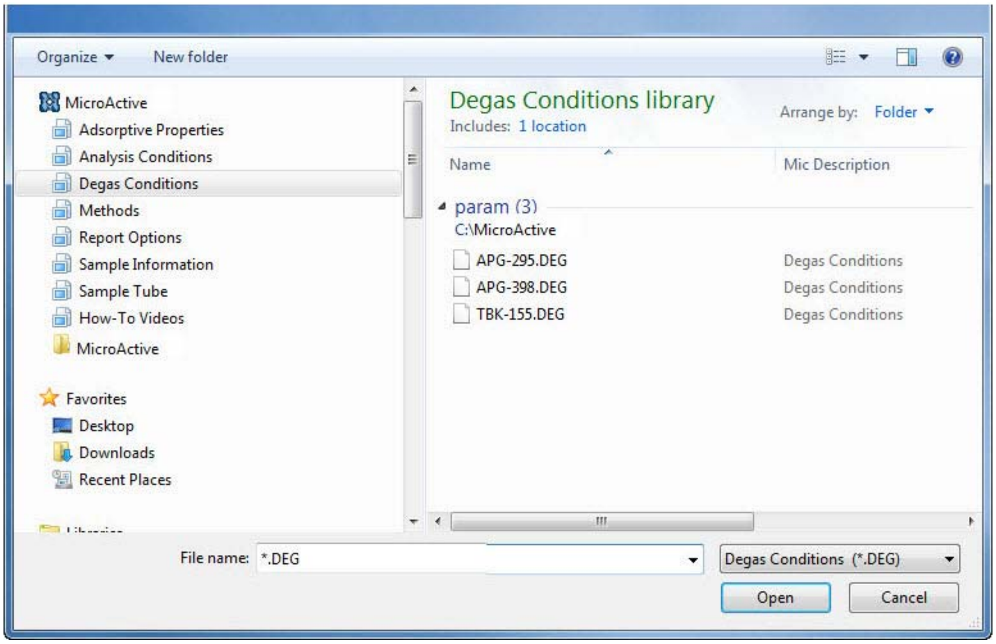
Degas Conditions

Degas Conditions files contain degassing information for sample preparation.

Degassing is a required step in preparation for the analysis; however, the Degas Conditions tab is only applicable if using the SmartPrep Degasser. This section contains degassing instructions that will be sent to the SmartPrep Degasser equipment.

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

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



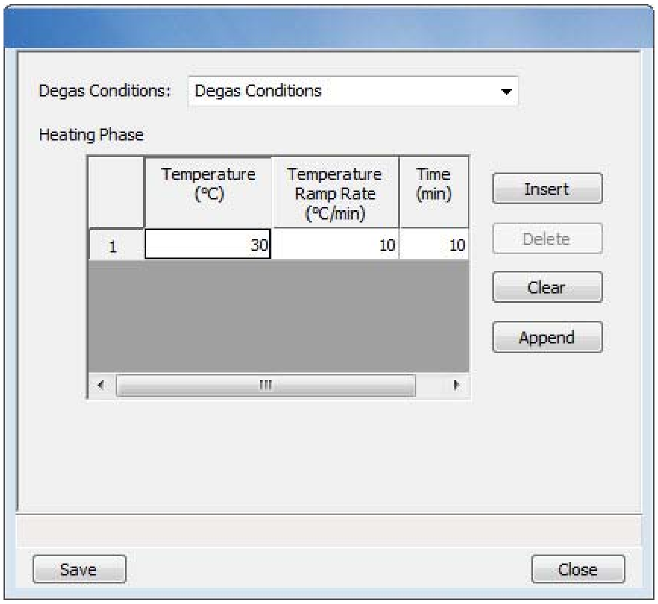
Name	Mic Description
APG-295.DEG	Degas Conditions
APG-398.DEG	Degas Conditions
TBK-155.DEG	Degas Conditions

File name: *.DEG

Degas Conditions (*.DEG)

2. Click **Open**.

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



Degas Conditions: Degas Conditions

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

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

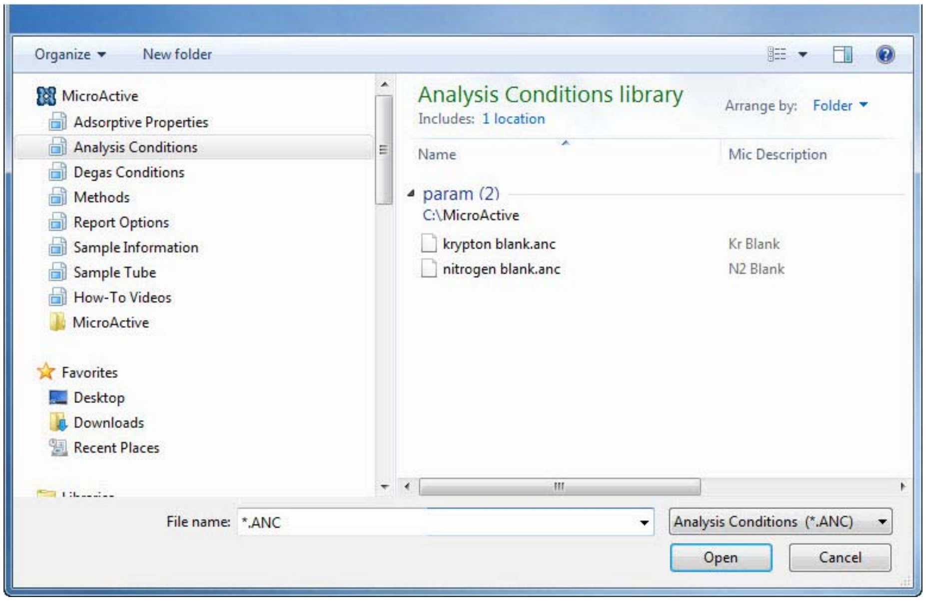
5. Use the **Insert** button to enter up to three stages of degassing (temperature, ramp rate, and time). When using the SmartPrep degasser, the maximum temperature that can be entered is 450 °C.

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

Analysis Conditions

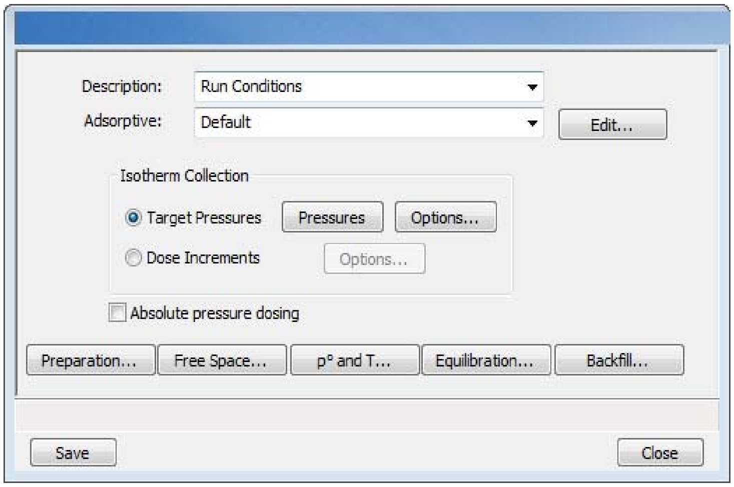
Analysis conditions specify the data used to guide an analysis.

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



2. Click **Open**.

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



4. To overwrite analysis conditions with parameters from another Analysis Conditions file, either click the **Description** dropdown arrow and select a file from the list or click **Browse** then locate and select the .ANC file containing the new parameters and click **Open**.

5. To overwrite adsorptive properties with parameters from another Adsorptive Properties file, click the **Adsorptive** dropdown arrow and select an adsorptive from the list or click **Browse**, then locate and select a .ADP file containing the new parameters and click **Open**. Refer to **Adsorptive Properties**, page **2-22** and **Analysis Conditions Files**, page **3-16**.

6. To enter starting and ending relative pressure points, select **Target Pressures** then click the **Pressures** button. Click the **Insert Range** button.

7. To indicate target pressure options, click the **Options** button and enter the appropriate values.

8. To specify dosing increments, select **Dose Increments** and click the **Options** button.

9. To specify pressure targets in mmHg, mbar, or kPa instead of relative pressure, select the **Absolute pressure dosing** checkbox. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, H_2 adsorption on carbon at liquid nitrogen temperature.

10. Click the following buttons to specify:

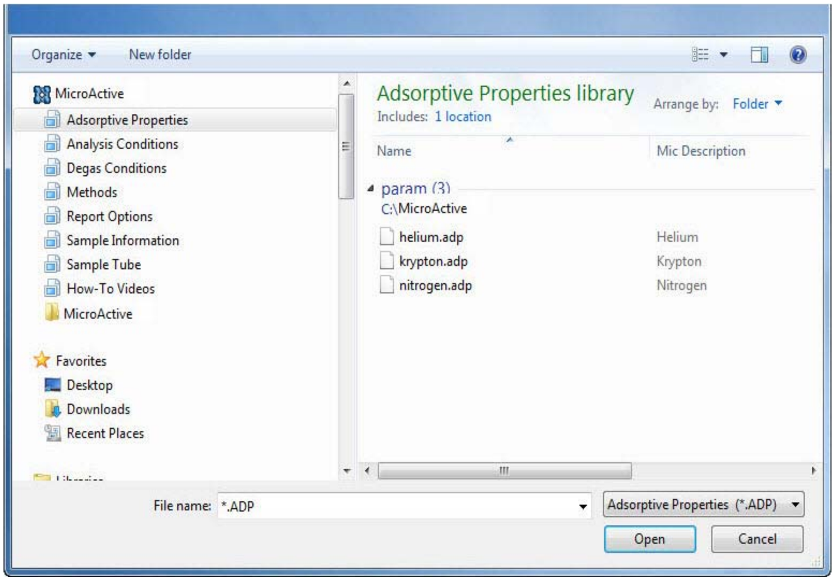
Button	Use to Specify...
Preparation	evacuation rate/time/level, leak test and time values, and elevator prompts
Free space	how the free space is to be measured
p^o and T	how the saturation pressure (Po) is to be measured or calculated and the analysis bath temperature
Equilibration	options for absolute and/or relative pressure tolerance
Backfill	backfill options after analysis

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

Adsorptive Properties

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

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

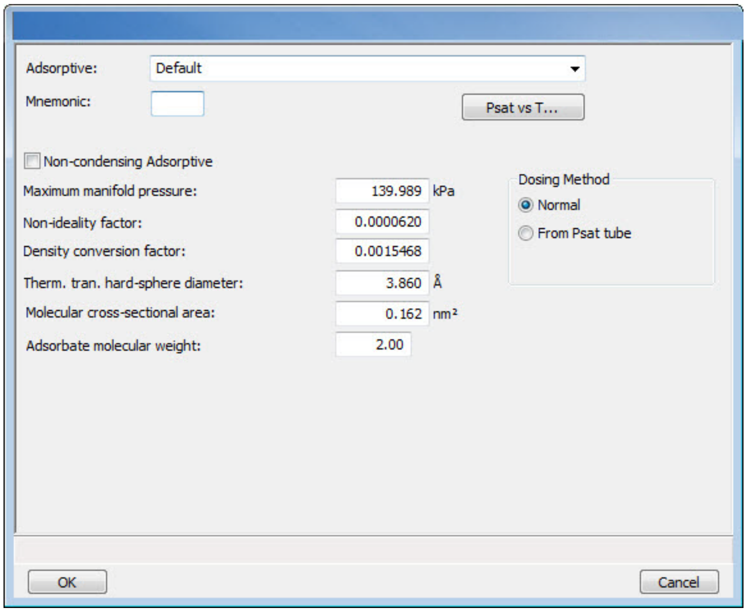


File name: *.ADP

2. Click **Open**.

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

4. 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** dropdown list of the Analysis Conditions tab.



Adsorptive: Default

Mnemonic:

☐ Non-condensing Adsorptive

Maximum manifold pressure: 139.989 kPa

Non-ideality factor: 0.0000620

Density conversion factor: 0.0015468

Therm. tran. hard-sphere diameter: 3.860 Å

Molecular cross-sectional area: 0.162 nm²

Adsorbate molecular weight: 2.00

Dosing Method: ● Normal ○ From Psat tube

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

6. Select the **Non-condensing Adsorptive** checkbox if this file is to be used for a non-condensing gas. The **Absolute pressure dosing** option on the Analysis Conditions tab must also be selected when **Non-condensing Adsorptive** is selected on this window.

   When the **Non-condensing Adsorptive** checkbox is selected, the **Density conversion factor** field and the **Psat vs T** button are disabled.

7. Enter information in the following text boxes:

   - **Maximum manifold pressure** - the highest pressure that the manifold will be dosed. To avoid damage to the instrument, this number is limited to 925 mmHg. Low pressure sources, such as vapors, will require lower numbers.

   - **Non-ideality factor** - factor used to compensate for the forces of attraction between molecules in a real gas. This value is used for a calculated free space.

   - **Density conversion factor** - enter the density conversion factor which is determined by obtaining the ratio of the gas density (STP) to the liquid volume. This field is disabled if the **Non-condensing Adsorptive** checkbox is selected.

$$\frac{\rho_{gas}}{\rho_{liq}} \quad \text{or} \quad \frac{\upsilon^{l}}{\upsilon^{g}}$$

where

ϱ_{gas} = gas density at STP
ϱ_{liq} = liquid density
υ^{l} = molar liquid volume
υ^{g} = molar gas volume at STP

   - **Therm. tran. hard-sphere diameter** - an estimate of molecular size used in calculating the thermal transpiration correction.

   - **Molecular cross-sectional area** - the area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations.

   - **Adsorbate molecular weight** - the molecular weight is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot.

8. In the **Dosing Method** group box, select the source to dose the adsorptive:

   - **Normal** - dose from a pressurized tank of gas attached to a gas inlet port.

   - **From Psat tube** - the Psat tube is filled with condensed adsorptive and dosed from the Psat tube. This is typically used for Krypton. The instrument will determine the maximum pressure that can be dosed based on the analysis temperature and the saturation pressure information in the Fluid Properties.

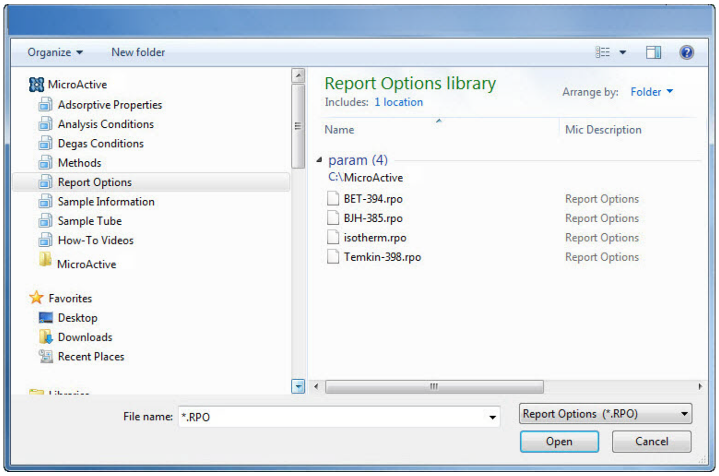
9. Click **Save**, then click **Close**.

Report Options

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

Report Options files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis. Refer to **Generating Graph Overlays**, page **2-49**.

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

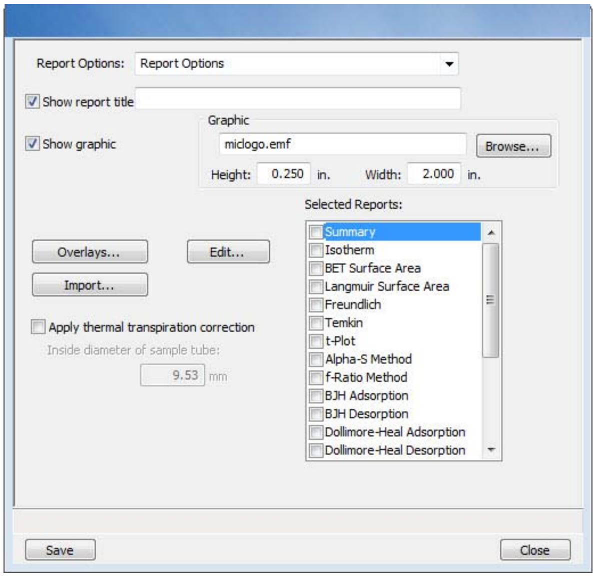


Name	Mic Description
BET-394.rpo	Report Options
BJH-385.rpo	Report Options
isotherm.rpo	Report Options
Temkin-398.rpo	Report Options

File name: *.RPO
Report Options (*.RPO)

2. Click **Open**.

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



Report Options: Report Options
☑ Show report title
☑ Show graphic
Graphic: micdogo.emf
Height: 0.250 in. Width: 2.000 in.
☐ Apply thermal transpiration correction
Inside diameter of sample tube: 9.53 mm

Selected Reports:
☐ Summary
☐ Isotherm
☐ BET Surface Area
☐ Langmuir Surface Area
☐ Freundlich
☐ Temkin
☐ t-Plot
☐ Alpha-S Method
☐ f-Ratio Method
☐ BJH Adsorption
☐ BJH Desorption
☐ Dollimore-Heal Adsorption
☐ Dollimore-Heal Desorption

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

For information on the **Overlays** and **Import** buttons, refer to **Generating Graph Overlays**, page **2-49**.

Manually Entering Isotherm Data in a Sample File

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

Importing Manually Entered Isotherm Data

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

ASCII text file format rules

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

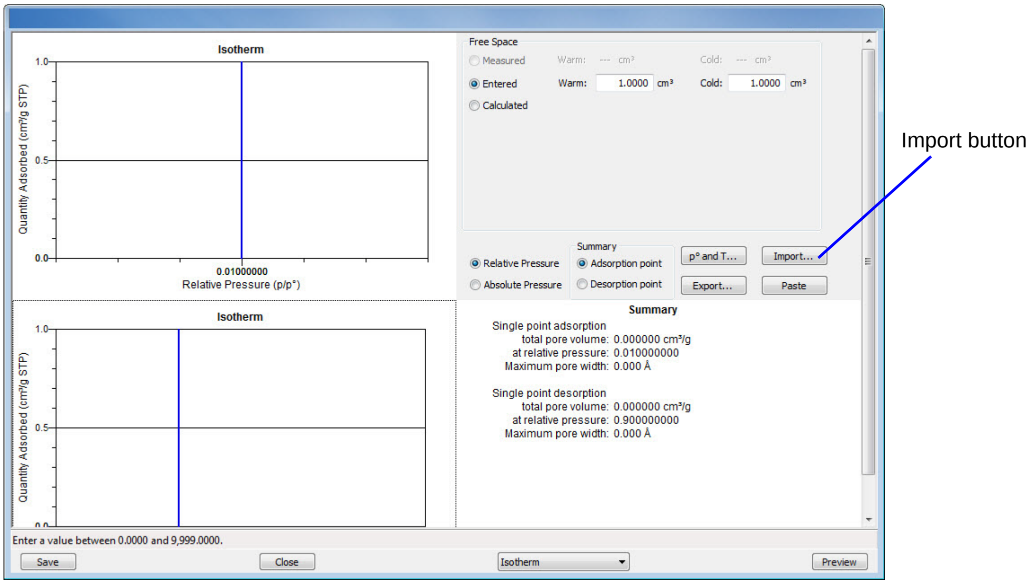
  Relative Pressure
  Absolute Pressure (mmHg)
  Absolute Pressure (kPa)
  Absolute Pressure (mbar)
  Quantity Adsorbed (mmol/g)
  Quantity Adsorbed (cm^3/g STP)
  Quantity Adsorbed (cm3/g STP)

Sample ASCII text file

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

To import the ASCII text file:

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



Free Space
○ Measured Warm: --- cm³ Cold: --- cm³
● Entered Warm: 1.0000 cm³ Cold: 1.0000 cm³
○ Calculated

● Relative Pressure
○ Absolute Pressure

Summary
● Adsorption point
○ Desorption point

Summary

Single point adsorption
total pore volume: 0.000000 cm³/g
at relative pressure: 0.010000000
Maximum pore width: 0.000 Å

Single point desorption
total pore volume: 0.000000 cm³/g
at relative pressure: 0.900000000
Maximum pore width: 0.000 Å

Enter a value between 0.0000 and 9,999.0000.

Isotherm

Import button

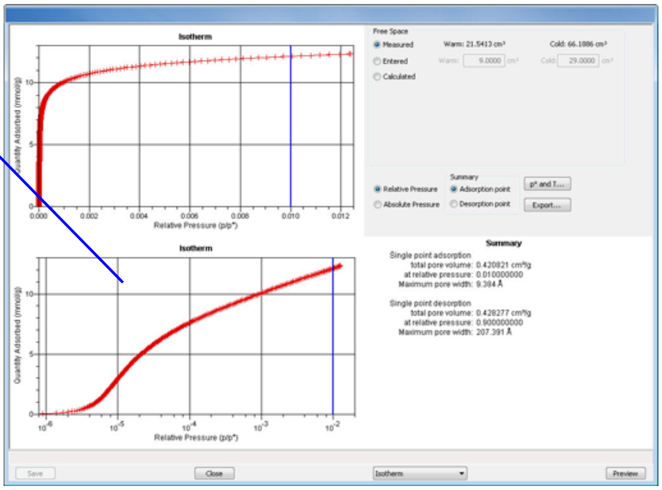
5. Ensure that all parameter fields are set appropriately then click **Import**.
6. On the File Selector window, locate and select the .TXT file and click **Open**. The isotherm data from the original sample file is imported and displays in the new sample file. If an error message appears instead, verify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

Copying / Pasting Manually Entered Isotherm Data

1. Go to ***File*** > ***Open [.SMP file]*** and select the sample information file that contains the isotherm data to be copied and pasted. This file must have a *Complete* status.

2. Click **Open**. The file will open to the interactive isotherm report window.

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



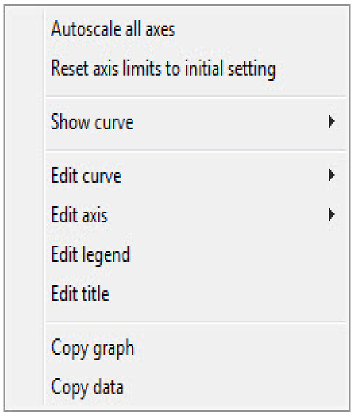
Free Space
● Measured Warm: 21.5413 cm³ Cold: 66.1806 cm³
○ Entered Warm: 9.0000 cm³ Cold: 29.0000 cm³
○ Calculated

Summary

Single point adsorption
total pore volume: 0.420821 cm³/g
at relative pressure: 0.010000000
Maximum pore width: 9.384 Å

Single point desorption
total pore volume: 0.428277 cm³/g
at relative pressure: 0.900000000
Maximum pore width: 207.391 Å

Right click in the graph area and select **Copy Data**

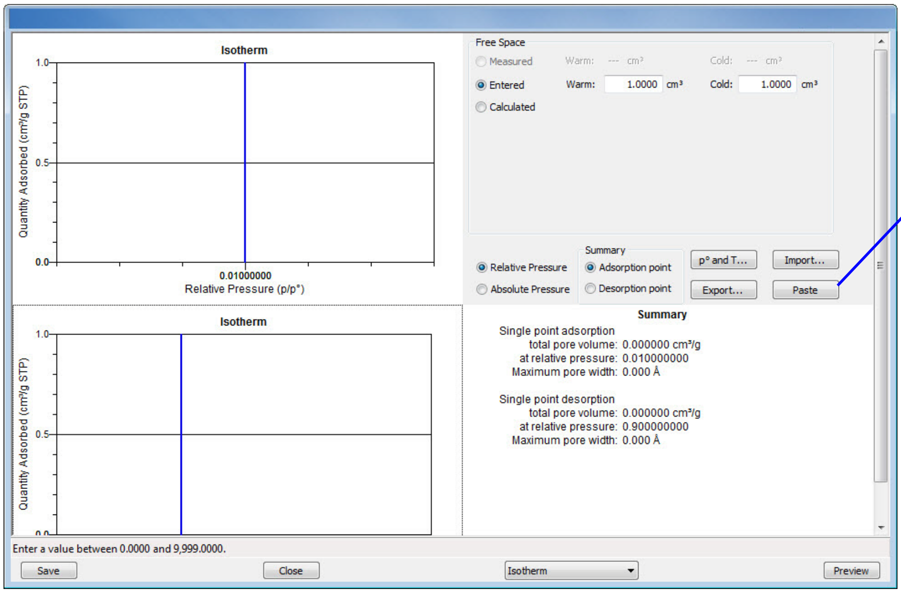


4. Go to ***File*** > ***New Sample*** and open a new sample information file.

5. On the **Sample Description** window, select **Manually entered** in the **Type of Data** group box.

6. In the dropdown list at the bottom of the window, select **Isotherm**.

7. Resize the interactive isotherm window until the **Paste** button displays.



Free Space
○ Measured Warm: --- cm³ Cold: --- cm³
● Entered Warm: 1.0000 cm³ Cold: 1.0000 cm³
○ Calculated

● Relative Pressure
○ Absolute Pressure

Summary
● Adsorption point
○ Desorption point

Summary

Single point adsorption
total pore volume: 0.000000 cm³/g
at relative pressure: 0.010000000
Maximum pore width: 0.000 Å

Single point desorption
total pore volume: 0.000000 cm³/g
at relative pressure: 0.900000000
Maximum pore width: 0.000 Å

Enter a value between 0.0000 and 9,999.0000.

Paste button

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

Preparing for Analysis

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

Task	Name and Location
Clean the sample tube	**Cleaning and Labeling Sample Tubes**, page **2-31**
Create the sample file	**Defining Sample Information Files**, page **2-9**
Weigh the sample	**Determining the Sample Mass**, page **2-33**
Degas the sample	**Degassing the Sample**, page **2-35**
Load sample on sample port	**Installing the Sample Tube**, page **2-35**
Fill dewar and check LN2 level	**Filling and Installing the Analysis Dewar**, page **2-37**

Cleaning and Labeling Sample Tubes

The equipment shown in the following section may differ slightly from your equipment however the process remains the same.

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

Materials Supplied by Micromeritics	Materials Supplied by User
• Sample tube • Filler rod • Sample tube brush • Stopper for sample tube • Sample tube rack • Sample weighing support • Sample data worksheet (copied from Appendix A)	• Drying oven • Ultrasonic cleaning unit • Detergent • Rubber gloves or lint-free cloth • Acetone or isopropyl alcohol • Safety glasses • Waste container • Analytical balance • Pipe cleaners

1. Preheat drying oven at 110 ºC.

2. Verify that the ultrasonic cleaning unit is clean.

3. Use 5 grams of Alconox (or other suitable detergent) per 500 mL of warm water and fill the ultrasonic unit with enough water to cover the sample tubes and filler rods (if used). Ensure the detergent is dissolved before placing the sample tubes and filler rods into the water. If too much detergent is used, it may be difficult to rinse from the sample tubes.

4. Fill the sample tubes with warm water and place them in the ultrasonic cleaning unit. Then place the filler rods in the unit. Turn on the ultrasonic cleaning unit for approximately fifteen minutes.

5. Use rubber gloves to ensure no oils or residue are transferred to the clean tubes and filler rods and remove the sample tubes and filler rods from the unit.

6. Clean the interior of the sample tubes with the brush supplied with the system.

7. Rinse the sample tubes and filler rods thoroughly with hot water. Then rinse again with isopropyl alcohol or acetone. If isopropyl alcohol or acetone is not available, deionized water may be used.

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

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

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

10. Wipe a rubber stopper with a lint-free cloth.

11. Label the sample tube and stopper for identification.

Determining the Sample Mass

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

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

A **Sample Data Worksheet** for recording the weights and calculating the mass is included in Appendix A. Make copies as needed.

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

1. Record the *Sample Tube Identification*.

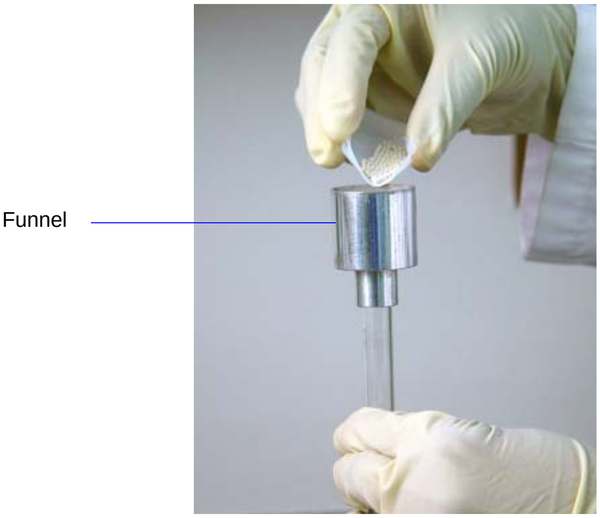
2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero.

3. Place the sample tube set on the balance.

4. Record the stabilized weight on the Sample Data Worksheet as *[A] Mass for empty sample tube set*. Remove the sample weighing support and sample tube set from the balance.

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

5. Place a sample container on the balance and slowly pour the sample into the container.
6. Remove the rubber stopper (check seal or TranSeal) from the sample tube.
7. Use the sample tube funnel (provided in the accessories kit) and pour the sample from the weighing container into the sample tube.



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

9. Weigh the sample tube set containing the sample and record the value on the Sample Data Worksheet as *[B] Sample tube set plus sample mass (Before Degas)*.

10. Subtract the *[A] Mass for empty sample tube set* from the *[B] Mass of sample tube set plus sample* and record this value as the *[C] Sample mass (Before Degas)*.

Degassing the Sample

After the sample has been weighed, use a degassing unit to remove any contaminants which may have adsorbed to the surface or pores. Appropriate degassing units are available from Micromeritics. Refer to **ORDERING INFORMATION**, page **8-1** for ordering information.

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

After degassing is complete, perform the following steps:

1. Weigh the sample tube set containing the sample and record the mass on the Sample Data Worksheet as *[B] Sample tube set plus sample mass (After Degas)*.

2. Subtract the *[A] Mass for empty sample tube set (Before Degas)* from the *[B] Sample tube set plus sample mass (After Degas)* to obtain the sample's mass. Record this value as *[C] Sample mass (After Degas)*.

Installing the Sample Tube

The equipment shown in the following section may differ slightly from your equipment however the process remains the same.

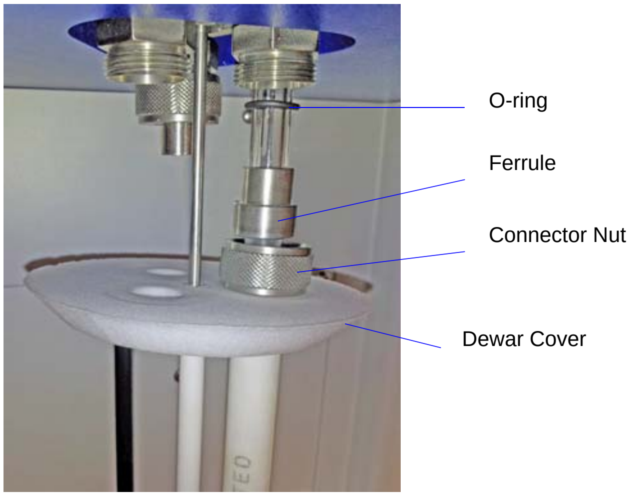
Repeat the following steps for each sample to be installed. Up to three sample tubes can be installed. To install a sample tube to a port:

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

1. Remove the sample tube stopper.
2. If using a filler rod, hold the sample tube horizontally and carefully slide the filler rod into the tube.

Do not hold the rod vertically and drop the rod into the tube; this could break the rod and/or the tube.

3. If using an isothermal jacket, slide the jacket down over the stem of the sample tube until it touches the sample tube bulb.
4. Insert the Po tube into the center hole of the Dewar cover and slide the cover up. Ensure the tapered side of the cover is facing downward.
5. Insert the sample tube through one of the holes in the cover.



6. Place the connector nut, ferrule and O-ring onto the sample tube stem.
7. Attach sample tube to the analysis port and ensure it is completely in the port. Securely screw the connector nut onto the analysis port. Hand-tighten the connector nut.
8. Slide the Dewar cover upward until it stops.

Filling and Installing the Analysis Dewar

The equipment shown in the following section may differ slightly from your equipment however the process remains the same.

Prepare the analysis dewar after installing the sample tubes.

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

When handling dewars containing cryogenic liquids:

- Wear protection by using:
  - goggles (or a face shield)
  - an insulated or rubber apron
  - insulated gloves
- When transferring cryogenic liquids from one container to another:
  - cool the receiving container gradually to minimize thermal shock
  - pour the liquefied gas slowly to prevent splashing
  - vent the receiving container to the atmosphere

For glass dewars:

- Use a plastic stirring rod when stirring substances in a dewar containing cryogenic liquids (or other materials of extremely low temperature). Do not use a glass or metal stirring rod unless it has a protective coating.
- Do not handle heavy objects above the dewar. If unavoidable, place a protective cover over the dewar’s opening. If an object of sufficient weight is accidentally dropped into the dewar, shattering may occur.

To fill and install the analysis dewar:

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

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

2. Insert the dipstick into the dewar and check the level of the analysis bath liquid. Condensation should not exceed the Level Indicator mark.

3. For best results, if the dewar has not been used for a while, allow approximately 30 minutes for the temperature of the dewar to stabilize with the bath liquid then recheck the level of the bath liquid. Add additional liquid if necessary.

4. If using isothermal jackets, slide the jackets down the sample tube until the jackets touch the sample tube bulbs.

5. Slide the dewar lid to approximately 3/4 in (or 19 mm) from the sample port nuts to ensure a proper seal on the top of the dewar.

6. Close the safety shield on the front of the instrument.

Performing an Analysis

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

Sample Analysis

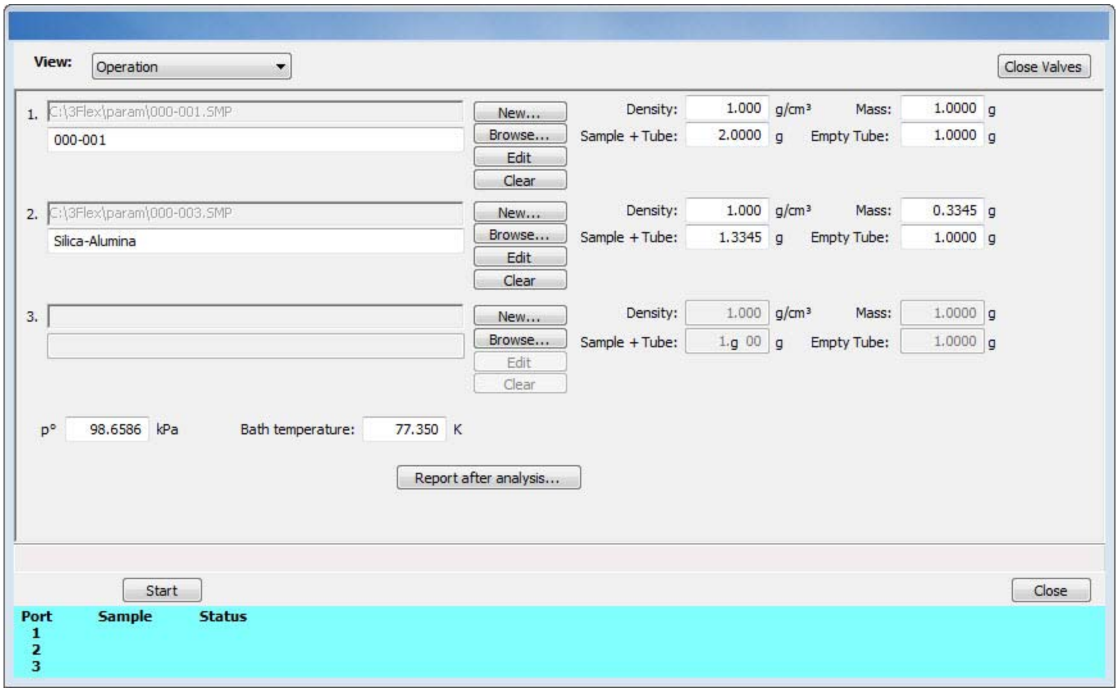
Allows one analysis using different analysis conditions to run on each port.

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

- If **Krypton** is selected as the adsorptive, there must be a 10 mmHg transducer present on the port.
- The minimum target pressure must be greater than the Minimum Record Pressure for the minimum range transducer present on the port.
- The selected sample files will be checked for matching adsorptive gases, matching Psat or Po gases if measured, and matching backfill gases.
- If any selected sample file specifies an Adsorptive Dose Method from port 3 and a sample file is selected for port 3, an error message displays indicating the problem and the **Start** window will remain active.

Additional analyses can be scheduled by clicking **Next** after the completion of the first series of analyses. The **Next** button appears after the first set of analyses is complete. Samples cannot be removed from or added to ports until the full set of analyses has completed.

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



View: Operation

1. C:\3Flex\param\000-001.SMP
000-001
Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 2.0000 g Empty Tube: 1.0000 g

2. C:\3Flex\param\000-003.SMP
Silica-Alumina
Density: 1.000 g/cm³ Mass: 0.3345 g
Sample + Tube: 1.3345 g Empty Tube: 1.0000 g

3.
Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 1.g 00 g Empty Tube: 1.0000 g

p° 98.6586 kPa Bath temperature: 77.350 K

Port	Sample	Status
1		
2		
3		

2. To manually close all instrument valves, click the **Close Valve**s button.
3. For each port to be used, either click **Browse** and select a sample information file or click **New** to create a new sample information file.
4. Verify the information populated into the sample identification, **Density**, **Mass**, **Sample + Tube**, and **Empty Tube** fields. This information is pulled from the selected or newly created file. The **Density** value is applicable only if using the **Calculate** method for the free space determination.
5. Edit the **p**^{o} and **Bath temperature** fields, if necessary.
6. Click **Report after analysis** to automatically generate reports when the analysis is complete. On the **Report Settings** window, select the report destination. Click **OK** to return to the previous window.
7. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the Idle state.
8. When the analysis is complete, remove the sample tube and store (or dispose of) the sample material as applicable.

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

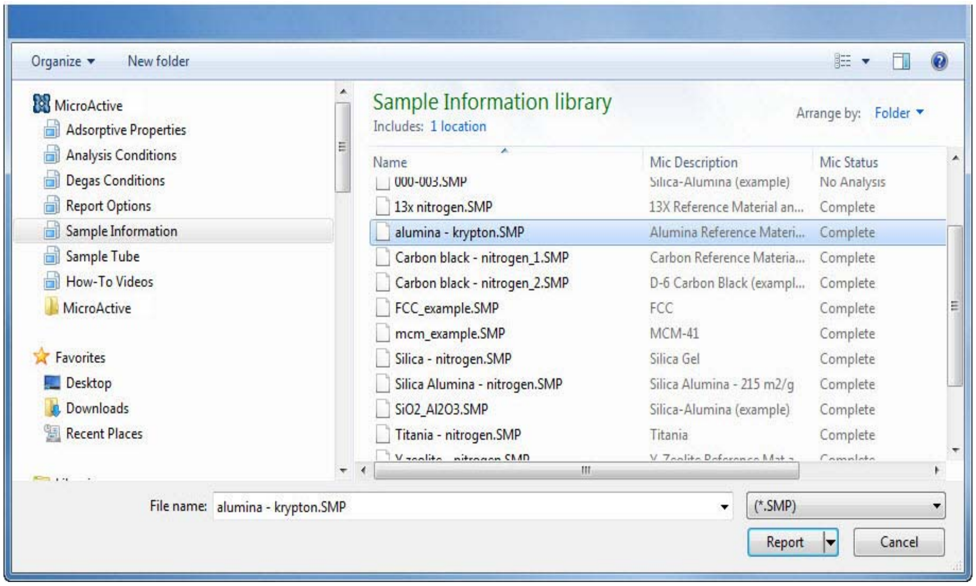
Empty Tube Analysis

An empty tube analysis is performed in the same manner as a sample analysis however, the sample tubes will not contain sample material.

1. Install empty tubes with filler rods and isothermal jackets into each of the sample ports.
2. Be sure to use new O-rings that are new or in good condition on the sample tubes.
3. Make sure the dewar lid is attached.
4. Verify that the isothermal jacket and filler rods are checked as being used in the sample tube file.
5. Reference **Defining Sample Information Files**, page **2-9** for instructions on defining the sample information file.

Generating Reports

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



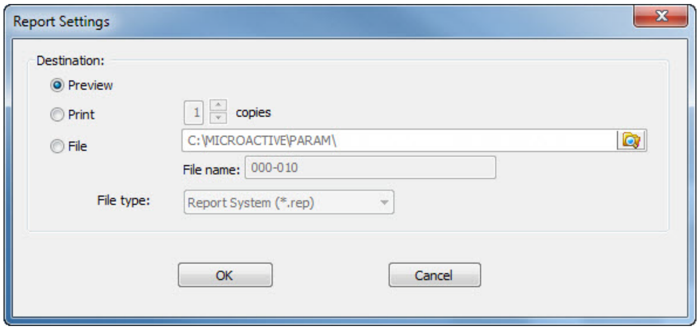
Name	Mic Description	Mic Status
000-003.SMP	Silica-Alumina (example)	No Analysis
13x nitrogen.SMP	13X Reference Material an...	Complete
alumina - krypton.SMP	Alumina Reference Materi...	Complete
Carbon black - nitrogen_1.SMP	Carbon Reference Materia...	Complete
Carbon black - nitrogen_2.SMP	D-6 Carbon Black (exampl...	Complete
FCC_example.SMP	FCC	Complete
mcm_example.SMP	MCM-41	Complete
Silica - nitrogen.SMP	Silica Gel	Complete
Silica Alumina - nitrogen.SMP	Silica Alumina - 215 m2/g	Complete
SiO2_Al2O3.SMP	Silica-Alumina (example)	Complete
Titania - nitrogen.SMP	Titania	Complete

File name: alumina - krypton.SMP

(*.SMP)

1. Select a .SMP file from the library. The selected report name appears in the **File name** text box. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. Click **Report**.

2. Select the report destination in the **Report Settings** window and click **OK**.



Destination:

● Preview

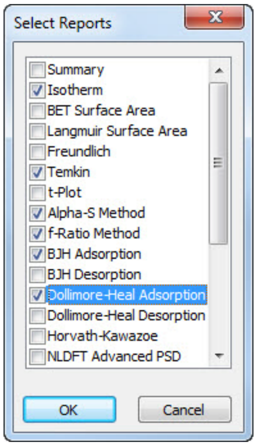
○ Print 1 copies

○ File C:\MICROACTIVE\PARAM\

File name: 000-010

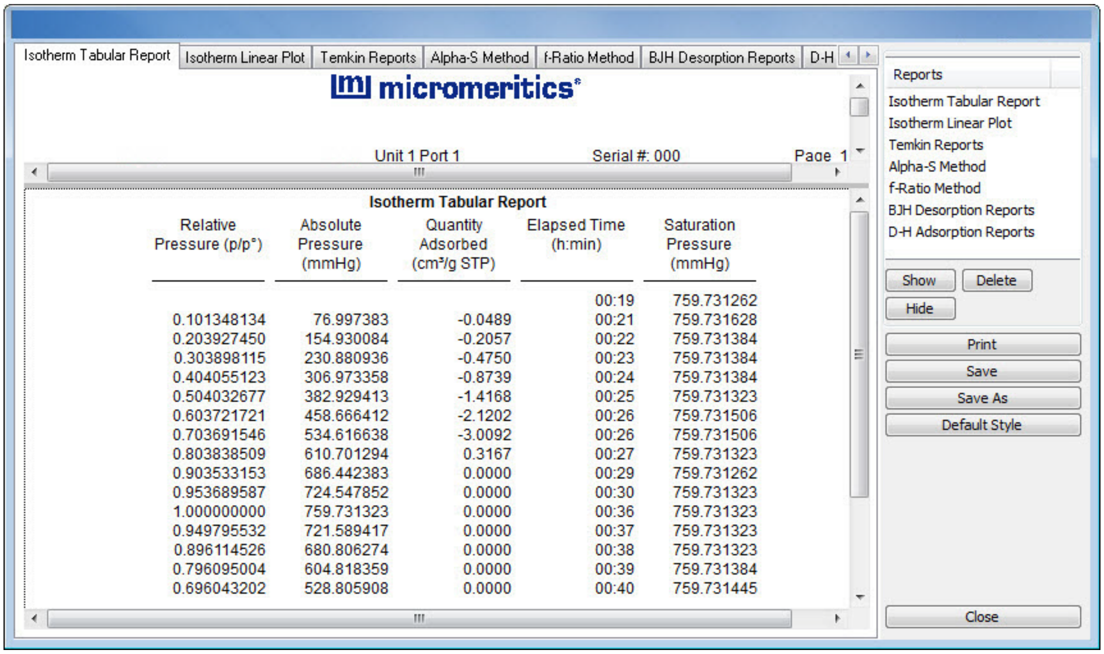
File type: Report System (*.rep)

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



☐ Summary
☑ Isotherm
☐ BET Surface Area
☐ Langmuir Surface Area
☐ Freundlich
☑ Temkin
☐ t-Plot
☑ Alpha-S Method
☑ f-Ratio Method
☑ BJH Adsorption
☐ BJH Desorption
☑ Dollimore-Heal Adsorption
☐ Dollimore-Heal Desorption
☐ Horvath-Kawazoe
☐ NLDFT Advanced PSD

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



Unit 1 Port 1 Serial #: 000 Page 1

Isotherm Tabular Report

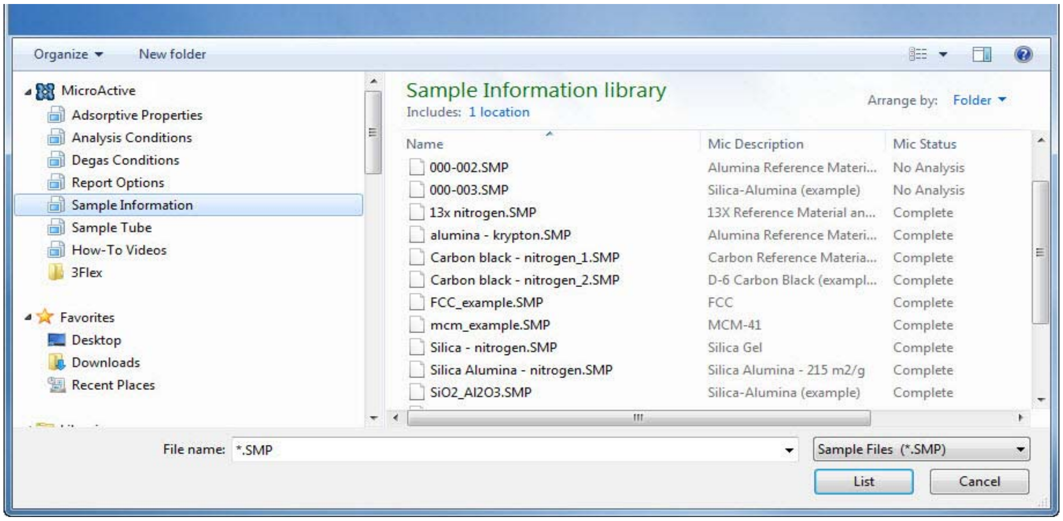
Relative Pressure (p/p°)	Absolute Pressure (mmHg)	Quantity Adsorbed (cm³/g STP)	Elapsed Time (h:min)	Saturation Pressure (mmHg)
			00:19	759.731262
0.101348134	76.997383	-0.0489	00:21	759.731628
0.203927450	154.930084	-0.2057	00:22	759.731384
0.303898115	230.880936	-0.4750	00:23	759.731384
0.404055123	306.973358	-0.8739	00:24	759.731384
0.504032677	382.929413	-1.4168	00:25	759.731323
0.603721721	458.666412	-2.1202	00:26	759.731506
0.703691546	534.616638	-3.0092	00:26	759.731506
0.803838509	610.701294	0.3167	00:27	759.731323
0.903533153	686.442383	0.0000	00:29	759.731262
0.953689587	724.547852	0.0000	00:30	759.731323
1.000000000	759.731323	0.0000	00:36	759.731323
0.949795532	721.589417	0.0000	00:37	759.731323
0.896114526	680.806274	0.0000	00:38	759.731323
0.796095004	604.818359	0.0000	00:39	759.731384
0.696043202	528.805908	0.0000	00:40	759.731445

Listing Files

File > List

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

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



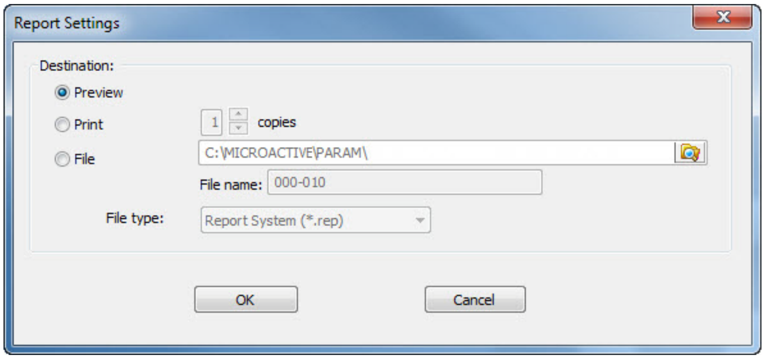
Name	Mic Description	Mic Status
000-002.SMP	Alumina Reference Materi...	No Analysis
000-003.SMP	Silica-Alumina (example)	No Analysis
13x nitrogen.SMP	13X Reference Material an...	Complete
alumina - krypton.SMP	Alumina Reference Materi...	Complete
Carbon black - nitrogen_1.SMP	Carbon Reference Materia...	Complete
Carbon black - nitrogen_2.SMP	D-6 Carbon Black (exampl...	Complete
FCC_example.SMP	FCC	Complete
mcm_example.SMP	MCM-41	Complete
Silica - nitrogen.SMP	Silica Gel	Complete
Silica Alumina - nitrogen.SMP	Silica Alumina - 215 m2/g	Complete
SiO2_Al2O3.SMP	Silica-Alumina (example)	Complete

File name: *.SMP

2. Click **List**.

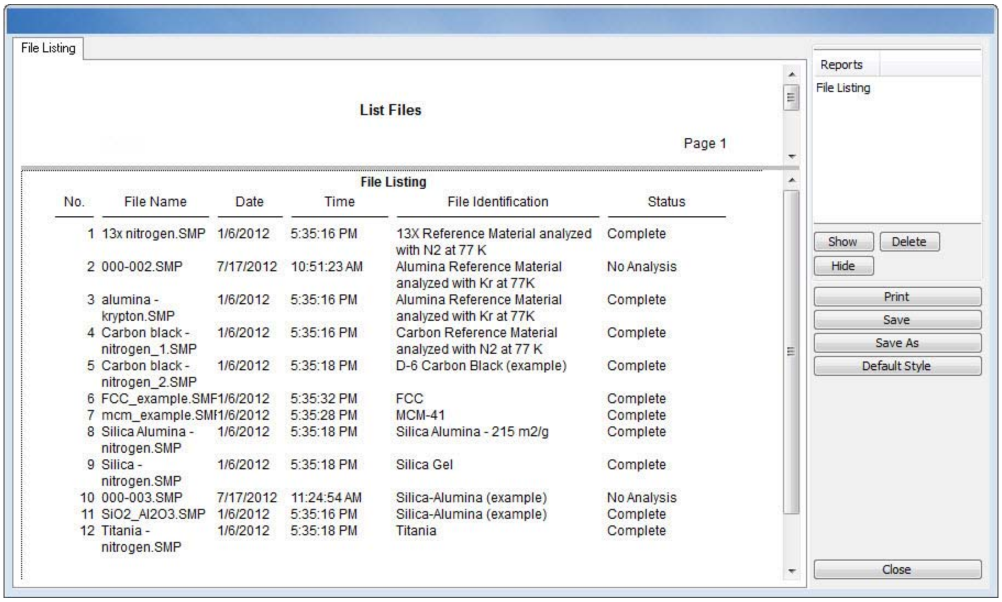
3. In the **Report Settings** window, select one of the following:

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



Destination:
● Preview
○ Print 1 copies
○ File C:\MICROACTIVE\PARAM\
File name: 000-010
File type: Report System (*.rep)

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



List Files

Page 1

File Listing

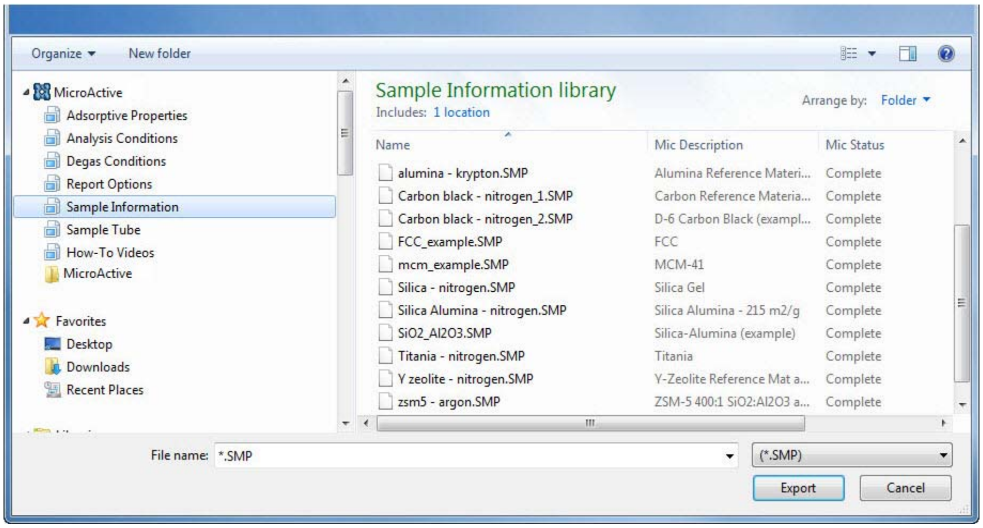
No.	File Name	Date	Time	File Identification	Status
1	13x nitrogen.SMP	1/6/2012	5:35:16 PM	13X Reference Material analyzed with N2 at 77 K	Complete
2	000-002.SMP	7/17/2012	10:51:23 AM	Alumina Reference Material analyzed with Kr at 77K	No Analysis
3	alumina - krypton.SMP	1/6/2012	5:35:16 PM	Alumina Reference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

Exporting Files

File > Export

Provides the option to print the contents of one or more sample files to either the screen, a printer, or to a file. Isotherm data can be exported as a .REP, .TXT, or .XLS file format. 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 applications that read .TXT or .XLS file formats.

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



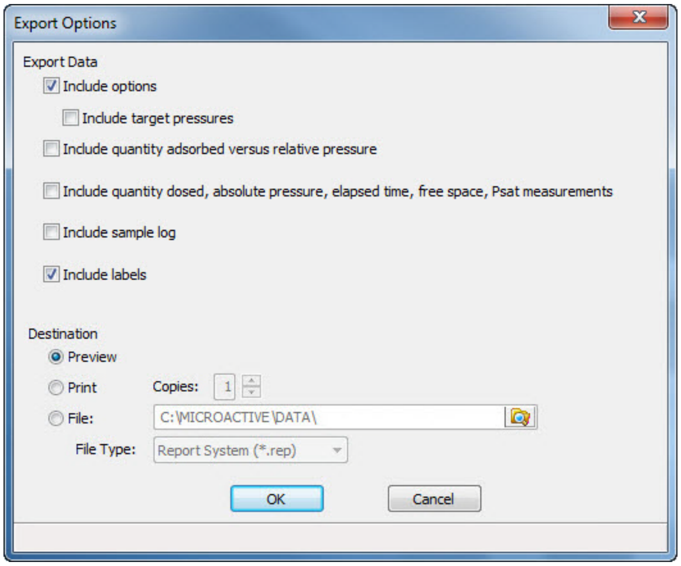
Name	Mic Description	Mic Status
alumina - krypton.SMP	Alumina Reference Materi...	Complete
Carbon black - nitrogen_1.SMP	Carbon Reference Materia...	Complete
Carbon black - nitrogen_2.SMP	D-6 Carbon Black (exampl...	Complete
FCC_example.SMP	FCC	Complete
mcm_example.SMP	MCM-41	Complete
Silica - nitrogen.SMP	Silica Gel	Complete
Silica Alumina - nitrogen.SMP	Silica Alumina - 215 m2/g	Complete
SiO2_Al2O3.SMP	Silica-Alumina (example)	Complete
Titania - nitrogen.SMP	Titania	Complete
Y zeolite - nitrogen.SMP	Y-Zeolite Reference Mat a...	Complete
zsm5 - argon.SMP	ZSM-5 400:1 SiO2:Al2O3 a...	Complete

File name: *.SMP

(*.SMP)

2. Click **Export**.

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



☑ Include options
☐ Include target pressures
☐ Include quantity adsorbed versus relative pressure
☐ Include quantity dosed, absolute pressure, elapsed time, free space, Psat measurements
☐ Include sample log
☑ Include labels

● Preview
○ Print Copies: 1
○ File: C:\MICROACTIVE\DATA\
File Type: Report System (*.rep)

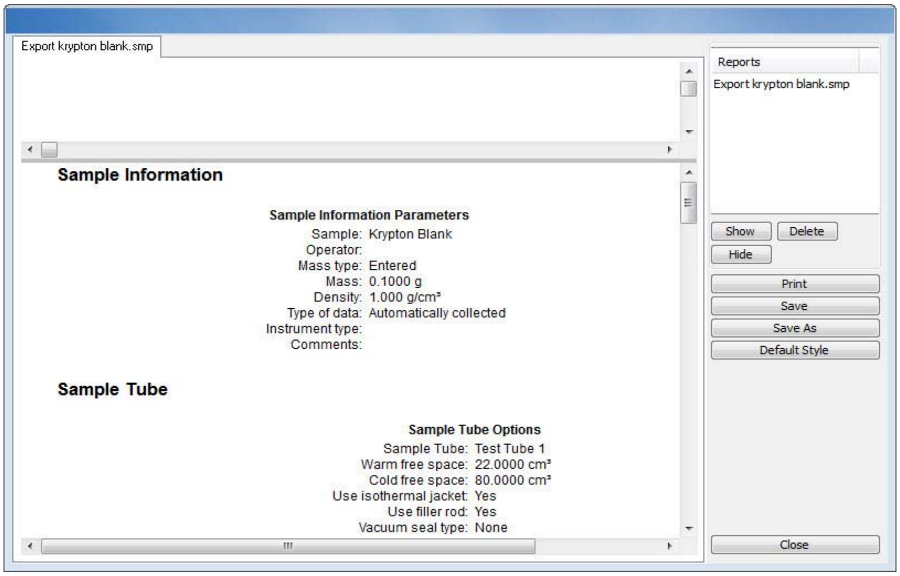
Types of data that can be included:

- 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 of the window:

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

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



Sample Information

Sample Information Parameters

Sample: Krypton Blank
Operator:
Mass type: Entered
Mass: 0.1000 g
Density: 1.000 g/cm^3
Type of data: Automatically collected
Instrument type:
Comments:

Sample Tube

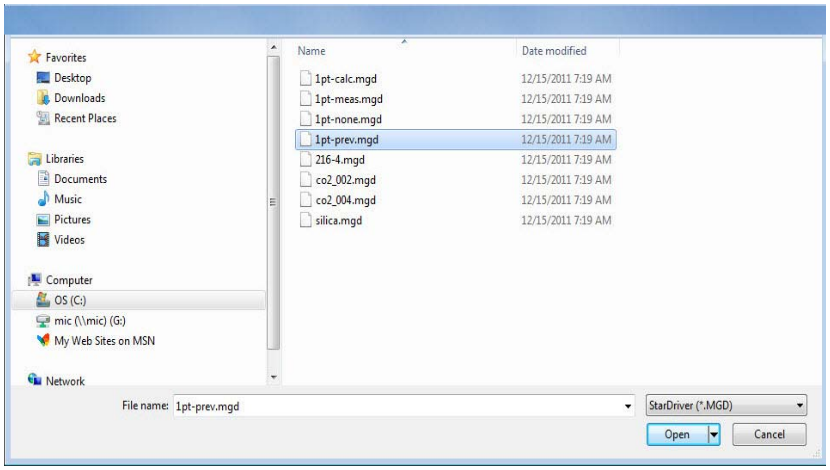
Sample Tube Options

Sample Tube: Test Tube 1
Warm free space: 22.0000 cm^3
Cold free space: 80.0000 cm^3
Use isothermal jacket: Yes
Use filler rod: Yes
Vacuum seal type: None

Converting Files

Converts StarDriver files (.MGD extension) to a sample information file with a .SMP file extension. Only those files with a .MGD file extension display in the **Name** column. Multiple files can be selected by holding down the **Ctrl** key on the keyboard while selecting the files.

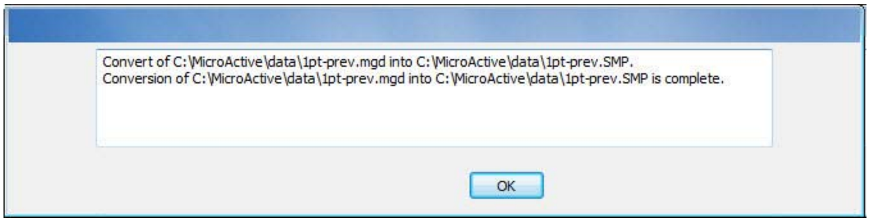
1. Go to ***File*** > ***Convert*** > ***[file]***.
2. From the **Name** column, select a file to convert. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files. To sort a column, click the column header.



Name	Date modified
1pt-calc.mgd	12/15/2011 7:19 AM
1pt-meas.mgd	12/15/2011 7:19 AM
1pt-none.mgd	12/15/2011 7:19 AM
1pt-prev.mgd	12/15/2011 7:19 AM
216-4.mgd	12/15/2011 7:19 AM
co2_002.mgd	12/15/2011 7:19 AM
co2_004.mgd	12/15/2011 7:19 AM
silica.mgd	12/15/2011 7:19 AM

File name: 1pt-prev.mgd

3. Click **Open.** The file is automatically converted to a .SMP file and the following window displays**.**



```
Convert of C:\MicroActive\data\1pt-prev.mgd into C:\MicroActive\data\1pt-prev.SMP.
Conversion of C:\MicroActive\data\1pt-prev.mgd into C:\MicroActive\data\1pt-prev.SMP is complete.
```

Click **OK** to close the window.

Generating Graph Overlays

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

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

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

Multiple Sample Overlays

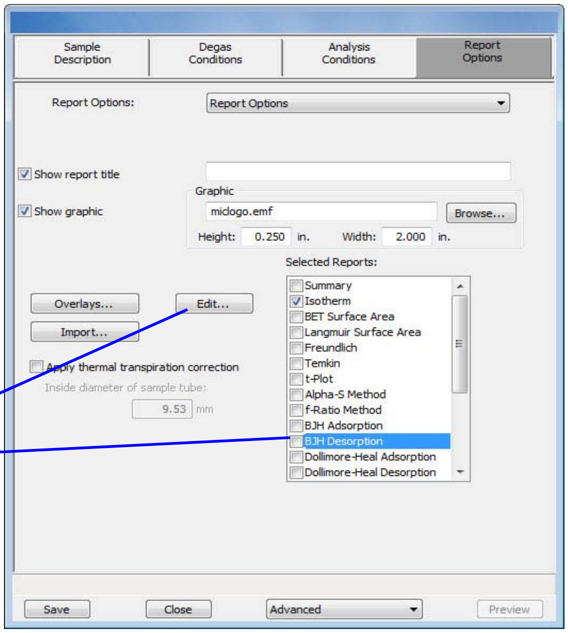
When working with an existing file, a copy of the file should be used rather than the original.

To overlay the same type of graph on multiple samples:

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

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

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



Report Options: Report Options

☑ Show report title

☑ Show graphic

Graphic: miclogo.emf

Height: 0.250 in. Width: 2.000 in.

Selected Reports:

☐ Summary
☑ Isotherm
☐ BET Surface Area
☐ Langmuir Surface Area
☐ Freundlich
☐ Temkin
☐ t-Plot
☐ Alpha-S Method
☐ f-Ratio Method
☐ BJH Adsorption
☐ BJH Desorption
☐ Dollimore-Heal Adsorption
☐ Dollimore-Heal Desorption

☐ Apply thermal transpiration correction

Inside diameter of sample tube: 9.53 mm

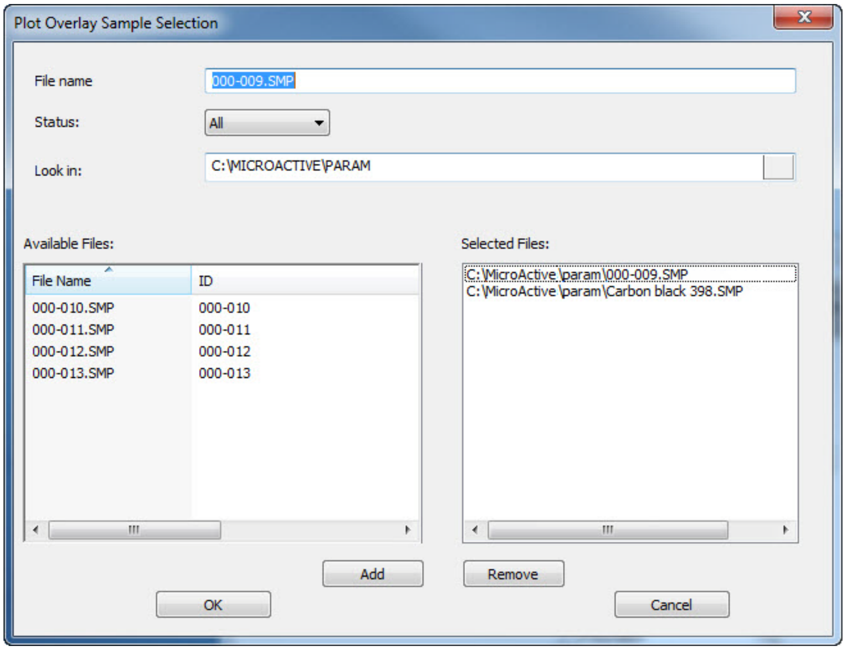
Advanced

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

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

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

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



File name: 000-009.SMP

Status: All

Look in: C:\MICROACTIVE\PARAM

Available Files:

File Name	ID
000-010.SMP	000-010
000-011.SMP	000-011
000-012.SMP	000-012
000-013.SMP	000-013

Selected Files:

C:\MicroActive\param\000-009.SMP
C:\MicroActive\param\Carbon black 398.SMP

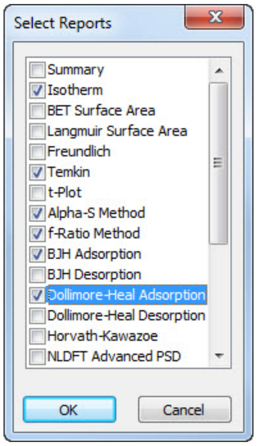
To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.

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

6. Click **OK**.

7. To view the report, click the **Preview** button on the **Sample Description** window.

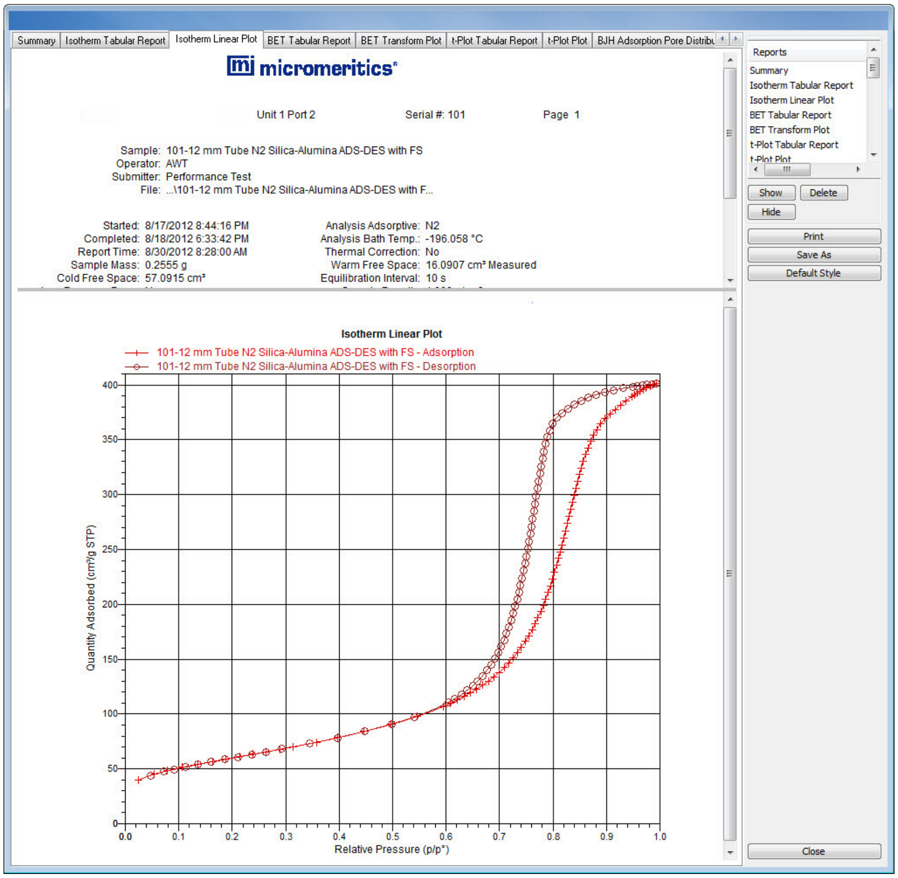
   If the sample file has been closed, go to ***Reports*** > ***Start Report***. Select the file used in the previous steps and click **Report**. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. Choose the report destination on the **Report Settings** window and click **OK**. If only one file was selected as an overlay, the **Select Reports** window displays. Verify the reports to generate and add or remove reports as necessary. Click **OK**. If multiple files were selected, the **Selected Reports** window will not display.



Select Reports

☐ Summary
☑ Isotherm
☐ BET Surface Area
☐ Langmuir Surface Area
☐ Freundlich
☑ Temkin
☐ t-Plot
☑ Alpha-S Method
☑ f-Ratio Method
☑ BJH Adsorption
☐ BJH Desorption
☑ Dollimore-Heal Adsorption
☐ Dollimore-Heal Desorption
☐ Horvath-Kawazoe
☐ NLDFT Advanced PSD

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



Unit 1 Port 2 Serial #: 101 Page 1

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 8:28:00 AM
Sample Mass: 0.2555 g
Cold Free Space: 57.0915 cm³

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

Multiple Graph Overlays

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

Multiple graph overlays can only be generated for:

- BJH Adsorption/Desorption
- Dollimore-Heal Adsorption/Desorption
- Horvath-Kawazoe
- DFT Pore Size/Surface Energy
- M-P Method

ASCII text file format rules

- The header must consist of one line to include title, two unit specifications, and distribution type:
  - Accepted pore dimension units are: A, nm, um
  - Accepted pore volume units are: cm3/g 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 ads (A, cm3/g, cumulative)
456.657          0.0133559
444.847          0.0546427
429.168          0.0869924
425.419          0.119721
419.629          0.132681
360.634          0.156611
340.859          0.197672
326.601          0.233092
```

To import the ASCII text file to generate graph overlays:

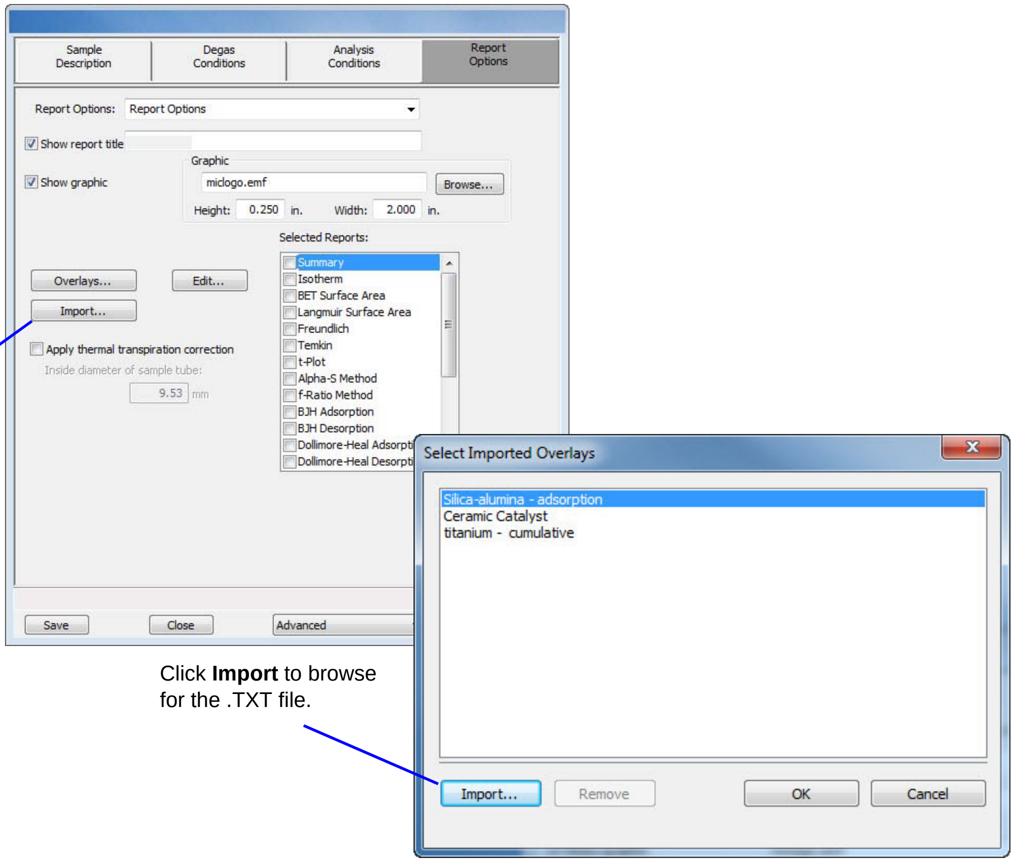
The following steps use BJH Adsorption as an example. Screen appearance will vary depending on the selected report.

1. Go to ***File*** > ***Open.*** Select a sample file to overlay graphs of other samples. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. Click **Open**.

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

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

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



Report Options: Report Options

☑ Show report title

☑ Show graphic

Graphic: miclogo.emf

Height: 0.250 in. Width: 2.000 in.

☐ Apply thermal transpiration correction

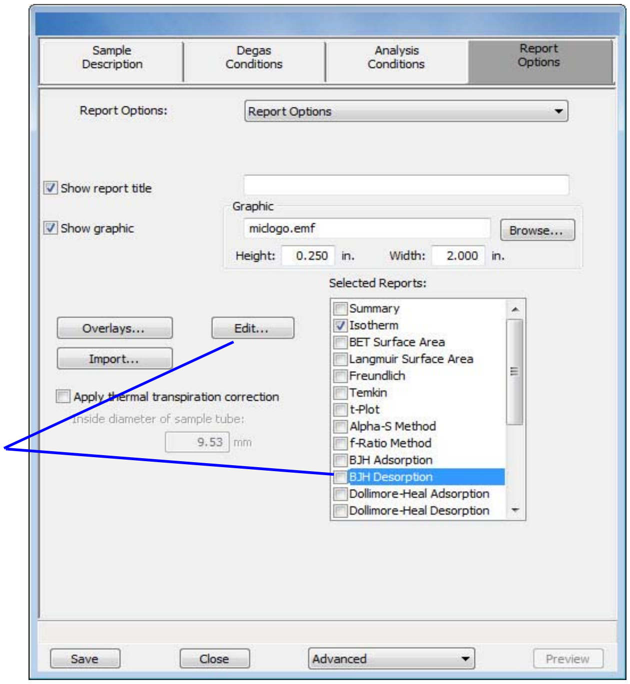
Inside diameter of sample tube: 9.53 mm

Silica-alumina - adsorption
Ceramic Catalyst
titanium - cumulative

Click **Import** to import a .TXT file for the overlay

Click **Import** to browse for the .TXT file.

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



Report Options: Report Options

☑ Show report title

☑ Show graphic

Graphic: miclogo.emf

Height: 0.250 in. Width: 2.000 in.

☐ Apply thermal transpiration correction

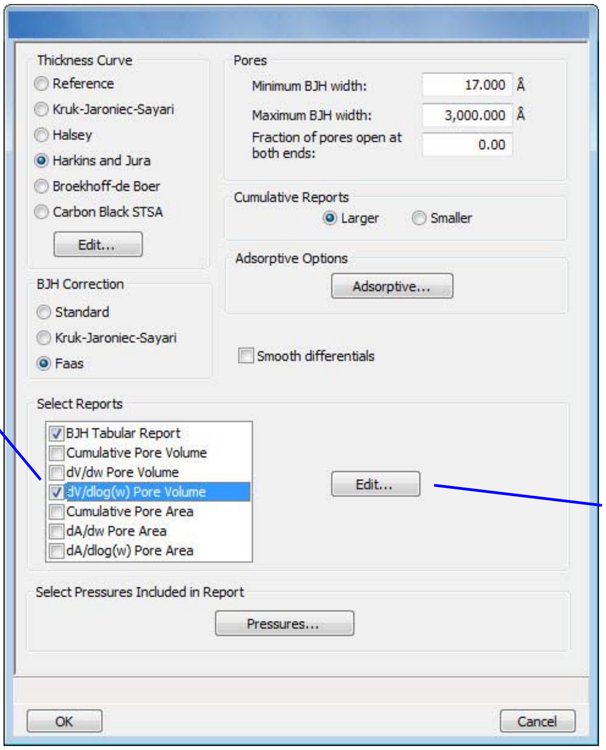
Inside diameter of sample tube: 9.53 mm

Selected Reports:

- ☐ Summary
- ☑ Isotherm
- ☐ BET Surface Area
- ☐ Langmuir Surface Area
- ☐ Freundlich
- ☐ Temkin
- ☐ t-Plot
- ☐ Alpha-S Method
- ☐ f-Ratio Method
- ☐ BJH Adsorption
- ☐ BJH Desorption
- ☐ Dollimore-Heal Adsorption
- ☐ Dollimore-Heal Desorption

Highlight the report then click **Edit**.

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



Thickness Curve

- ○ Reference
- ○ Kruk-Jaroniec-Sayari
- ○ Halsey
- ● Harkins and Jura
- ○ Broekhoff-de Boer
- ○ Carbon Black STSA

Pores

Minimum BJH width: 17.000 Å

Maximum BJH width: 3,000.000 Å

Fraction of pores open at both ends: 0.00

Cumulative Reports: ● Larger ○ Smaller

Adsorptive Options

BJH Correction

- ○ Standard
- ○ Kruk-Jaroniec-Sayari
- ● Faas

☐ Smooth differentials

Select Reports

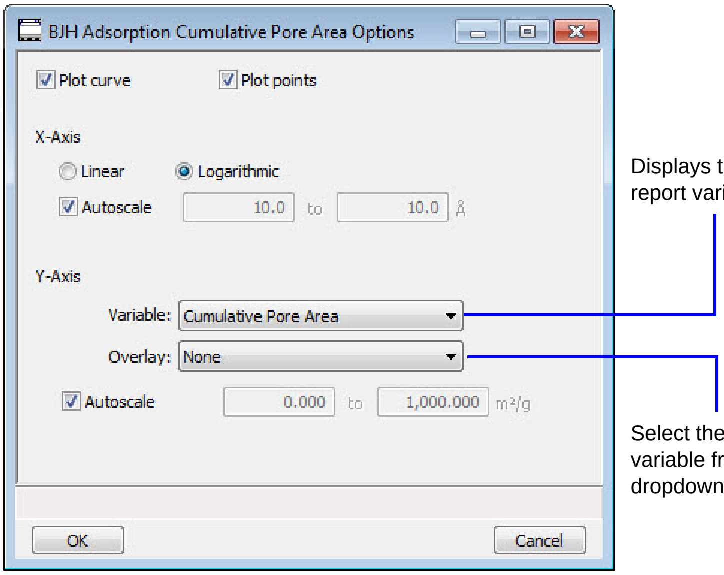
- ☑ BJH Tabular Report
- ☐ Cumulative Pore Volume
- ☐ dV/dw Pore Volume
- ☑ dV/dlog(w) Pore Volume
- ☐ Cumulative Pore Area
- ☐ dA/dw Pore Area
- ☐ dA/dlog(w) Pore Area

Select Pressures Included in Report

Select a sub-report

Click **Edit**

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



BJH Adsorption Cumulative Pore Area Options

☑ Plot curve ☑ Plot points

X-Axis

○ Linear ● Logarithmic

☑ Autoscale 10.0 to 10.0 Å

Y-Axis

Variable: Cumulative Pore Area

Overlay: None

☑ Autoscale 0.000 to 1,000.000 m²/g

Displays the first report variable

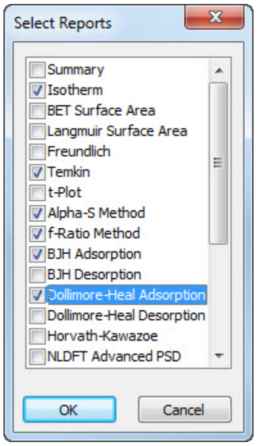
Select the second variable from the dropdown list

6. Click **OK** again to return to the **Report Options** tab.

7. Click **Save** to save the selections.

8. To view the report, click the **Preview** button on the **Sample Description** window.

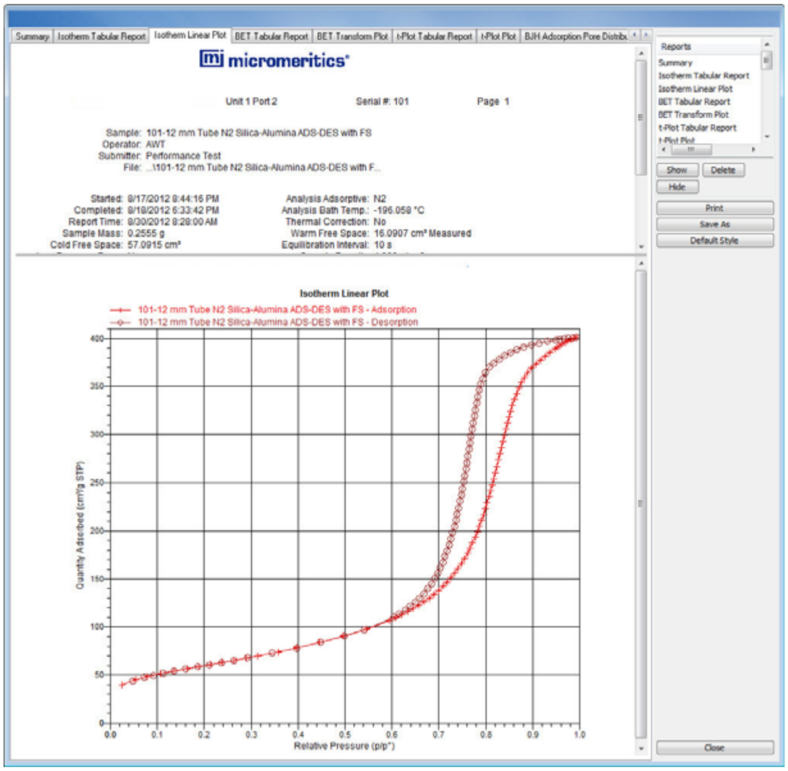
   If the sample file has been closed, go to ***Reports*** > ***Start Report***. Select the file used in the previous steps and click **Report**. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. Choose the report destination on the **Report Settings** window and click **OK**. If only one file was selected as an overlay, the **Select Reports** window displays. Verify the reports to generate and add or remove reports as necessary. Click **OK**. If multiple files were selected, the **Selected Reports** window will not display.



Select Reports

☐ Summary
☑ Isotherm
☐ BET Surface Area
☐ Langmuir Surface Area
☐ Freundlich
☑ Temkin
☐ t-Plot
☑ Alpha-S Method
☑ f-Ratio Method
☑ BJH Adsorption
☐ BJH Desorption
☑ Dollimore-Heal Adsorption
☐ Dollimore-Heal Desorption
☐ Horvath-Kawazoe
☐ NLDFT Advanced PSD

9. The report screen displays with tabs across the top. Click each tab to view the reports. Refer to **Report Tool Bar**, page **5-17**.



Unit 1 Port 2 Serial #: 101 Page 1

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...

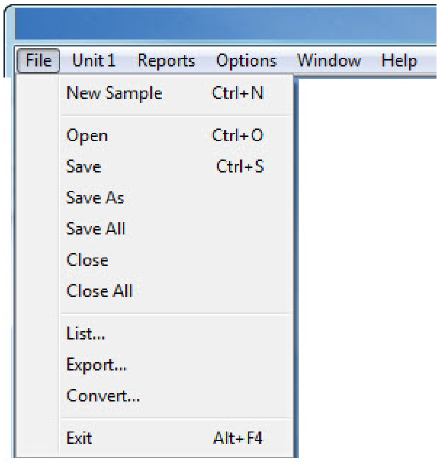
Started: 8/17/2012 8:44:16 PM
Completed: 8/18/2012 6:33:42 PM
Report Time: 8/30/2012 8:28:00 AM
Sample Mass: 0.2555 g
Cold Free Space: 57.0915 cm³

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

3. FILE MENU

Introduction

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



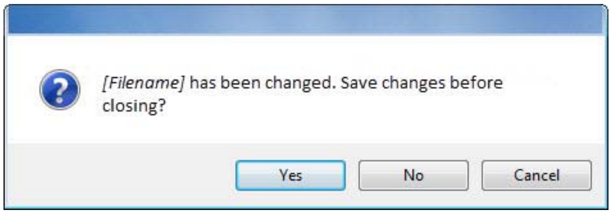
Common field and button descriptions are listed in a Common table at the beginning of their respective chapters. Field and button descriptions not listed in the Common table are listed in their appropriate heading.

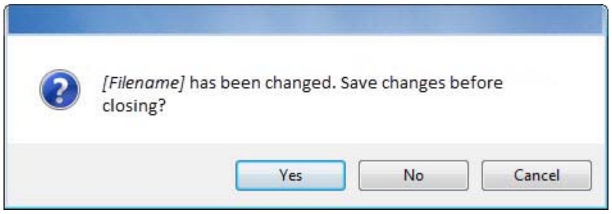
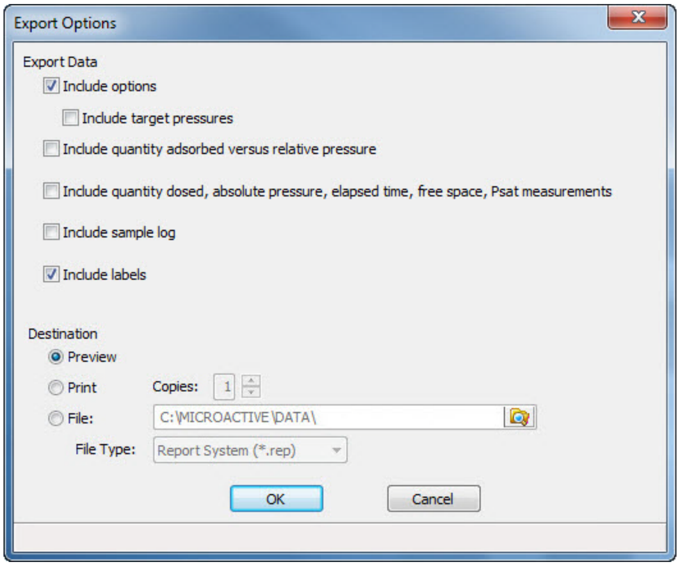
Refer to the Appendix section of this manual for further details on report calculations (CALCULATIONS, page C-1**), free space correction (**FREE SPACE, page D-1**), and DFT models (**DFT MODELS, page F-1**).**

Common Fields and Buttons - File Menu Options

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

Field or Button	Description
Autoscale *checkbox*	When enabled on report parameters screens, allows the x- and y-axes to be scaled automatically. **Autoscale** means that the x- and y- ranges will be set so that all the data is shown. If **Autoscale** is not selected, the entered range is used.
Axis Range	On report parameters screens, the **From / To** fields are enabled when **Autoscale** options are not selected. Enter the starting and ending values for the x- and/or y-axes.

Field or Button	Description *(continued)*
***Browse** button or icon*	Click to search for a file. Select a file from either the **Name** column or from the library and click **Open** or double click the file name to open (or import) the file.
***Cancel** button*	Cancels any changes made to the screen.
Close	Closes the active window. If changes were made to the file and not yet saved, a prompt displays providing the option to save the file.
Close All	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.  *[Filename]* has been changed. Save changes before closing?
***Delete** button*	When working with report parameters screens, click **Delete** to remove the selected report. Deleted reports will have to be regenerated if deleted in error.
***Destination** group box*	• **Preview** - sends the file to the screen. Click **Print** on the report screen to send the file to the printer. • **Print** - sends the file to the default printer. • **Copies** - select the number of copies to print. This field is only enabled when **Print** is selected. • **File** - saves the report as a file. Click the **Browse** icon to the right of the text field to select the directory where the new file will be stored. Enter the new file name in the **File name** text box. • **File Type** - use to save the new file with a .TXT, .XLS or file extension. This field is only enabled when **File** is selected. **.REP** (Report system) - saves the report in a format that can be opened within any MicroActive program. **.TXT** (ASCII text) - saves the report as an ASCII text file. **.XLS** (Spreadsheet file) - saves the report in a spreadsheet format

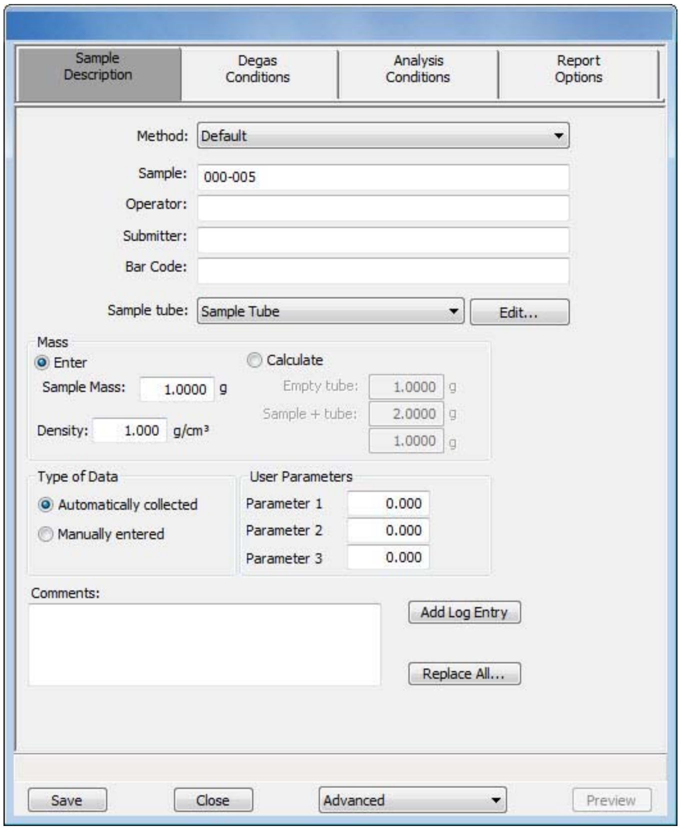
Field or Button	Description *(continued)*
***Edit** button*	When working with report parameters screens, highlight the item in the **Selected Reports** list box and click **Edit** to modify report details.
Exit	Exits the program. If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the program without saving the changes.  *[Filename]* has been changed. Save changes before closing? • **Yes** - saves the changes, then closes the window • **No -** closes the window without saving the changes • **Cancel -** cancels the **Close** command
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.  Export Options Export Data ☑ Include options ☐ Include target pressures ☐ Include quantity adsorbed versus relative pressure ☐ Include quantity dosed, absolute pressure, elapsed time, free space, Psat measurements ☐ Include sample log ☑ Include labels Destination ● Preview ○ Print Copies: 1 ○ File: C:\MICROACTIVE\DATA\ File Type: Report System (*.rep)
***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. Multiple files can be selected by holding down the **Ctrl** key on the keyboard while selecting multiple files.

Field or Button	**Description *(continued)***
***From / To** text boxes*	When working with report parameters screens, 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.
***OK** button*	Click to save and close the active window.
***Open** button*	Click to open the selected file (or double-click the file name in the **Name** column to open the file).
***Preview** button*	Click to preview predefined reports. Click the tabs across the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled. Refer to **Report Tool Bar**, page **5-17**.
***Print** button*	Sends the report to the selected destination (screen, printer or file).
Remove	Removes an item from a list.
***Replace** button*	Click to select another file where the values will replace the current file's values.
Save	Saves the active window under the current file name.
Save All	Saves all active windows under the current file names.
Save As	• Saves a file in the active window under a different file name. • Saves a subset (parameter) of the sample file in the active window as a standalone parameter file. For example, to create a standalone parameter file of the analysis conditions portion of the active sample file, go to ***File > Save As***, select the **Analysis Conditions** folder in the library and enter a file name in the **File name** field. Click **Save**.
***Table** buttons*	• **Insert** - inserts one row above the selected row. • **Delete** - deletes the selected row. • **Clear** - clears all table entries and displays only one default value. • **Append** - inserts one row at the end of the table.

Field or Button	Description *(continued)*
View Instrument Log	For use by a Service Technician. Operators should use ***Unit [n] > Show Instrument Log***. Refer to **Show Instrument Log**, page **4-19**.

New Sample

Provides the option to create a new sample file or parameter file. Refer to **Sample Information Files**, page **3-9**.



Method: Default
Sample: 000-005
Operator:
Submitter:
Bar Code:
Sample tube: Sample Tube

Mass
● Enter
Sample Mass: 1.0000 g
Density: 1.000 g/cm³
○ Calculate
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g

Type of Data
● Automatically collected
○ Manually entered

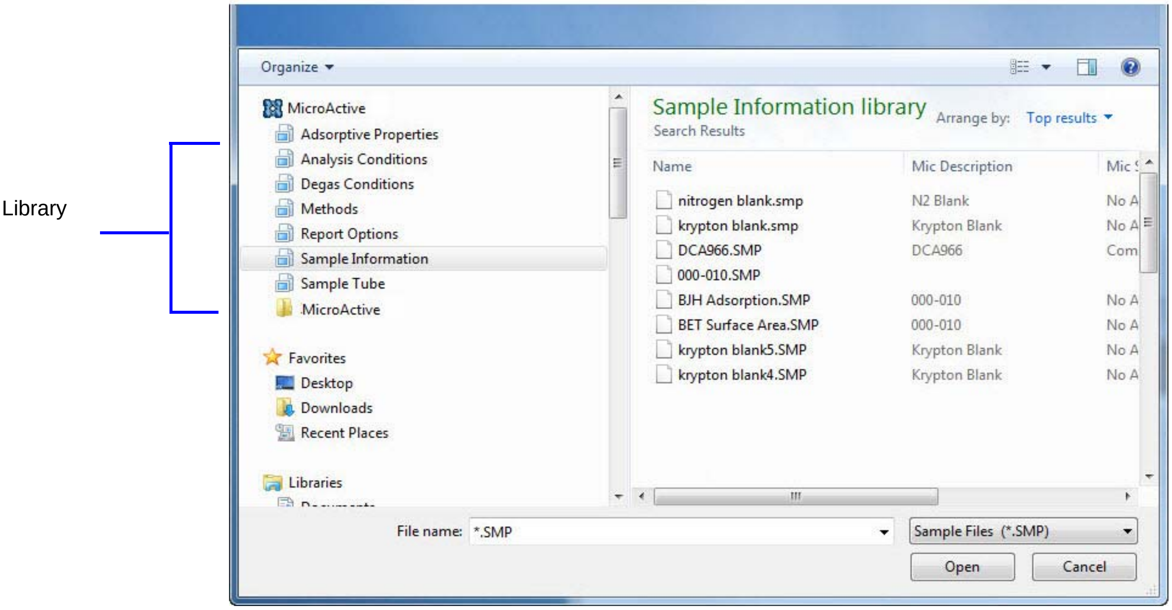
User Parameters
Parameter 1 0.000
Parameter 2 0.000
Parameter 3 0.000

Comments:

Advanced

Open

Provides access to the library. Refer to **Manage Libraries**, page **6-3** for information on managing libraries.



Name	Mic Description	Mic S
nitrogen blank.smp	N2 Blank	No A
krypton blank.smp	Krypton Blank	No A
DCA966.SMP	DCA966	Com
000-010.SMP		
BJH Adsorption.SMP	000-010	No A
BET Surface Area.SMP	000-010	No A
krypton blank5.SMP	Krypton Blank	No A
krypton blank4.SMP	Krypton Blank	No A

File name: *.SMP

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

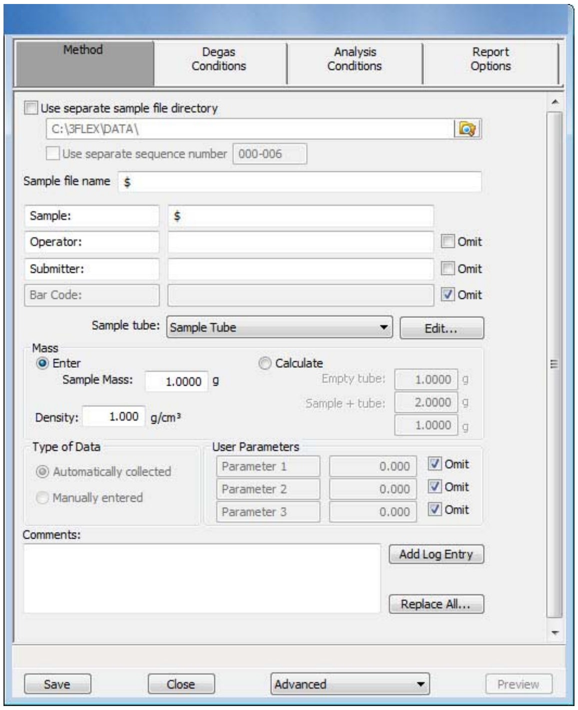
*.ADP	Adsorptive Properties
*.ANC	Analysis Conditions
*.DEG	Degas Conditions
*.FPI	Fluid Properties
*.RPO	Report Options
*.STB	Sample Tube Properties
*.MTH	Method

Create a New Method

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

The default Method that comes with the analyzer program can be modified in ***Options*** > ***Default Method***. Refer to **Editing the Default Method**, page **2-7** for additional information on modifying the default method.

1. Go to ***File*** > ***Open*** and create a new Sample Information file.
2. Go to ***File*** > ***Save As***. Enter a new file name for the method and select Method (*.MTH) as the file type.
3. On the **Method** tab, if files created using this Method are to be saved in a file directory other than the default, select the **Use separate sample file directory** checkbox and click the **Browse** icon to select a directory. The **Browse** icon is enabled only when the **Use separate sample file directory** checkbox is selected. Select the new directory then click **OK** on the **Browse for Folder** window.



☐ Use separate sample file directory
C:\3FLEX\DATA\
☐ Use separate sequence number 000-006
Sample file name $
Sample: $
Operator: ☐ Omit
Submitter: ☐ Omit
Bar Code: ☑ Omit
Sample tube: Sample Tube
Mass
● Enter
Sample Mass: 1.0000 g
○ Calculate
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g
Density: 1.000 g/cm³
Type of Data
● Automatically collected
○ Manually entered
User Parameters
Parameter 1 0.000 ☑ Omit
Parameter 2 0.000 ☑ Omit
Parameter 3 0.000 ☑ Omit
Comments:
Advanced

4. If the file sequence numbers for this Method will differ from other Methods, select the **Use separate sequence number** checkbox and enter the new sequence number in the text box.
5. In the **Sample file name** text box, enter a default form for file names. The $ symbol is required and represents the position of the sequence number in the file name.

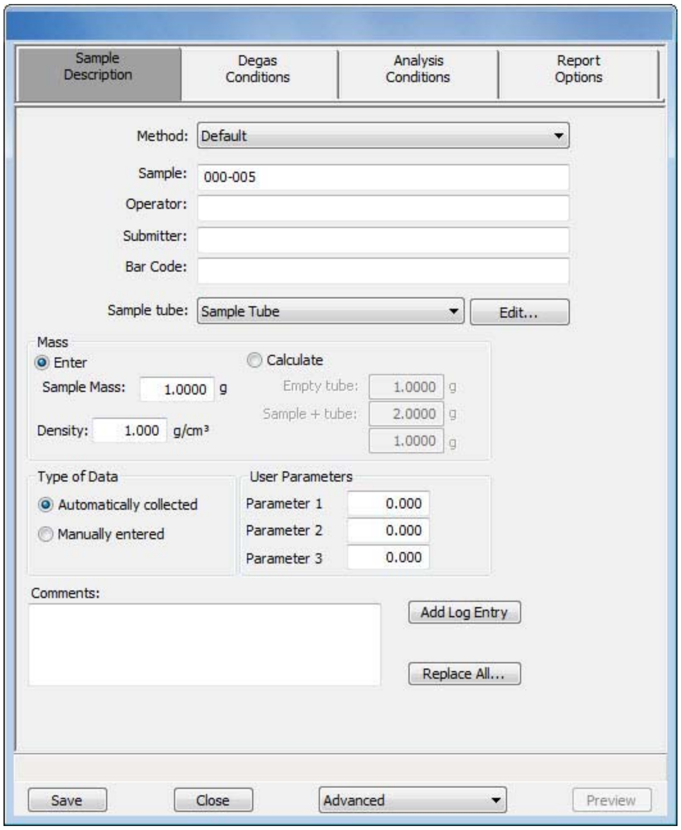
6. Refer to **Sample Information Files**, page **3-9** for details on completing the remainder of this window. The information entered into the **Sample** field becomes a part of the **Method** dropdown list when creating new sample files. The **Type of Data** group box is disabled when creating a Method.

7. Click **Save**. The **Save as Sample Information File** window displays. Select **Methods** in the library and enter a file name for the Method in the **File name** text box.

8. Click **Save**.

Sample Information Files

File > New Sample



Method: Default
Sample: 000-005
Operator:
Submitter:
Bar Code:
Sample tube: Sample Tube

Mass
● Enter
Sample Mass: 1.0000 g
Density: 1.000 g/cm³
○ Calculate
Empty tube: 1.0000 g
Sample + tube: 2.0000 g
1.0000 g

Type of Data
● Automatically collected
○ Manually entered

User Parameters
Parameter 1 0.000
Parameter 2 0.000
Parameter 3 0.000

Comments:

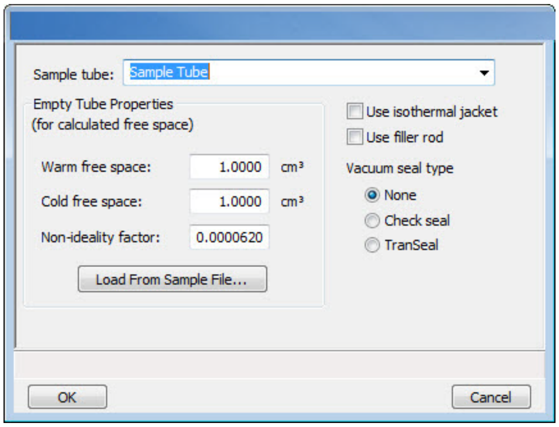
Advanced

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

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

Specify or change the default format by selecting ***Options > Option Presentation*** **or select** ***Basic / Advanced*** **from the dropdown list at the bottom of the window. Refer to** Editing the Default Method, page 2-7 **for a description of the Advanced, Basic, and Restricted formats.**

Field or Button	Description
Method *dropdown list*	Select a method to use for the sample information file. To select an ASTM method, click **Browse** and navigate to **C:\3Flex\Data\Examples**. Select an ASTM method from the list.
Sample *text box*	Enter a description of the sample.
Operator / Submitter *text boxes*	Enter identification information in the respective text boxes. Some text boxes may have been renamed or may not display if modified in ***Options*** > ***Default Methods***.
Bar Code *text box*	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.

Field or Button	Description *(continued)*
Sample Tube *dropdown list*	• **Edit** button - click to edit the sample tube parameters. To save the sample tube parameters as a file, go to ***File*** > ***Save As***. Select .STB as the **File Type** and enter a **File name**.  Sample tube: Sample Tube Empty Tube Properties (for calculated free space) Warm free space: 1.0000 cm^3 Cold free space: 1.0000 cm^3 Non-ideality factor: 0.0000620 ☐ Use isothermal jacket ☐ Use filler rod Vacuum seal type ● None ○ Check seal ○ TranSeal • **Description** - enter a description of the sample tube, such as the tube size. • **Empty Tube Properties** group box - use for calculated free space, enter the values for the following or use the **Load from Sample File** button to import this information from a sample information file: – **Warm free space** - empty sample tube gas capacity measured at room temperature. – **Cold free space** - empty sample tube gas capacity measured with the dewar raised. – **Non-ideality factor** - factor used to compensate for the forces of attraction between molecules in a real gas. This value is used for a calculated free space. • **Use isothermal jacket** checkbox - select if an isothermal jacket is to be used. An isothermal jacket maintains a constant temperature profile along the sample tube stem during an extended analysis of more than 1 or 2 hours. • **Use filler rod** checkbox - select if a filler rod is to be used in the sample tube. A filler rod reduces the stem free space volume resulting in reduction of free space error. • **Vacuum seal type** option - select the seal type to be used.

Field or Button	Description *(continued)*
Mass *group box*	If mass = 1, the reported surface area equals the total surface area but it is always shown as m^2. 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 and/or density. Both of these values may be edited at the time of 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: $\text{Mass}_{\text{sample}} = \text{Mass}_{\text{sample+tube}} - \text{Mass}_{\text{tube}}$ • **Density** - value is used for the Calculated free space method only. Use **0.000** for a blank analysis.
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 manually enter data collected from another source. If **Manually entered** is selected, the Isotherm Report becomes available in the Basic/Advanced dropdown list allowing the pasting or importing of data into the file. Refer to **Manually Entering Isotherm Data in a Sample File**, page **2-27**.
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 **Sample Description** window, in ***Reports > SPC Report Options***, and the **Summary Report**. Choose to hide or display these fields or modify the default alphanumeric field labels in ***Options > Default Method***.
Comments *text box*	Enter comments about the sample or analysis. Comments display in the report header.
Add Log Entry *button*	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.

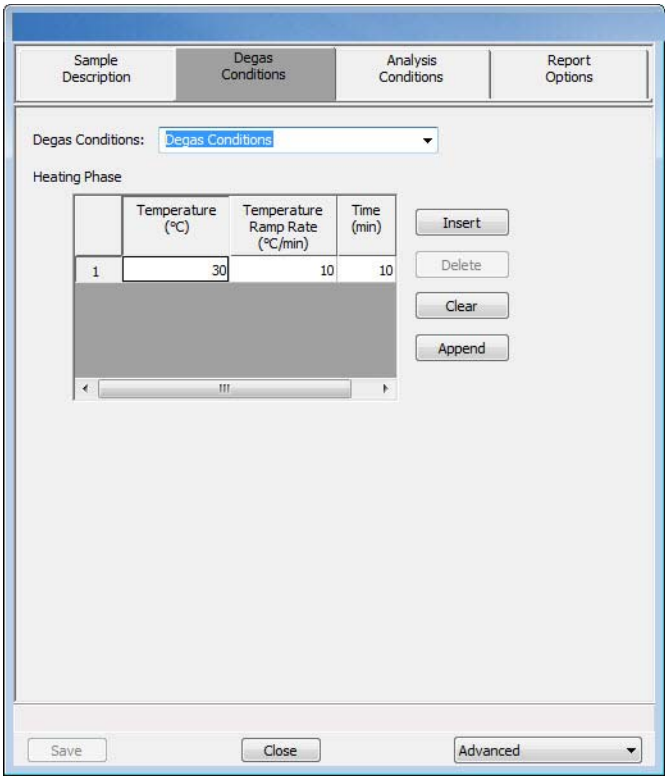
Field or Button	Description *(continued)*
Replace All *button*	Click to select another .SMP file where the values will replace all values for the active Sample Information file. The original file will remain unchanged.
Close *button* ***Preview*** *button* ***Save*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

Degas Conditions Files

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

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

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



Degas Conditions: Degas Conditions

Heating Phase

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

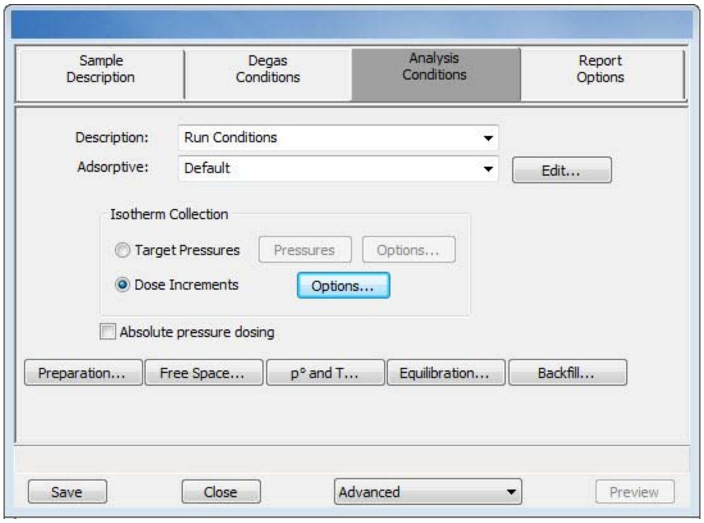
Field or Button	Description
Degas Conditions *dropdown list*	Use to browse for a .DEG file that contains degas condition parameters to be used in the analysis.
Heating Phase *table*	Enter up to five stages of degas conditions. • **Temperature -** soaking temperature with flowing gas. • **Temperature Ramp Rate -** rate at which the temperature is to change when advancing to the soak temperature. • **Time -** amount of time to soak the sample.

Field or Button	**Description *(continued)***
***Close** button* ***Preview** button* ***Save** button* ***Table** buttons*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

Analysis Conditions Files

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

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



Description: Run Conditions

Adsorptive: Default

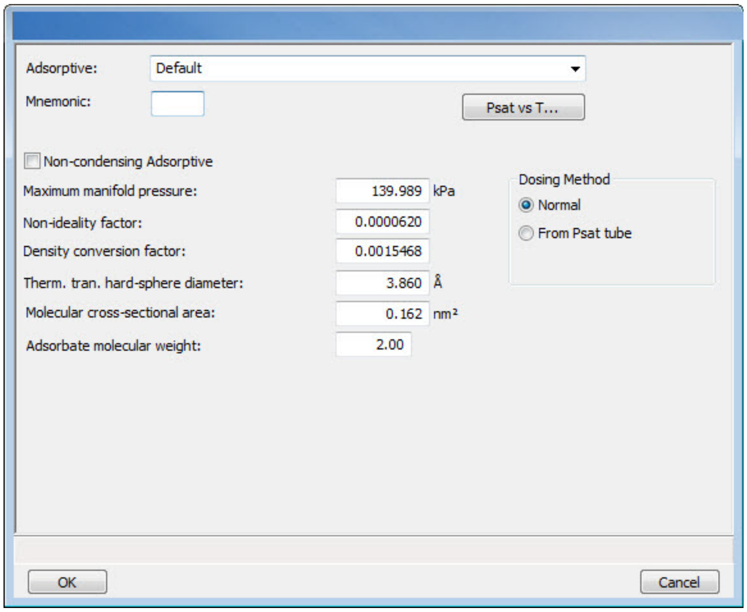
Isotherm Collection

○ Target Pressures

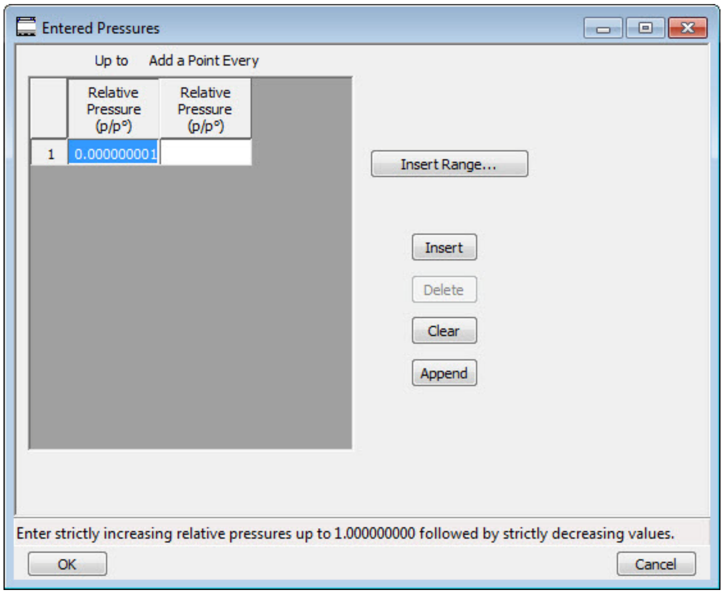
● Dose Increments

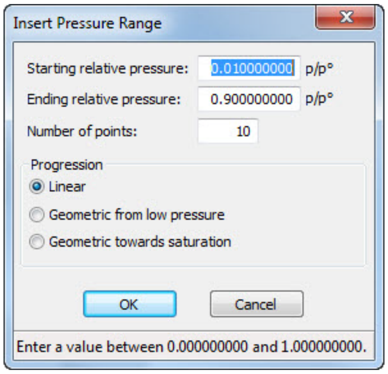
☐ Absolute pressure dosing

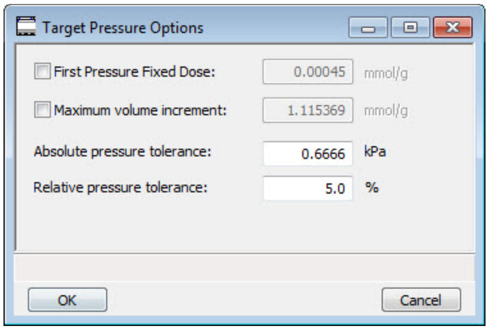
Advanced

Field or Button	Description
Description *dropdown list*	Use to browse for a .ANC file that contains analysis condition parameters to be used in the analysis.
Adsorptive *dropdown list*	Displays a list of defined gases. Select the adsorptive to be used for the analysis. Click the **Edit** button to edit the adsorptive properties.  Adsorptive: Default Mnemonic: ☐ Non-condensing Adsorptive Maximum manifold pressure: 139.989 kPa Non-ideality factor: 0.0000620 Density conversion factor: 0.0015468 Therm. tran. hard-sphere diameter: 3.860 Å Molecular cross-sectional area: 0.162 nm² Adsorbate molecular weight: 2.00 Dosing Method ● Normal ○ From Psat tube

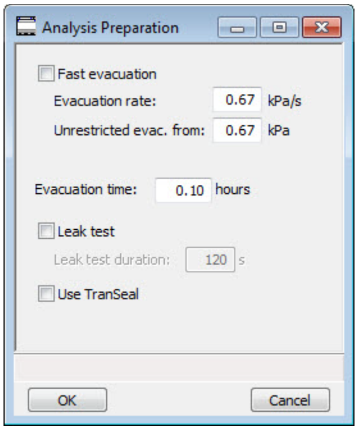
Field or Button	Description *(continued)*
Adsorptive Properties *window*	• **Adsorptive** text box - defaults to the adsorptive listed on the previous window. • **Mnemonic** - enter the mnemonic name for the adsorptive. • **Psat vs T** - table for inserting saturation pressure and temperature. • **Non-condensing Adsorptive** checkbox - used for a non-condensing gas. The **Absolute pressure dosing** option on the Analysis Conditions tab must also be selected when **Non-condensing Adsorptive** is selected on this window. When selected, the **Density conversion factor** field and the **Psat vs T** button are disabled. • **Maximum manifold pressure** - the highest pressure that the manifold will be dosed to. To avoid damage to the instrument, this number is limited to 925 mmHg. Low pressure sources, such as vapors, will require lower numbers. • **Non-ideality factor** - enter a factor to compensate for the forces of attraction between molecules in a real gas. This value is used for a calculated free space. • **Density conversion factor** - enter the density conversion factor which is determined by obtaining the ratio of the gas density (STP) to the liquid volume. • **Therm. tran. hard-sphere diameter** - an estimate of molecular size used in calculating the thermal transpiration correction. • **Molecular cross-sectional area** - the area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations. • **Adsorbate molecular weight** - the molecular weight is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot. • **Dosing Method** group box - select the dosing method to be used. – **Normal** - select for standard and high throughput analyses. Dose from a pressurized tank of gas attached to a gas inlet port. – **From Psat tube -** select if the Psat tube is filled with condensed adsorptive and dosed from the Psat tube. Select this option if using Krypton.

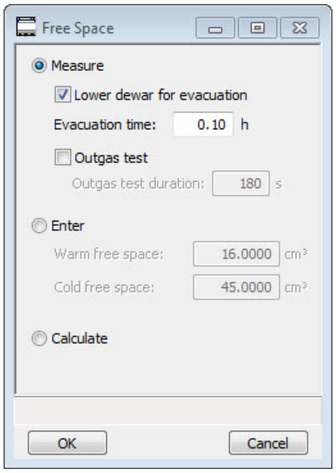
Field or Button	Description *(continued)*
Isotherm Collection *group box*	Specify how isotherms are to be collected: Target Pressures or Dose Increments.
Target Pressures *option*	Select to have isotherms collected by specifying target pressures. Select this option to enable the **Pressures** and **Options** buttons. • ***Pressures*** *button* - click to edit the Entered Pressures table. Click **Insert Range** to manually enter the pressures.  Enter strictly increasing relative pressures up to 1.000000000 followed by strictly decreasing values. A pressure table consists of an **Up to** column of relative pressure points at which isotherm data are to be collected. An optional pressure increment can be entered in the **Add a Point Every** column, which will cause additional points to be collected at intervals of the pressure increment up to the relative pressure specified in the **Up to** column. The relative pressures points may span the entire range of 0.00000001 to 0.995 p/po. There must be one adsorption branch (strictly increasing pressures) followed optionally by one desorption branch (strictly decreasing pressures).

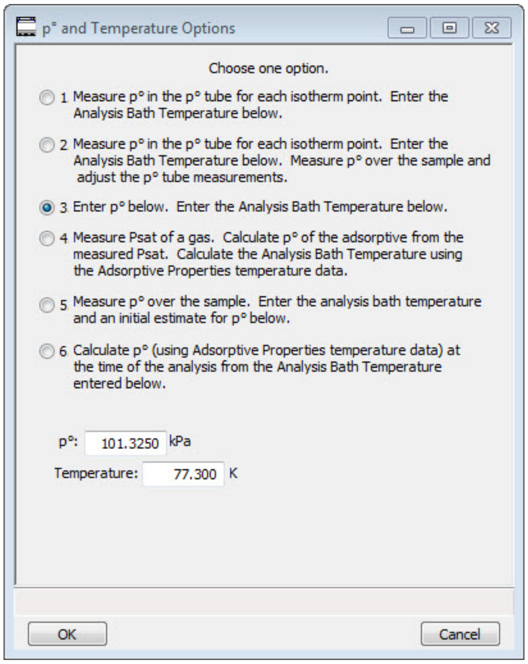
Field or Button	Description *(continued)*
Target Pressures *option (continued)*	Several analysis conditions files containing complete pressure tables are included with the analysis program. When using these files, it is recommended to use the ***File > Save As*** option to save the file as a different name and edit the new file. • ***Insert Range*** *button* - the pressure table for automatically collected data includes the pressure points for data collection and (when the **Use calculation assignments** checkbox is selected) identifies calculations through which the data are processed. Click the **Insert Range** button to enter 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: 0.010000000 p/p° Ending relative pressure: 0.900000000 p/p° Number of points: 10 Progression ● Linear ○ Geometric from low pressure ○ Geometric towards saturation Enter a value between 0.000000000 and 1.000000000. **Linear** - select to insert evenly spaced points into the table. **Geometric from low pressure -** select to insert geometrically spaced points from the low pressure range. For example, to insert 5 points with a 0.01 starting pressure and a 0.16 ending pressure, the following points are inserted into the table: 0.01 0.02 0.04 0.08 0.16 **Geometric towards saturation** - select to insert geometrically spaced points from the saturation pressure. For example, to insert 5 points with a 0.99 starting pressure and a 0.84 ending pressure, the following points are inserted into the table: 0.84 0.92 0.96 0.98 0.99

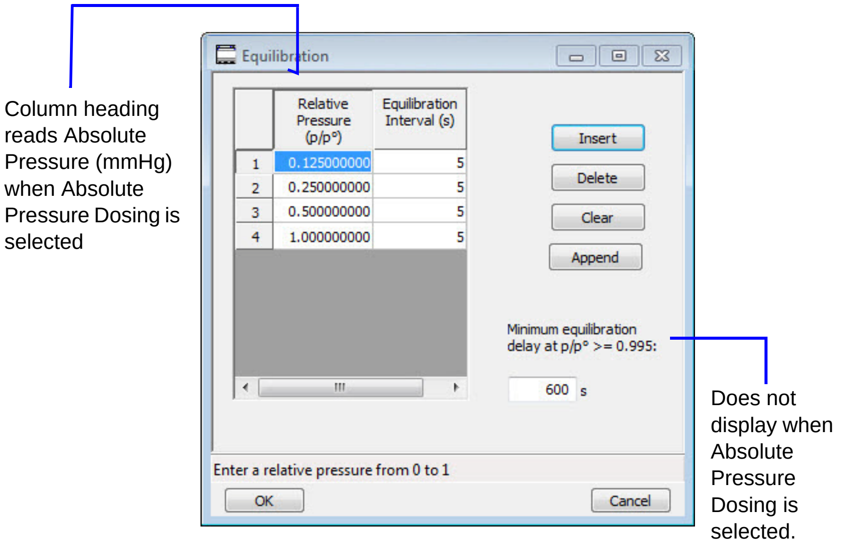
Field or Button	Description *(continued)*
Target Pressures Options *button*	 ☐ First Pressure Fixed Dose: 0.00045 mmol/g ☐ Maximum volume increment: 1.115369 mmol/g Absolute pressure tolerance: 0.6666 kPa Relative pressure tolerance: 5.0 % • **First Pressure Fixed Dose -** select to have the sample dosed with a specified amount of gas until the first pressure point is reached. This initial dosing quickly meets the adsorptive demand of the sample. The first point on the pressure table is the threshold value. Once this first pressure point is reached, points are equilibrated and recorded in accordance with the specified pressure table. Use this mode if performing a standard nitrogen analysis of microporous or highly mesoporous materials such as catalysts. If the first pressure table point is low, and the gas uptake of the sample is expected to be high, choosing this mode can shorten the time required to reach the first point on the pressure table. • **Maximum volume increment -** select to specify when additional data points are to be collected between target pressures in regions of high adsorption. When the maximum increment has been adsorbed since the last collected data point, another point is equilibrated and collected. When using this option, reaching pressure points exactly is not important; therefore, the tolerances should be set relatively large (10 mmHg and 10% or so) for proper functioning of the algorithm. The pressure table should also have several points scattered over the region of interest. During desorption, this field is treated as a maximum volume “decrement” value.

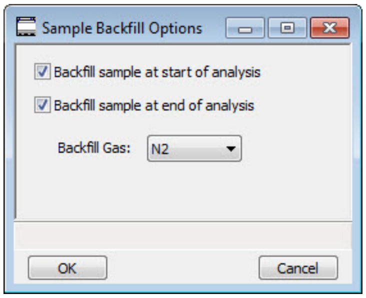
Field or Button	Description *(continued)*
***Target Pressures Options** button (continued)*	• **Absolute / Relative pressure tolerance -** values used to determine how close the actual pressure must be to each target pressure from the pressure table. At lower pressures, the relative tolerance value is less. At higher pressures, the absolute tolerance value is less. For example: **Experiment 1:** You have an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 40 mmHg; 5% of 40 mmHg is 2 mmHg. Since 2 mmHg (relative tolerance) is less than 5 mmHg (absolute tolerance), 2 mmHg is used. Therefore a minimum pressure of 38 mmHg (40 - 2) must be attained to collect data for a target pressure of 40 mmHg. **Experiment 2:** You have an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 200 mmHg; 5% of 200 mmHg is 10 mmHg. Since 5 mmHg (absolute tolerance) is less than 10 mmHg (relative tolerance), 5 mmHg is used. Therefore a minimum pressure of 195 mmHg (200 - 5) must be attained to collect data for a target pressure of 200 mmHg. Normally, surface area measurement points are widely spaced and the resulting measurement is not very sensitive to the precise location of points so wider tolerances may be used. Unnecessarily tight tolerances lengthen the analysis.
***Absolute pressure dosing** checkbox*	Use to specify pressure targets in mmHg, mbar, or kPa instead of relative pressure. This option is typically selected when using adsorptives at analysis conditions above the critical point of the gas; for example, 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.

Field or Button	Description *(continued)*
Preparation *button*	Use to enter analysis preparation details.  ☐ Fast evacuation Evacuation rate: 0.67 kPa/s Unrestricted evac. from: 0.67 kPa Evacuation time: 0.10 hours ☐ Leak test Leak test duration: 120 s ☐ Use TranSeal • **Fast Evacuation -** select if a fast evacuation is to be performed. – **Evacuation rate -** enter the rate for restricted evacuation. – **Unrestricted evac. from -** enter the pressure at which unrestricted evacuation is to begin. • **Evacuation time -** enter the length of time for preliminary evacuation which takes place prior to the free space measurement or sample analysis if free space is to be entered or calculated. The timer starts when the entered vacuum level is reached. • **Leak test -** select if a leak test is to be performed. • **Leak test duration -** enter the duration of the leak test. • **Use TranSeal -** select if using the TranSeal to transfer the sample from the preparation port to the analysis port under vacuum.

Field or Button	Description *(continued)*
Free Space *button*	Use to enter the type of free space measurement and if it is to be measured, entered or calculated.  ● Measure ☑ Lower dewar for evacuation Evacuation time: 0.10 h ☐ Outgas test Outgas test duration: 180 s ○ Enter Warm free space: 16.0000 cm^3 Cold free space: 45.0000 cm^3 ○ Calculate • **Measure -** select if the free space is to be measured. – **Lower dewar for evacuation -** select if the dewar is to be lowered for evacuation. – **Evacuation time -** if the dewar is to be lowered for evacuation, enter the length of time for evacuation after the free-space measurement. – **Outgas test -** select if an outgas test is to be performed before analysis. – **Outgas test duration -** if an outgas test is to be performed, enter the duration of the outgas test. • **Enter -** use to enter warm and cold free space manually and enter the amount in the text box. • **Calculate -** use to have the free space measurement calculated using the sample and tube parameters.

Field or Button	Description *(continued)*
p^o and T *button*	Click to select one option for obtaining the saturation pressure (Po) and analysis bath temperature. Each selected option presents a different set of parameters at the bottom of the screen.  Choose one option. ○ 1 Measure p° in the p° tube for each isotherm point. Enter the Analysis Bath Temperature below. ○ 2 Measure p° in the p° tube for each isotherm point. Enter the Analysis Bath Temperature below. Measure p° over the sample and adjust the p° tube measurements. ● 3 Enter p° below. Enter the Analysis Bath Temperature below. ○ 4 Measure Psat of a gas. Calculate p° of the adsorptive from the measured Psat. Calculate the Analysis Bath Temperature using the Adsorptive Properties temperature data. ○ 5 Measure p° over the sample. Enter the analysis bath temperature and an initial estimate for p° below. ○ 6 Calculate p° (using Adsorptive Properties temperature data) at the time of the analysis from the Analysis Bath Temperature entered below. p°: 101.3250 kPa Temperature: 77.300 K

Field or Button	**Description *(continued)***
***Equilibration** button*	Click to enter values for relative or absolute pressure.  Column heading reads Absolute Pressure (mmHg) when Absolute Pressure Dosing is selected Equilibration Relative Pressure (p/p°) · Equilibration Interval (s) 1 · 0.125000000 · 5 2 · 0.250000000 · 5 3 · 0.500000000 · 5 4 · 1.000000000 · 5 Minimum equilibration delay at p/p° >= 0.995: 600 s Enter a relative pressure from 0 to 1 Does not display when Absolute Pressure Dosing is selected. • **Relative Pressure (p/p°)** or **Absolute Pressure (mmHg) -** the pressure the equilibration interval will be applied. • **Equilibration Interval -** the number of seconds between successive pressure readings during equilibration. At least 11 pressure readings for each equilibration are required to obtain an accurate reading of the pressure changes. Long equilibration intervals tend to lengthen analyses, but improve data integrity. Short equilibration intervals produce a faster analysis but may reduce the accuracy of data. • **Minimum equilibration delay at p/p°> =0.995 -** the minimum number of seconds required before equilibration can occur for a relative pressure greater than or equal to 0.995. This field is not available if **Absolute pressure dosing** is selected on the **Analysis Conditions** tab.

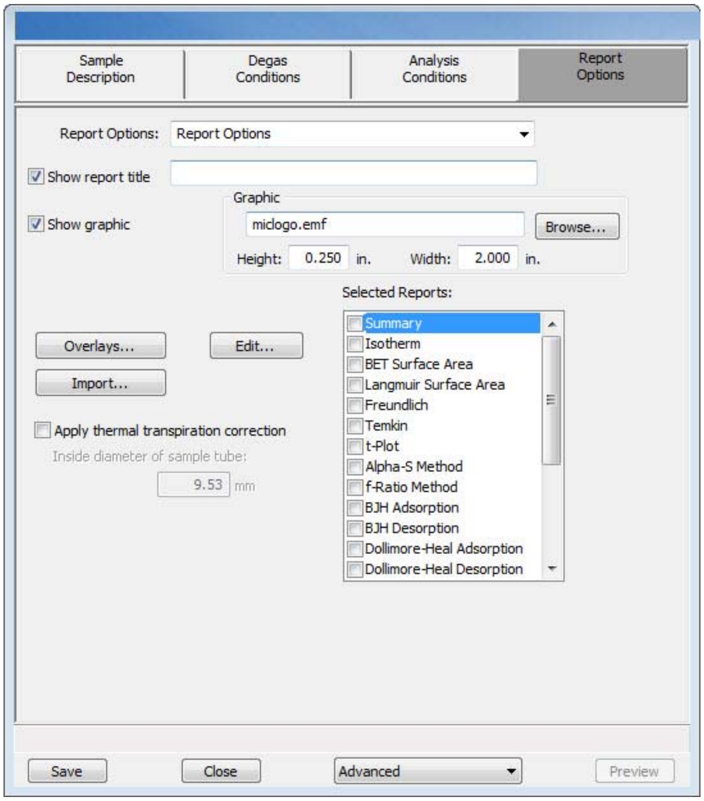
Field or Button	Description *(continued)*
Backfill *button*	Click to enter options for backfilling the sample tube. Indicate if the sample should be backfilled at the start and/or end of the analysis. Click the dropdown arrow and select the backfill gas to be used.  Sample Backfill Options ☑ Backfill sample at start of analysis ☑ Backfill sample at end of analysis Backfill Gas: N2
Cancel *button* ***Close*** *button* ***OK*** *button* ***Preview*** *button* ***Save*** *button* ***Table*** *buttons*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

Report Options Files

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

Use to specify report options for collected (from an analysis) or manually entered data. Report Options files also help in customizing report details such as axis scale, axis range, column headings, and components of thickness curve equations. Refer to **REPORTS MENU**, page **5-1**.

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



Report Options: Report Options

☑ Show report title

☑ Show graphic

Graphic: miclogo.emf

Height: 0.250 in. Width: 2.000 in.

☐ Apply thermal transpiration correction

Inside diameter of sample tube: 9.53 mm

Selected Reports:

- ☐ Summary
- ☐ Isotherm
- ☐ BET Surface Area
- ☐ Langmuir Surface Area
- ☐ Freundlich
- ☐ Temkin
- ☐ t-Plot
- ☐ Alpha-S Method
- ☐ f-Ratio Method
- ☐ BJH Adsorption
- ☐ BJH Desorption
- ☐ Dollimore-Heal Adsorption
- ☐ Dollimore-Heal Desorption

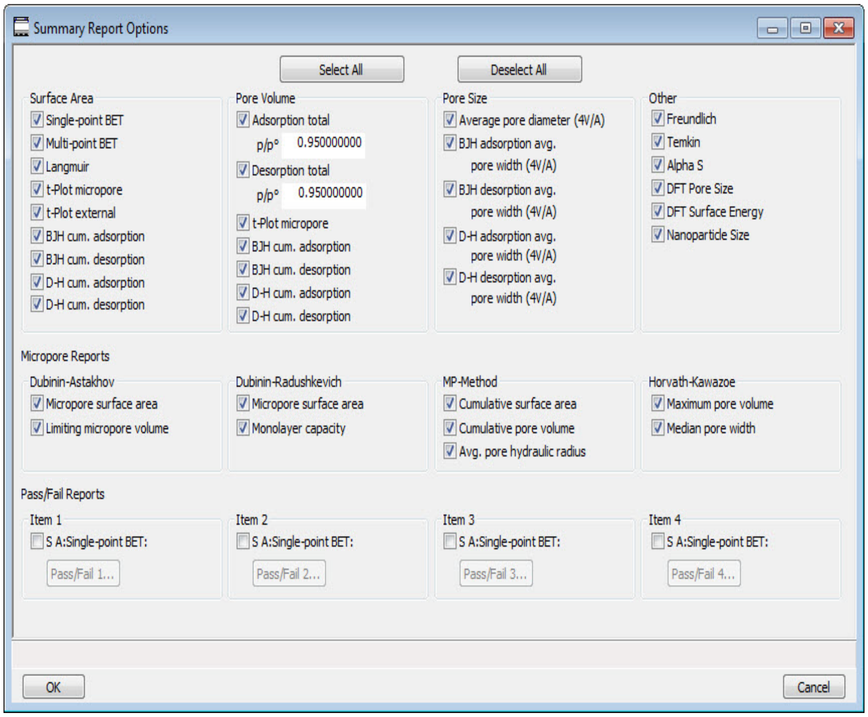
Advanced

Field or Button	Description
Report Options *dropdown list*	Use to browse for a .RPO file that contains report options parameters to be used in the report.
Show report title *text box*	Enter a report title to appear on the report header.
Show graphic *text box*	Use to show a graphic on the report header. Click the **Browse** button 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.

Field or Button	Description *(continued)*
Overlays *button*	Refer to **Multiple Sample Overlays**, page **2-50**.
Import *button*	Use to import up to 25 pore distribution data files. These datasets are shown only in BJH and Dollimore-Heal reports.
Apply thermal transpiration correction *checkbox*	Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than 1.0 mmHg. It is OK to use a sample tube filler rod and thermal transpiration if the filler rod has a bore. Always use thermal transpiration when performing micropore analyses. Refer to **Thermal Transpiration Correction**, page **C-5** for additional information on thermal transpiration. • **Inside diameter of sample tube text box -** enabled when **Apply thermal transpiration correction** is selected. Enter the inside diameter of the sample tube used in the analysis.
Selected Reports *list box*	Select the checkbox to the left of 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.
Browse *button* ***Cancel*** *button* ***Close*** *button* ***Edit*** *button* ***OK*** *button* ***Preview*** *button* ***Save*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

Summary Report

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



Surface Area
- ☑ Single-point BET
- ☑ Multi-point BET
- ☑ Langmuir
- ☑ t-Plot micropore
- ☑ t-Plot external
- ☑ BJH cum. adsorption
- ☑ BJH cum. desorption
- ☑ D-H cum. adsorption
- ☑ D-H cum. desorption

Pore Volume
- ☑ Adsorption total
  - p/p° 0.950000000
- ☑ Desorption total
  - p/p° 0.950000000
- ☑ t-Plot micropore
- ☑ BJH cum. adsorption
- ☑ BJH cum. desorption
- ☑ D-H cum. adsorption
- ☑ D-H cum. desorption

Pore Size
- ☑ Average pore diameter (4V/A)
- ☑ BJH adsorption avg. pore width (4V/A)
- ☑ BJH desorption avg. pore width (4V/A)
- ☑ D-H adsorption avg. pore width (4V/A)
- ☑ D-H desorption avg. pore width (4V/A)

Other
- ☑ Freundlich
- ☑ Temkin
- ☑ Alpha S
- ☑ DFT Pore Size
- ☑ DFT Surface Energy
- ☑ Nanoparticle Size

Micropore Reports

Dubinin-Astakhov
- ☑ Micropore surface area
- ☑ Limiting micropore volume

Dubinin-Radushkevich
- ☑ Micropore surface area
- ☑ Monolayer capacity

MP-Method
- ☑ Cumulative surface area
- ☑ Cumulative pore volume
- ☑ Avg. pore hydraulic radius

Horvath-Kawazoe
- ☑ Maximum pore volume
- ☑ Median pore width

Pass/Fail Reports

Item 1
- ☐ S A:Single-point BET:
- Pass/Fail 1...

Item 2
- ☐ S A:Single-point BET:
- Pass/Fail 2...

Item 3
- ☐ S A:Single-point BET:
- Pass/Fail 3...

Item 4
- ☐ S A:Single-point BET:
- Pass/Fail 4...

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.

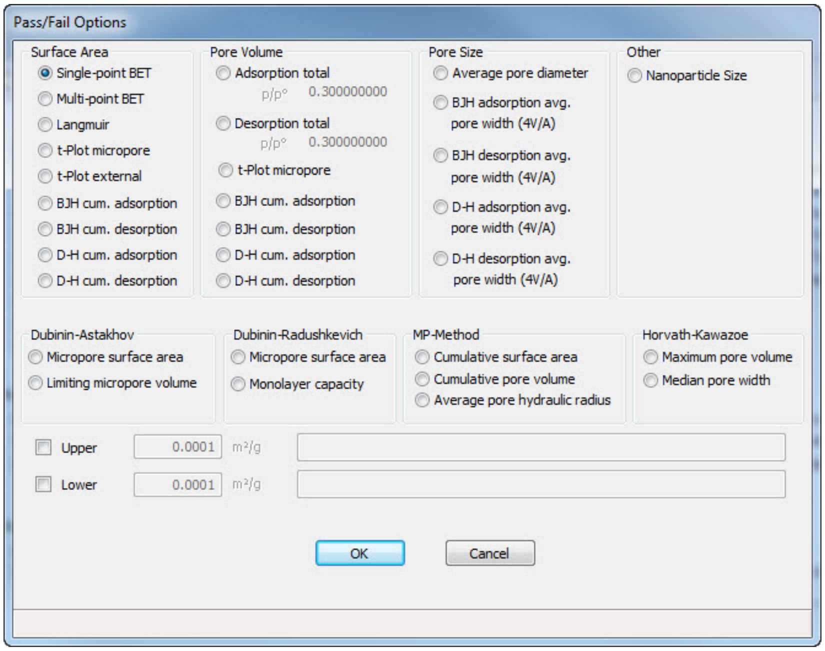
If **Use calculation assignments** is not selected on the **Collected Data** window, the isotherm is interpolated to the value in the **p/p^o** fields and the point is used for the total pore volume calculation. Otherwise, the point selected with calculation assignment is used. Pass/Fail criteria can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

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

<table>
<tr><th>Field or Button</th><th>Description (continued)</th></tr>
<tr><td>Item [n]</td><td>

Use to enable the first Pass/Fail item. Until the **Summary Report** is selected, **S A Single-point BET** will be displayed by default. When the checkbox is selected, click the **Pass/Fail** button and select criteria options for pass/fail options.

- **S A: Single-point BET** checkbox - use to enable the **Pass/Fail [*n*]** button in the **Item [*n*]** group box.
- **Pass/Fail [*n*]** button - select the **S A: Single-point BET** checkbox to enable this button. Click the **Pass/Fail [*n*]** button to display the Pass/Fail Options window for selection of pass/fail criteria.

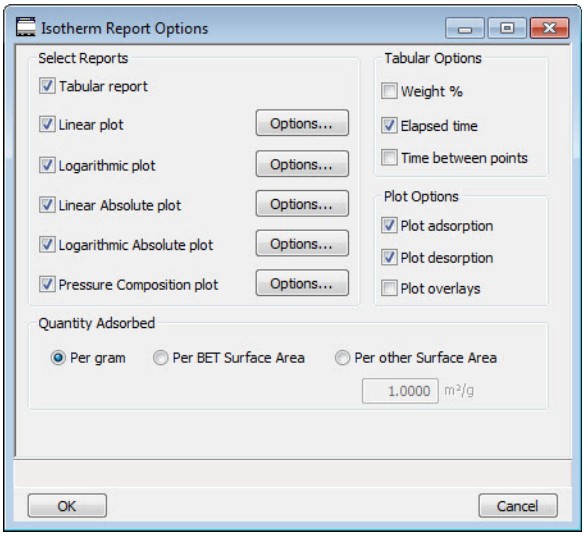


- **Upper/Lower** options and text boxes - specify upper and lower limits for the selected parameter. A range can be left open by not selecting the limit. In the text box to the right of **Upper** / **Lower**, enter operator instructions to be displayed if a failure is encountered.

</td></tr>
<tr><td>Cancel button
OK button</td><td>Refer to Common Fields and Buttons - File Menu Options, page 3-1.</td></tr>
</table>

Isotherm Report Options

The **Isotherm** report indicates adsorption (up to saturation pressure) and desorption (down from saturation pressure) of a gas by a solid held at constant temperature. In the **Selected Reports** list box, highlight **Isotherm**, then click **Edit**.



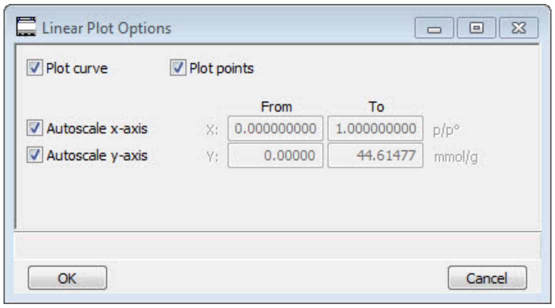
Select Reports
- ☑ Tabular report
- ☑ Linear plot — Options...
- ☑ Logarithmic plot — Options...
- ☑ Linear Absolute plot — Options...
- ☑ Logarithmic Absolute plot — Options...
- ☑ Pressure Composition plot — Options...

Tabular Options
- ☐ Weight %
- ☑ Elapsed time
- ☐ Time between points

Plot Options
- ☑ Plot adsorption
- ☑ Plot desorption
- ☐ Plot overlays

Quantity Adsorbed
- ● Per gram
- ○ Per BET Surface Area
- ○ Per other Surface Area — 1.0000 m²/g

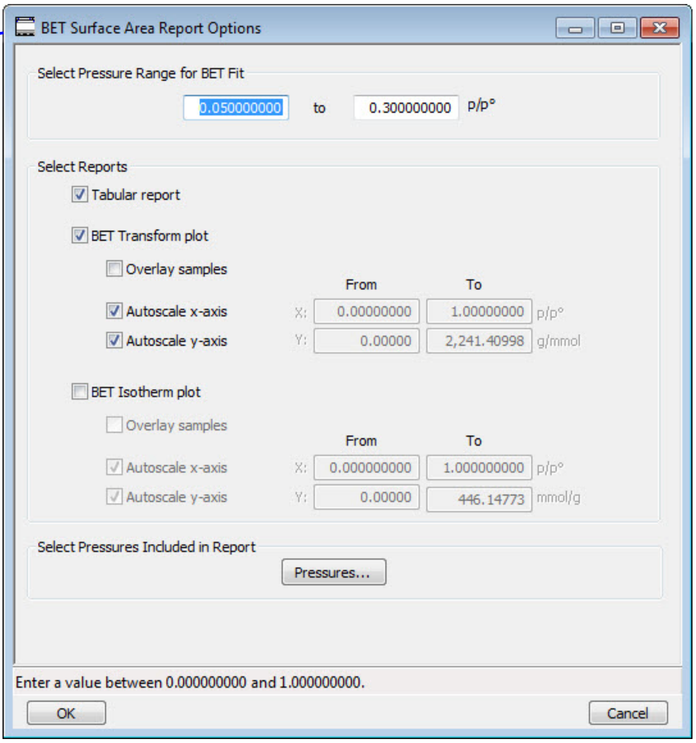
Field or Button	Description
Select Reports *group box*	Select the checkbox to the left of each option to include on the final report. To produce a tabular report of the pressure composition data, select **Pressure Composition plot** in the **Select Reports** group box and select the **Weight %** option in the **Tabular Options** group box.

Field or Button	Description *(continued)*
Options *buttons*	Click to display related linear plot options. All plot windows contain identical fields.  ☑ Plot curve ☑ Plot points From To ☑ Autoscale x-axis X: 0.000000000 1.000000000 p/p° ☑ Autoscale y-axis Y: 0.00000 44.61477 mmol/g • **Plot curve / Plot points -** use to plot curves and/or points. • **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.
Tabular Options *group box*	Select the options to include on the report. • **Weight % -** To produce a graphical report of the weight percentage data, select this option and select **Pressure Composition plot** in the **Select Reports** group box. • **Elapsed time -** the time elapsed during the analysis • **Time between points -** time elapsed between points during the analysis
Plot Options *group box*	Select the types of isotherm to plot: • adsorption • desorption • overlays
Quantity Adsorbed *group box*	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
Cancel *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

BET/Langmuir Surface Area Report Options

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

- The BET calculation obtains the sample surface area value by determining the monolayer volume of adsorbed gas from the isotherm data. BET uses a multilayer model.
- The Langmuir calculation determines the surface area of a sample by relating the surface area to the volume of gas adsorbed as a monolayer Langmuir uses a single layer model.



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

BET Surface Area Report Options

Select Pressure Range for BET Fit

0.050000000 to 0.300000000 p/p°

Select Reports

☑ Tabular report

☑ BET Transform plot

☐ Overlay samples

		From	To	
☑ Autoscale x-axis	X:	0.00000000	1.00000000	p/p°
☑ Autoscale y-axis	Y:	0.00000	2,241.40998	g/mmol

☐ BET Isotherm plot

☐ Overlay samples

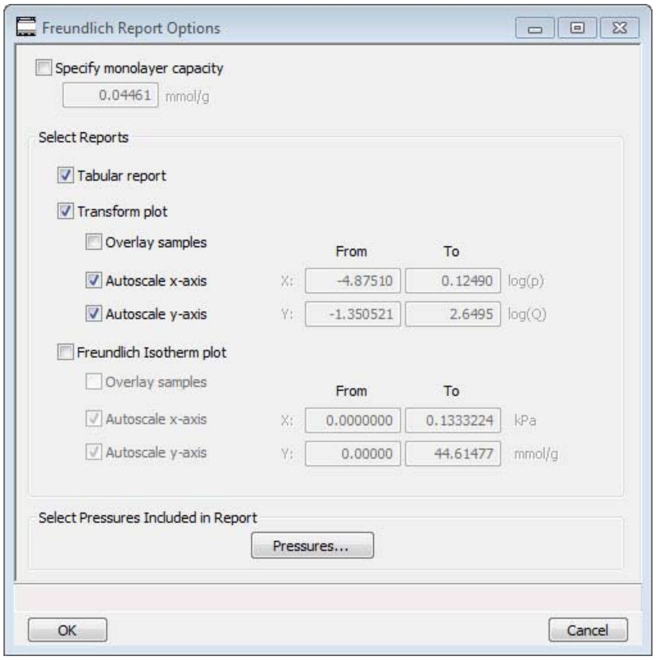
		From	To	
☑ Autoscale x-axis	X:	0.000000000	1.000000000	p/p°
☑ Autoscale y-axis	Y:	0.00000	446.14773	mmol/g

Select Pressures Included in Report

Enter a value between 0.000000000 and 1.000000000.

Freundlich Report Options

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



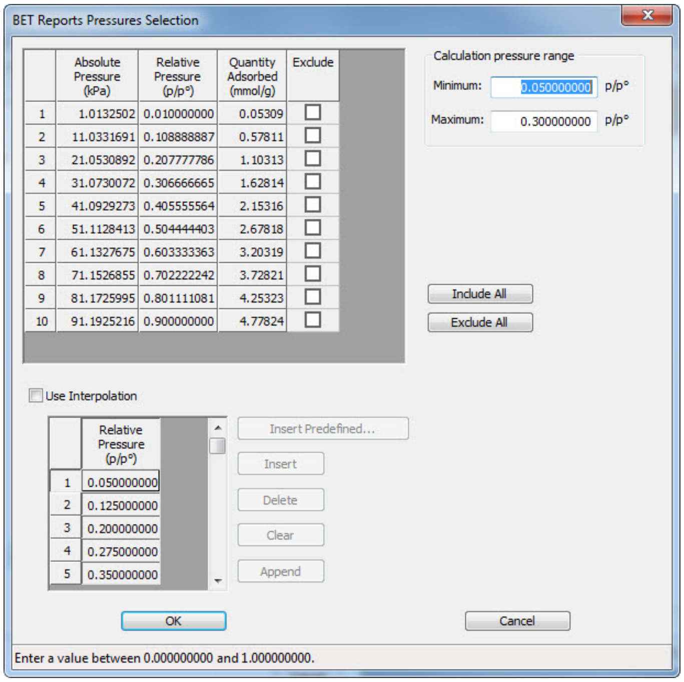
☐ Specify monolayer capacity
0.04461 mmol/g

Select Reports
☑ Tabular report
☑ Transform plot
☐ Overlay samples
From To
☑ Autoscale x-axis X: -4.87510 0.12490 log(p)
☑ Autoscale y-axis Y: -1.350521 2.6495 log(Q)
☐ Freundlich Isotherm plot
☐ Overlay samples
From To
☑ Autoscale x-axis X: 0.0000000 0.1333224 kPa
☑ Autoscale y-axis Y: 0.00000 44.61477 mmol/g

Select Pressures Included in Report

Field or Button	Description
Specify monolayer capacity *checkbox and text box*	Select and enter the monolayer capacity of the sample.
Tabular report *checkbox*	Use to have a report of the pressure points generated.
Transform plot *checkbox*	Plots the log(P) vs log(Q) and the best fit. • **Overlay samples** checkbox **-** use to overlay sample files on the Freundlich transform plot. • **Autoscale x-axis -** the x-axis field shows the absolute pressure. • **Autoscale y-axis -** the y-axis field shows the quantity of gas adsorbed.

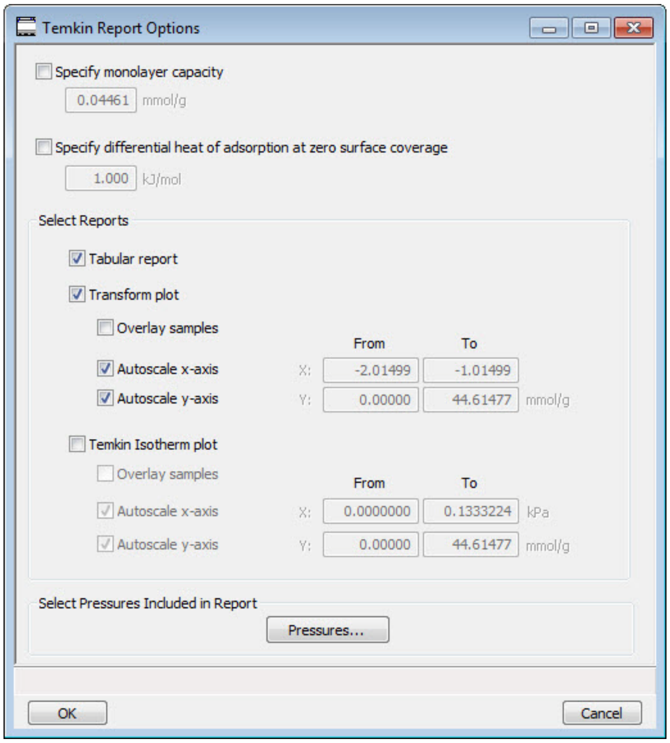
Field or Button	Description *(continued)*
Freundlich Isotherm plot *checkbox*	Plots the absolute pressure vs quantity adsorbed. Shows best fit line. • **Overlay samples** checkbox - use to overlay sample files on the Freundlich isotherm plot. • **Autoscale x-axis -** linear x-axes begin at zero. The x-axis field shows the absolute pressure. • **Autoscale y-axis -** y-axes begin at zero. The y-axis field shows the quantity of gas adsorbed.

<table>
<tr><th>Field or Button</th><th>Description (continued)</th></tr>
<tr><td>Pressures button</td><td>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.

BET Reports Pressures Selection
<table>
<tr><th></th><th>Absolute Pressure (kPa)</th><th>Relative Pressure (p/p°)</th><th>Quantity Adsorbed (mmol/g)</th><th>Exclude</th></tr>
<tr><td>1</td><td>1.0132502</td><td>0.010000000</td><td>0.05309</td><td>☐</td></tr>
<tr><td>2</td><td>11.0331691</td><td>0.108888887</td><td>0.57811</td><td>☐</td></tr>
<tr><td>3</td><td>21.0530892</td><td>0.207777786</td><td>1.10313</td><td>☐</td></tr>
<tr><td>4</td><td>31.0730072</td><td>0.306666665</td><td>1.62814</td><td>☐</td></tr>
<tr><td>5</td><td>41.0929273</td><td>0.405555564</td><td>2.15316</td><td>☐</td></tr>
<tr><td>6</td><td>51.1128413</td><td>0.504444403</td><td>2.67818</td><td>☐</td></tr>
<tr><td>7</td><td>61.1327675</td><td>0.603333363</td><td>3.20319</td><td>☐</td></tr>
<tr><td>8</td><td>71.1526855</td><td>0.702222242</td><td>3.72821</td><td>☐</td></tr>
<tr><td>9</td><td>81.1725995</td><td>0.801111081</td><td>4.25323</td><td>☐</td></tr>
<tr><td>10</td><td>91.1925216</td><td>0.900000000</td><td>4.77824</td><td>☐</td></tr>
</table>
Calculation pressure range
Minimum: 0.050000000 p/p°
Maximum: 0.300000000 p/p°
☐ Use Interpolation
<table>
<tr><th></th><th>Relative Pressure (p/p°)</th></tr>
<tr><td>1</td><td>0.050000000</td></tr>
<tr><td>2</td><td>0.125000000</td></tr>
<tr><td>3</td><td>0.200000000</td></tr>
<tr><td>4</td><td>0.275000000</td></tr>
<tr><td>5</td><td>0.350000000</td></tr>
</table>
Enter a value between 0.000000000 and 1.000000000.

• Calculation pressure range group box - enter the minimum and maximum pressures to be included in the pressure table.
• Include All button - include all pressures in the table.
• Exclude All button - exclude all pressures in the table.
• Use Interpolation checkbox - 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 button - click to insert a predefined (default) set of points into the report. The Use Interpolation checkbox must be selected to enable this button. This button displays for BET reports only. Refer to Table buttons, page 3-4 for a description of the Insert, Delete, Clear and Append buttons.</td></tr>
<tr><td>Cancel button
OK button</td><td>Refer to Common Fields and Buttons - File Menu Options, page 3-1.</td></tr>
</table>

Temkin Isotherm Report Options

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



☐ Specify monolayer capacity
0.04461 mmol/g

☐ Specify differential heat of adsorption at zero surface coverage
1.000 kJ/mol

Select Reports

☑ Tabular report

☑ Transform plot
☐ Overlay samples

		From	To	
☑ Autoscale x-axis	X:	-2.01499	-1.01499	
☑ Autoscale y-axis	Y:	0.00000	44.61477	mmol/g

☐ Temkin Isotherm plot
☐ Overlay samples

		From	To	
☑ Autoscale x-axis	X:	0.0000000	0.1333224	kPa
☑ Autoscale y-axis	Y:	0.00000	44.61477	mmol/g

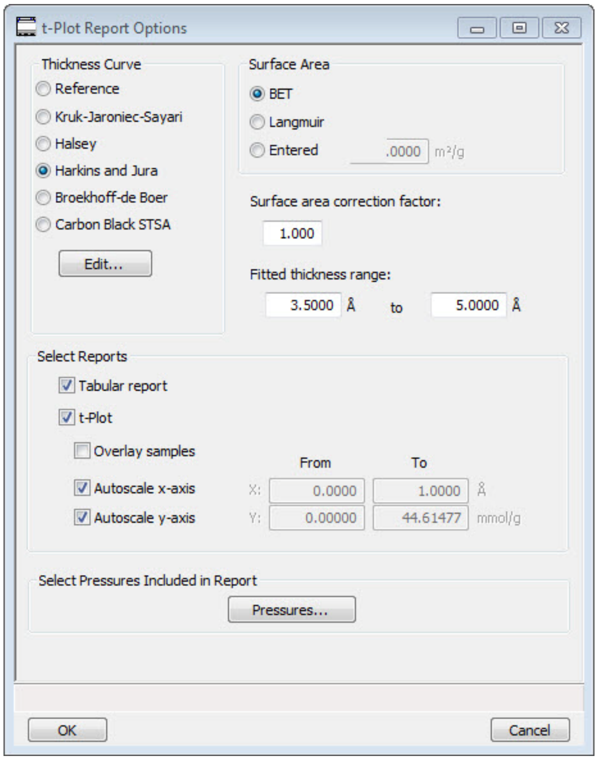
Select Pressures Included in Report

Field or Button	Description
Specify monolayer capacity *checkbox and text box*	Select and enter the monolayer capacity of the sample.
Specify differential heat of adsorption *checkbox and text box*	Select and enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all Temkin constants.
Tabular report *checkbox*	Use to have a report of the pressure points generated.
Transform plot *checkbox*	Plots a linear form of the Temkin transform plot. • **Overlay samples** checkbox - use to overlay sample files on the transform plot. • **Autoscale x-axis -** the x-axis field shows the logarithm of pressure (ln). • **Autoscale y-axis-** the y-axis field shows the quantity of gas adsorbed.

Field or Button	Description *(continued)*
Temkin Isotherm plot *checkbox*	Overlays the Temkin isotherm with the analysis data. • **Overlay samples** checkbox - use to overlay sample files on the isotherm plot. • **Autoscale x-axis -** linear x-axes begin at zero. The x-axis field shows the absolute pressure. • **Autoscale y-axis -** y-axes begin at zero. The y-axis field shows the quantity of gas adsorbed.
Pressures *button*	Refer to **Pressures button**, page **3-36**.
Cancel *button* ***From / To*** *text boxes* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

t-Plot Report Options

The t-Plot calculation allows quantitative analysis of the area and total volume ascribed to micropores. Matrix area (the area external to micropores) is directly determined and often proves to be a valuable way of characterizing complex mixed materials. In the **Selected Reports** list box, highlight **t-Plot**, then click **Edit.**



t-Plot Report Options

Thickness Curve
○ Reference
○ Kruk-Jaroniec-Sayari
○ Halsey
● Harkins and Jura
○ Broekhoff-de Boer
○ Carbon Black STSA

Surface Area
● BET
○ Langmuir
○ Entered .0000 m²/g

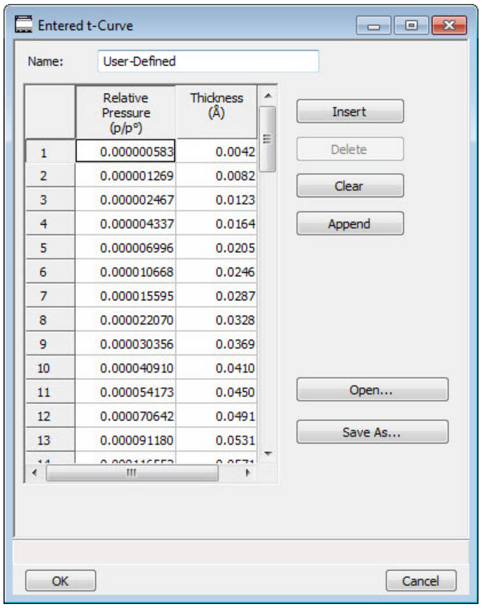
Surface area correction factor: 1.000

Fitted thickness range: 3.5000 Å to 5.0000 Å

Select Reports
☑ Tabular report
☑ t-Plot
☐ Overlay samples
☑ Autoscale x-axis X: From 0.0000 To 1.0000 Å
☑ Autoscale y-axis Y: From 0.00000 To 44.61477 mmol/g

Select Pressures Included in Report

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

<table>
<tr><th>Field or Button</th><th>Description (continued)</th></tr>
<tr><td>Thickness Curve group box (continued)</td><td>


Entered t-Curve

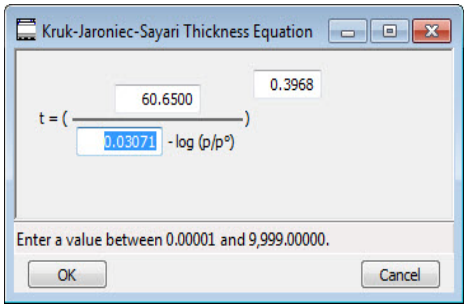
Name: User-Defined

<table>
<tr><th></th><th>Relative Pressure (p/p°)</th><th>Thickness (Å)</th></tr>
<tr><td>1</td><td>0.000000583</td><td>0.0042</td></tr>
<tr><td>2</td><td>0.000001269</td><td>0.0082</td></tr>
<tr><td>3</td><td>0.000002467</td><td>0.0123</td></tr>
<tr><td>4</td><td>0.000004337</td><td>0.0164</td></tr>
<tr><td>5</td><td>0.000006996</td><td>0.0205</td></tr>
<tr><td>6</td><td>0.000010668</td><td>0.0246</td></tr>
<tr><td>7</td><td>0.000015595</td><td>0.0287</td></tr>
<tr><td>8</td><td>0.000022070</td><td>0.0328</td></tr>
<tr><td>9</td><td>0.000030356</td><td>0.0369</td></tr>
<tr><td>10</td><td>0.000040910</td><td>0.0410</td></tr>
<tr><td>11</td><td>0.000054173</td><td>0.0450</td></tr>
<tr><td>12</td><td>0.000070642</td><td>0.0491</td></tr>
<tr><td>13</td><td>0.000091180</td><td>0.0531</td></tr>
</table>

To import values from an existing thickness curve (.THK file), click **Open** and select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space or a tab.

Refer to **Table buttons**, page **3-4** for a description of the **Insert**, **Delete, Clear**, and **Append** buttons.

Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA - select the thickness curve option and click **Edit**. Modify the equation for the selected curve as needed.



Kruk-Jaroniec-Sayari Thickness Equation

$t = \left(\frac{60.6500}{0.03071 - \log(p/p°)}\right)^{0.3968}$

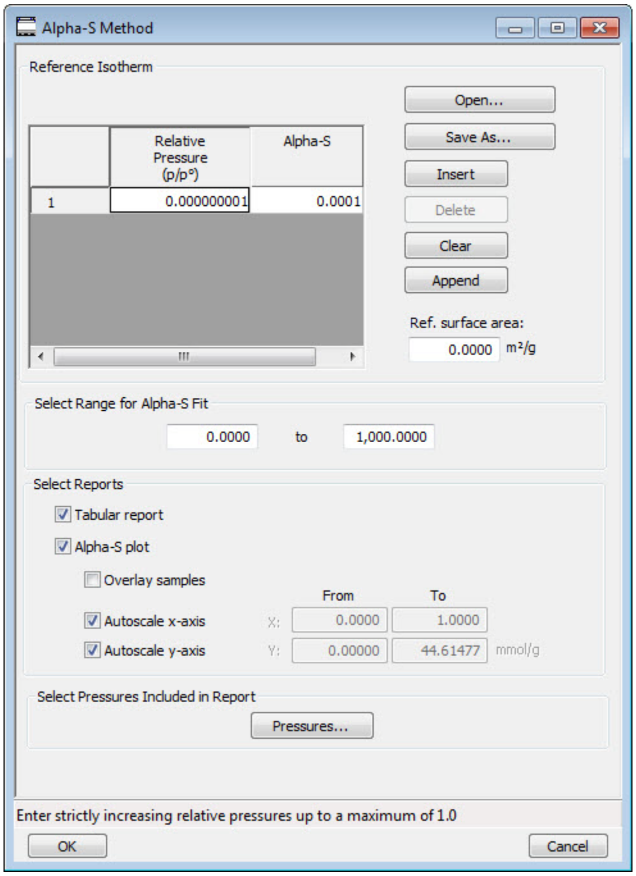
Enter a value between 0.00001 and 9,999.00000.

</td></tr>
<tr><td>Surface Area
group box</td><td>Select the surface area value used for thickness calculations. BET is the most commonly used option.</td></tr>
</table>

Field or Button	Description *(continued)*
Surface area correction factor *text box*	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.
Fitted thickness range *text boxes*	Enter the minimum and maximum thicknesses (in angstroms or nanometers) to include in the thickness curve. Go to ***Options*** > ***Units*** to specify default units. Refer to **UNIT MENU**, page **4-1**.
Tabular report *checkbox*	Use to have a tabular report of data generated.
t-Plot *checkbox*	Use to have a graphical representation of data generated. • **Overlay samples** checkbox - use to overlay sample files on the t-plot. • **Autoscale x-axis -** the x-axis field shows the statistical thickness of the adsorbed film. • **Autoscale y-axis -** the y-axis field shows the quantity of gas adsorbed.
Pressures *button*	Refer to **Pressures button**, page **3-36**.
Cancel *button* ***From / To*** *text boxes* ***OK*** *button* ***Table*** *buttons*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1.**

Alpha-S Method

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



	Relative Pressure (p/p°)	Alpha-S
1	0.000000001	0.0001

Ref. surface area: 0.0000 m²/g

0.0000 to 1,000.0000

☑ Tabular report

☑ Alpha-S plot

☐ Overlay samples

☑ Autoscale x-axis X: 0.0000 1.0000

☑ Autoscale y-axis Y: 0.00000 44.61477 mmol/g

Enter strictly increasing relative pressures up to a maximum of 1.0

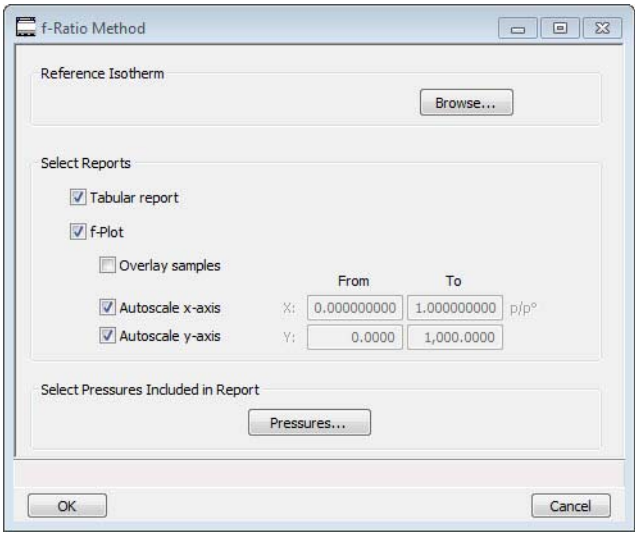
One predefined curve is shipped with the analysis program and is located in the Reference directory. Use the table to enter relative pressure and the alpha-s values.

Field or Button	Description
***Open** button*	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.
***Insert / Delete / Clear / Append** buttons*	Refer to **Table buttons**, page **3-4**.

Field or Button	**Description *(continued)***
Ref. surface area *text box*	Enter the surface area from the reference curve. This value is used to calculate the sample surface area.
Select Range for Alpha-S Fit *text boxes*	Enter minimum and maximum relative pressures to determine the fit.
Tabular report *checkbox*	Use to have a tabular report of data generated.
Alpha-S plot *checkbox*	Use to plot data in graph format. • **Overlay samples** checkbox - use to overlay sample files on the plot. • **Autoscale x-axis -** the x-axis field shows the relative pressure. • **Autoscale y-axis -** the y-axis field shows the quantity of gas adsorbed.
Pressures *button*	Refer to **Pressures button**, page **3-36**.
Browse *button* ***Cancel*** *button* ***From / To*** *buttons* ***OK*** *button* ***Save As*** *button* ***Table*** *buttons*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

f-Ratio Method

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



☑ Tabular report
☑ f-Plot
☐ Overlay samples
☑ Autoscale x-axis X: 0.000000000 1.000000000 p/p°
☑ Autoscale y-axis Y: 0.0000 1,000.0000

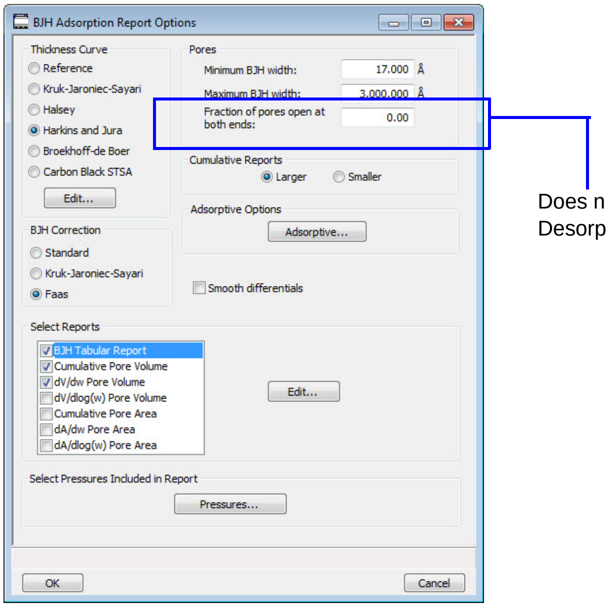
Field or Button	Description
Reference isotherm	Click **Browse** to select a sample file to use as a reference for the isotherm. Select a file containing an isotherm measured from a non-porous sample of the same material as the current sample. When the referenced file is selected, the file name appears to the left of the **Browse** button.
Tabular report *checkbox*	Select to have a tabular report of data generated.
f-Plot *checkbox*	Use to generate a normalized isotherm. • **Overlay samples** checkbox - use to overlay sample files on the f-plot. • **Autoscale x-axis -** the x-axis field is dimensionless in units of f-ratio. • **Autoscale y-axis -** the y-axis field shows the quantity of gas adsorbed.
Pressures *button*	Refer to **Pressures button**, page **3-36**.
Browse *button* ***Cancel*** *button* ***From / To*** *text boxes* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1.**

BJH Adsorption/Desorption Report Options

The BJH calculation determines the mesopore volume/area distribution which accounts for both the change in adsorbate layer thickness and the liquid condensed in pore cores. Reports can be generated from both adsorption and desorption data.

The fields for both report options are identical unless otherwise specified.

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


BJH Adsorption Report Options

Thickness Curve
○ Reference
○ Kruk-Jaroniec-Sayari
○ Halsey
● Harkins and Jura
○ Broekhoff-de Boer
○ Carbon Black STSA

Pores
Minimum BJH width: 17.000 Å
Maximum BJH width: 3,000.000 Å
Fraction of pores open at both ends: 0.00

Cumulative Reports
● Larger ○ Smaller

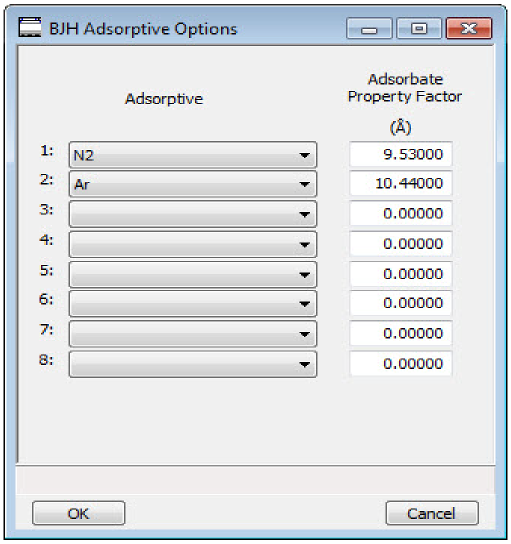
BJH Correction
○ Standard
○ Kruk-Jaroniec-Sayari
● Faas

☐ Smooth differentials

Select Reports
☑ BJH Tabular Report
☑ Cumulative Pore Volume
☑ dV/dw Pore Volume
☐ dV/dlog(w) Pore Volume
☐ Cumulative Pore Area
☐ dA/dw Pore Area
☐ dA/dlog(w) Pore Area

Does not display on the Desorption window.

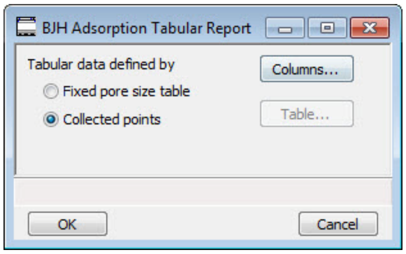
Field or Button	Description
Thickness Curve *group box*	Refer to **Thickness Curve group box**, page **3-39**.
BJH Correction *group box*	Select the type of correction to apply to calculations. The selected type displays in the report header. • **Standard -** uses original BJH models • **Kruk-Jaroniec-Sayari -** good for reference thickness curves • **Faas -** good for statistical thickness curves

Field or Button	Description *(continued)*
Pores *group box*	• **Minimum / Maximum BJH width**- 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. ***<u>Note for desorption only</u>*** - 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.
Cumulative Reports *options*	• **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.
Adsorptive *button*	Displays the **BJH Adsorptive Options** window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.  BJH Adsorptive Options Adsorptive — Adsorbate Property Factor (Å) 1: N2 — 9.53000 2: Ar — 10.44000 3: — 0.00000 4: — 0.00000 5: — 0.00000 6: — 0.00000 7: — 0.00000 8: — 0.00000
Smooth differentials *checkbox*	Use to smooth all differential calculations thus eliminating variations in the differential computation caused by noise in the input data.

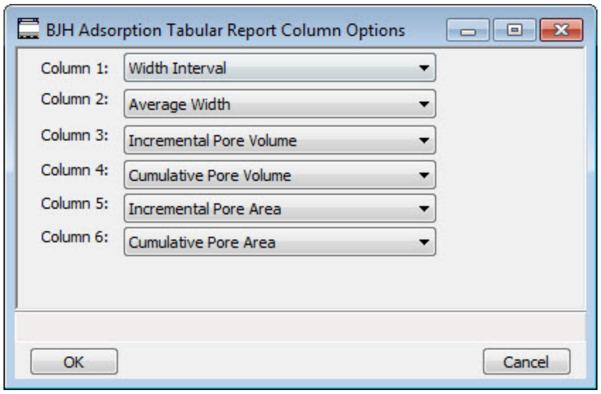
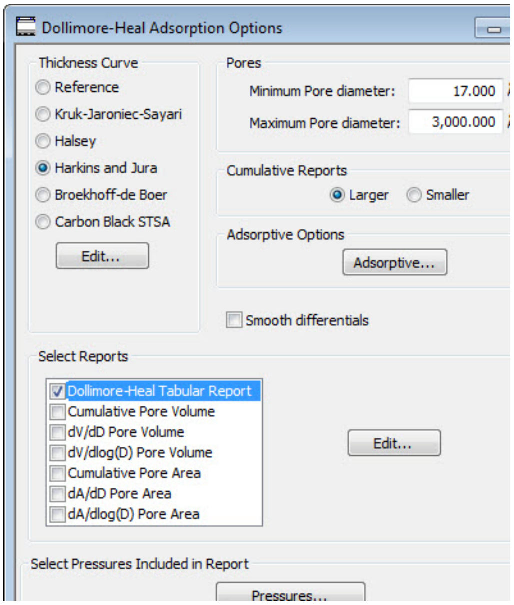
Field or Button	Description *(continued)*
Selected Reports *list box*	Select the checkbox to the left of the report names to include in the report. Highlight the report name and click the **Edit** button to modify report parameters.
Pressures *button*	Refer to **Pressures button**, page **3-36**.
Cancel *button* ***Edit*** *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

Tabular Report Options

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

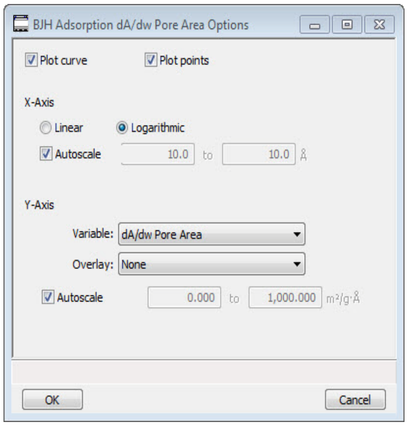


Field or Button	Description
Fixed pore size table	Use to specify exact pore sizes for volume or area data. Click the **Table** button to modify the fixed pore size table. Refer to **Table** and **Columns** below for information on the use of these buttons.
Collected points	Use to include all relative pressure points collected. Refer to the **Columns** button shown below.

Field or Button	Description *(continued)*
Columns *button*	Select the data types to include in the report. **Column [*n*]** indicates the column order and data contents for the report.  BJH Adsorption Tabular Report Column Options Column 1: Width Interval Column 2: Average Width Column 3: Incremental Pore Volume Column 4: Cumulative Pore Volume Column 5: Incremental Pore Area Column 6: Cumulative Pore Area
Table *button*	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.  Dollimore-Heal Adsorption Options Thickness Curve ○ Reference ○ Kruk-Jaroniec-Sayari ○ Halsey ● Harkins and Jura ○ Broekhoff-de Boer ○ Carbon Black STSA Pores Minimum Pore diameter: 17.000 Maximum Pore diameter: 3,000.000 Cumulative Reports ● Larger ○ Smaller Adsorptive Options ☐ Smooth differentials Select Reports ☑ Dollimore-Heal Tabular Report ☐ Cumulative Pore Volume ☐ dV/dD Pore Volume ☐ dV/dlog(D) Pore Volume ☐ Cumulative Pore Area ☐ dA/dD Pore Area ☐ dA/dlog(D) Pore Area Select Pressures Included in Report
Cancel *button* ***OK*** *button* ***Table*** *buttons*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

Plot Options

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


BJH Adsorption dA/dw Pore Area Options

☑ Plot curve ☑ Plot points

X-Axis

○ Linear ● Logarithmic

☑ Autoscale 10.0 to 10.0 Å

Y-Axis

Variable: dA/dw Pore Area

Overlay: None

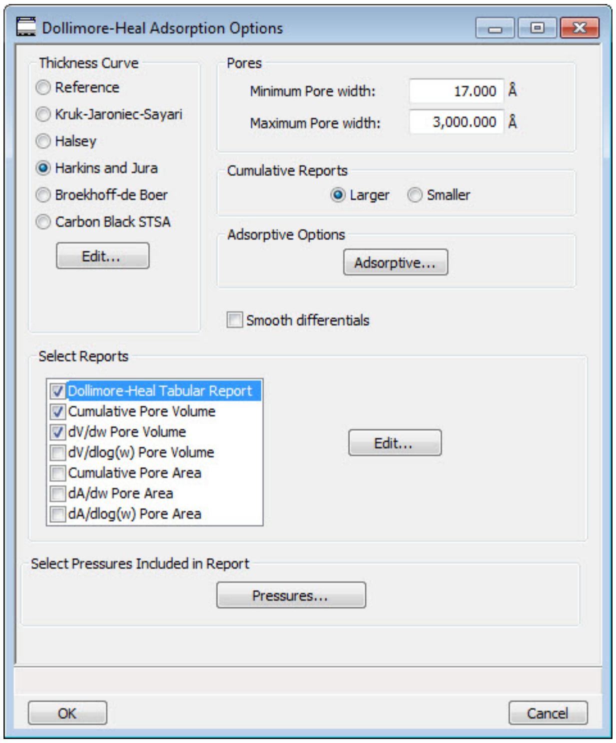
☑ Autoscale 0.000 to 1,000.000 m²/g·Å

Field or Button	Description
Plot curve / Plot points *checkboxes*	Refer to **Isotherm Report Options**, page **3-31**.
X-Axis	Use to have the x-axis on a logarithmic or linear scale.
Y-Axis	• **Variable** dropdown list - select a variable**.** • **Overlay** dropdown list **-** select an option to overlay on the current report.
Autoscale *checkbox* ***Cancel*** *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1.**

Dollimore-Heal Adsorption/Desorption Report Options

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

Dollimore-Heal Adsorption/Desorption fields and buttons are identical to the **BJH Adsorption/ Desorption Report Options**, page **3-45.**


Or Dollimore-Heal Desorption Report Options

Dollimore-Heal Adsorption Options

Thickness Curve
○ Reference
○ Kruk-Jaroniec-Sayari
○ Halsey
● Harkins and Jura
○ Broekhoff-de Boer
○ Carbon Black STSA

Pores
Minimum Pore width: 17.000 Å
Maximum Pore width: 3,000.000 Å

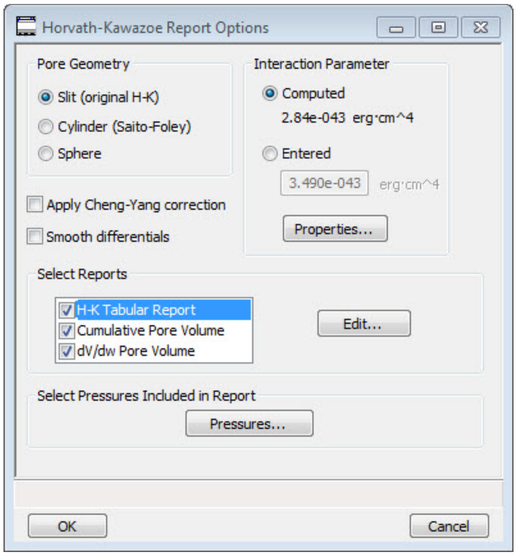
Cumulative Reports
● Larger ○ Smaller

☐ Smooth differentials

Select Reports
☑ Dollimore-Heal Tabular Report
☑ Cumulative Pore Volume
☑ dV/dw Pore Volume
☐ dV/dlog(w) Pore Volume
☐ Cumulative Pore Area
☐ dA/dw Pore Area
☐ dA/dlog(w) Pore Area

Horvath-Kawazoe Report Options

The Horvath-Kawazoe method plots individual peaks for different pore sizes even if the difference between one pore size and the next is only one angstrom (0.10 nm) or less. In the **Selected Reports** list box, highlight **Horvath-Kawazoe**, then click **Edit**.



Horvath-Kawazoe Report Options

Pore Geometry
● Slit (original H-K)
○ Cylinder (Saito-Foley)
○ Sphere

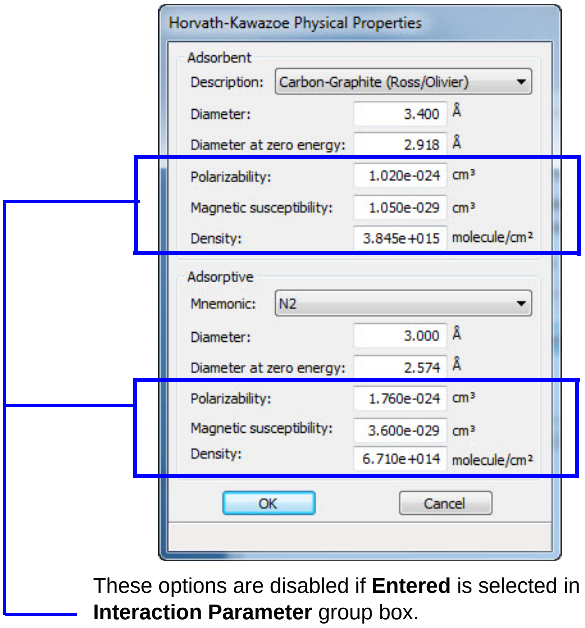
☐ Apply Cheng-Yang correction
☐ Smooth differentials

Interaction Parameter
● Computed
2.84e-043 erg·cm^4
○ Entered
3.490e-043 erg·cm^4

Select Reports
☑ H-K Tabular Report
☑ Cumulative Pore Volume
☑ dV/dw Pore Volume

Select Pressures Included in Report

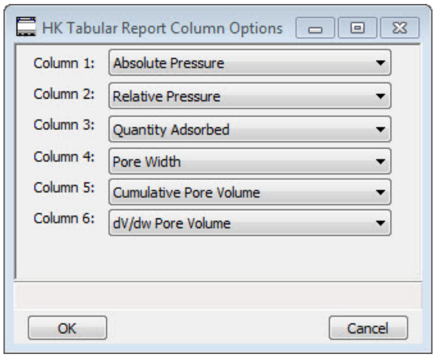
Field or Button	Description
Pore Geometry *group box*	Select the option that best represents the physical geometry of the micropores in the sample material. When **Sphere** is selected, options in the **Interaction Parameter** group box are disabled.
Interaction Parameter *group box*	Use to determine which interaction parameter will be used in the report. These options are disabled if **Sphere** is selected in the **Pore Geometry** group box. • **Computed -** use to calculate using the parameters on the **Horvath-Kawazoe Physical Properties** window (click the **Properties** button to display the **Physical Properties** window). The interaction parameter is recalculated each time a parameter in the **Physical Properties** window is edited. • **Entered -** select to calculate using the value entered in the text box.

Field or Button	Description *(continued)*
***Properties** button*	Click to view or edit the constants describing the physical properties of the adsorbent and adsorptive.  Horvath-Kawazoe Physical Properties Adsorbent Description: Carbon-Graphite (Ross/Olivier) Diameter: 3.400 Å Diameter at zero energy: 2.918 Å Polarizability: 1.020e-024 cm^3 Magnetic susceptibility: 1.050e-029 cm^3 Density: 3.845e+015 molecule/cm^2 Adsorptive Mnemonic: N2 Diameter: 3.000 Å Diameter at zero energy: 2.574 Å Polarizability: 1.760e-024 cm^3 Magnetic susceptibility: 3.600e-029 cm^3 Density: 6.710e+014 molecule/cm^2 These options are disabled if **Entered** is selected in the **Interaction Parameter** group box. **Adsorbent** group box: 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.

Field or Button	Description *(continued)*
Properties *button (continued)*	**Adsorptive** group box: Contains the parameters for the adsorptives (provided with the software and/or user-defined). If using **Computed** for the interaction parameter, all fields are enabled. If using **Entered**, only the values in the **Diameter** and **Diameter at zero energy** text fields may be edited. • **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: $(2/5)^{1/6}$ × diameter. • **Polarizability -** enter the polarizability of the adsorptive. • **Magnetic susceptibility -** enter the magnetic susceptibility of the adsorptive. • **Density -** enter the density per unit area of the adsorptive.
Apply Cheng-Yang correction *checkbox*	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.
Smooth differentials *checkbox*	Refer to **BJH Adsorption/Desorption Report Options**, page **3-45**.
Selected Reports *list box*	Select the types of reports to generate.
Cancel *button* ***Edit*** *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

Tabular Report Options

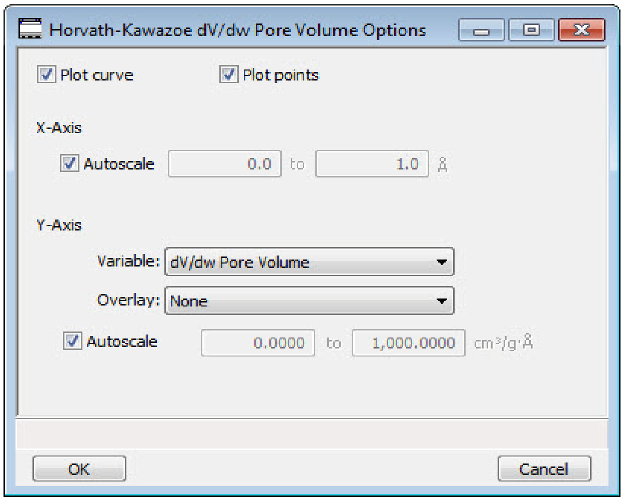
Highlight **H-K Tabular Report** in the **Selected Reports** list box in the **Horvath-Kawazoe Report Options** window and click **Edit.** Select the data types to include in the report. **Column [*n*]** indicates the column order and data contents for the report.



Column 1: Absolute Pressure
Column 2: Relative Pressure
Column 3: Quantity Adsorbed
Column 4: Pore Width
Column 5: Cumulative Pore Volume
Column 6: dV/dw Pore Volume

Plot Options

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



☑ Plot curve ☑ Plot points

X-Axis
☑ Autoscale 0.0 to 1.0 Å

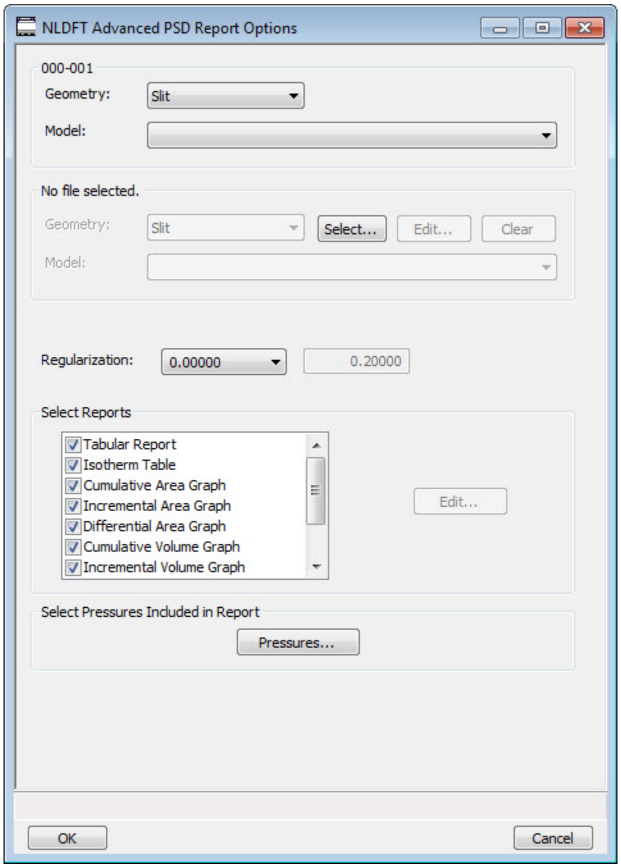
Y-Axis
Variable: dV/dw Pore Volume
Overlay: None
☑ Autoscale 0.0000 to 1,000.0000 cm³/g·Å

Field or Button	Description
Plot curve / Plot points *checkboxes*	Use to plot curves and/or points.

Field or Button	Description *(continued)*
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 adsorbed. • **Variable** dropdown list - select a y-axis variable for the report. • **Overlay** dropdown list - select an option to overlay on the current report.
Autoscale *checkbox* ***Cancel*** *button* ***Edit*** *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**

NLDFT Advanced PSD Report Options

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. In the **Selected Reports** list box, highlight **NLDFT Advanced PSD**, then click **Edit.**



000-001

Geometry: Slit

Model:

No file selected.

Geometry: Slit

Model:

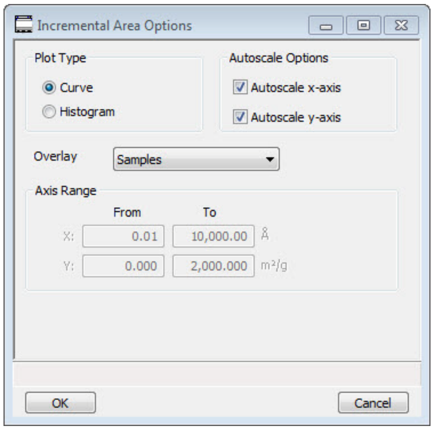
Regularization: 0.00000 0.20000

Select Reports

- ☑ Tabular Report
- ☑ Isotherm Table
- ☑ Cumulative Area Graph
- ☑ Incremental Area Graph
- ☑ Differential Area Graph
- ☑ Cumulative Volume Graph
- ☑ Incremental Volume Graph

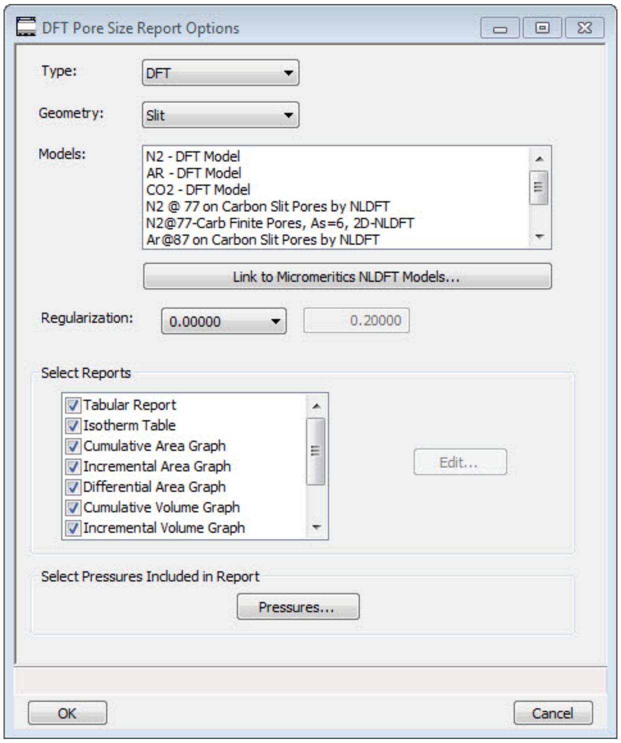
Select Pressures Included in Report

Field or Button	Description
Geometry *dropdown list*	Select the pore shape.
Model *list box*	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.
Select *button*	Use to select the second sample file.
Regularization *dropdown list and text box*	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.

Field or Button	Description *(continued)*
Selected Reports *group box*	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. Incremental Area Options Plot Type: ● Curve ○ Histogram Autoscale Options: ☑ Autoscale x-axis ☑ Autoscale y-axis Overlay: Samples Axis Range: X: From 0.01 To 10,000.00 Å; Y: From 0.000 To 2,000.000 m²/g OK Cancel • **Plot Type** group box - select the method for data display. • **Autoscale Options** group box - use to autoscale the x-axis and/or y-axes. • **Overlay** dropdown list - select an overlay for the report. • **Axis Range** group box - **From / To** fields are enabled when Autoscale options are not selected. Enter the starting and ending values for the x- and/or y-axes. – **X-axis** - shows the pore size. – **Y-axis** - shows the area.
Pressures *button*	Refer to **Pressures button**, page **3-36**.
Cancel *button* ***Clear*** ***Edit*** ***From / To*** *text boxes* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

DFT Pore Size Report Options

The DFT Pore Size report contains the results of pore size distribution analyses using a non-local DFT model for determining the pore size distribution. In the **Selected Reports** list box, highlight **DFT Pore Size**, then click **Edit**.



Type: DFT

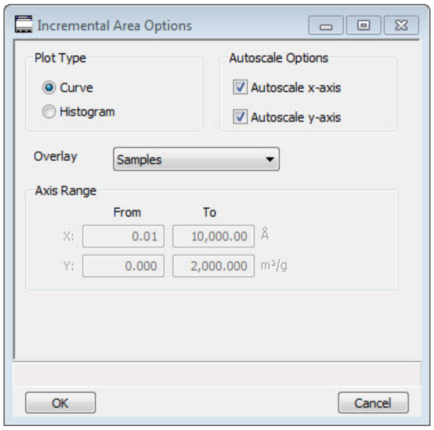
Geometry: Slit

Models:
N2 - DFT Model
AR - DFT Model
CO2 - DFT Model
N2 @ 77 on Carbon Slit Pores by NLDFT
N2@77-Carb Finite Pores, As=6, 2D-NLDFT
Ar@87 on Carbon Slit Pores by NLDFT

Regularization: 0.00000 0.20000

- ☑ Tabular Report
- ☑ Isotherm Table
- ☑ Cumulative Area Graph
- ☑ Incremental Area Graph
- ☑ Differential Area Graph
- ☑ Cumulative Volume Graph
- ☑ Incremental Volume Graph

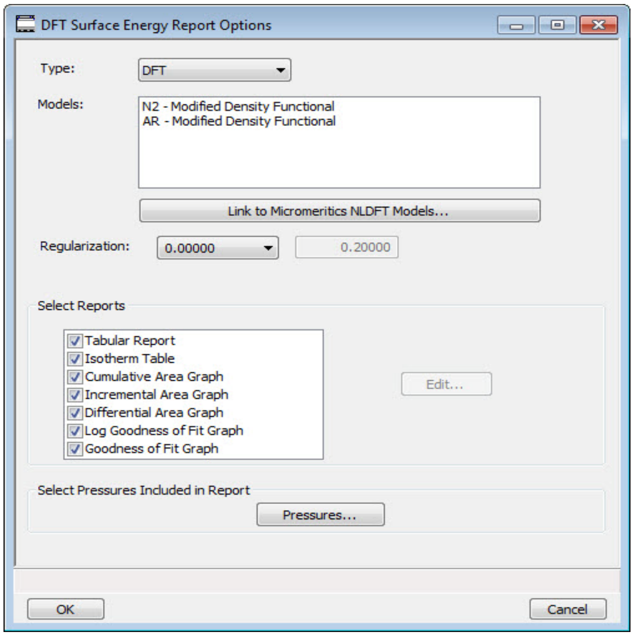
Field or Button	Description
***Type** dropdown list*	• **DFT -** model based on the density functional theory. • **Classical -** model based on the Kelvin equation and thickness for determining the pore size distribution. Refer to **DFT MODELS**, page **F-1** for further discussion on models.
***Geometry** dropdown list*	Select the pore shape.
***Model** list box*	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.
***Link to Micromeritics NLDFT Models** button*	Click to hyperlink to the **NLDFT Model Table** on the Micromeritics web page.

Field or Button	Description *(continued)*
Regularization *dropdown list and text box*	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.
Selected Reports *group box*	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. Incremental Area Options Plot Type ● Curve ○ Histogram Autoscale Options ☑ Autoscale x-axis ☑ Autoscale y-axis Overlay: Samples Axis Range From / To X: 0.01 / 10,000.00 Å Y: 0.000 / 2,000.000 m²/g OK Cancel • **Plot Type** group box - select the method for data display. • **Autoscale Options** group box - use to autoscale the x-axis and/or y-axes. • **Overlay** dropdown list - select an overlay for the report. • **Axis Range** group box - **From / To** fields are enabled when Autoscale options are not selected. Enter the starting and ending values for the x- and/or y-axes. – **X-axis** - shows the pore size. – **Y-axis** - shows the area.
Pressures *button*	Refer to **Pressures button**, page **3-36**.
Cancel *button* ***From / To*** *text boxes* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1.**

DFT Surface Energy Report Options

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

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



Type: DFT

Models:
N2 - Modified Density Functional
AR - Modified Density Functional

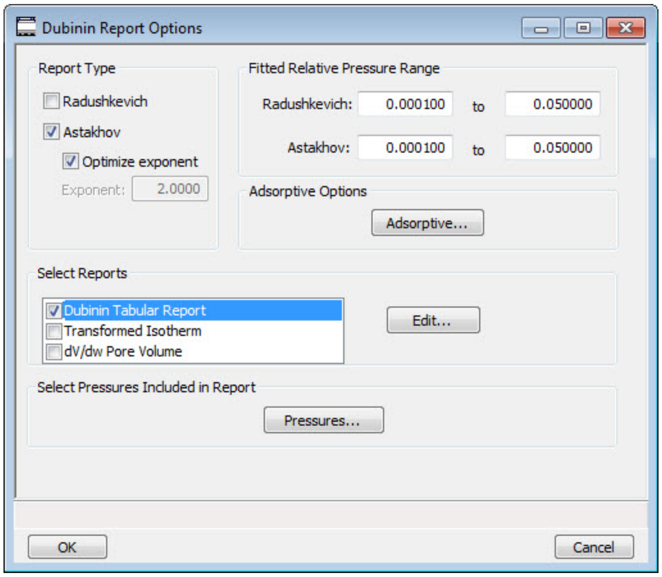
Regularization: 0.00000 0.20000

Select Reports
- ☑ Tabular Report
- ☑ Isotherm Table
- ☑ Cumulative Area Graph
- ☑ Incremental Area Graph
- ☑ Differential Area Graph
- ☑ Log Goodness of Fit Graph
- ☑ Goodness of Fit Graph

Select Pressures Included in Report

Dubinin Report Options

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



Report Type

☐ Radushkevich
☑ Astakhov
☑ Optimize exponent
Exponent: 2.0000

Fitted Relative Pressure Range

Radushkevich: 0.000100 to 0.050000
Astakhov: 0.000100 to 0.050000

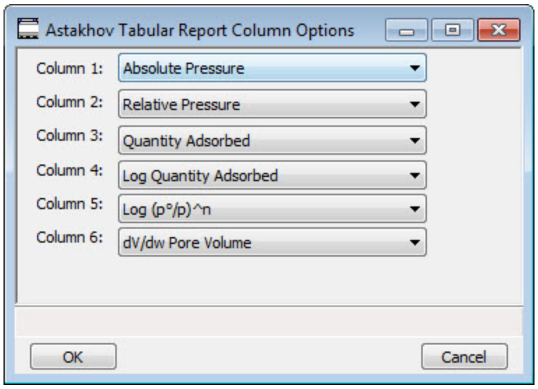
Select Reports

☑ Dubinin Tabular Report
☐ Transformed Isotherm
☐ dV/dw Pore Volume

Field or Button	Description
Report Type *group box*	Select report types. If **Astakhov** is selected, either select the **Optimize exponent** checkbox or enter an appropriate exponent value in the text box.
Fitted Relative Pressure Range *text boxes*	Enter the minimum and maximum limits for Radushkevich or Astakhov relative pressures included in the line fit.
Selected Reports *list box*	Select the reports to generate. Highlight the report and click the **Edit** button to modify report options.
Pressures *button* ***Adsorptive*** *button*	Refer to **BJH Adsorption/Desorption Report Options**, page **3-45.**
Cancel *button* ***Edit*** *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1.**

Tabular Report Options

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



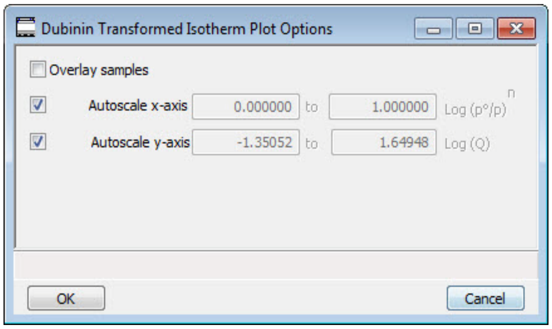
Column 1: Absolute Pressure
Column 2: Relative Pressure
Column 3: Quantity Adsorbed
Column 4: Log Quantity Adsorbed
Column 5: Log (p°/p)^n
Column 6: dV/dw Pore Volume

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

Transformed Isotherm Plot Options

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

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



☐ Overlay samples
☑ Autoscale x-axis 0.000000 to 1.000000 Log (p°/p)^n
☑ Autoscale y-axis -1.35052 to 1.64948 Log (Q)

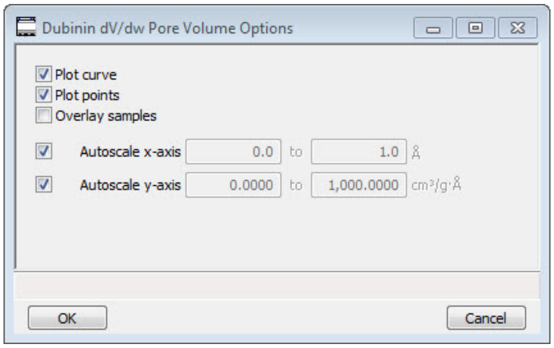
Field or Button	Description
Overlay samples *checkbox*	Use to overlay sample files on the plot.

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

Pore Volume Options

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

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

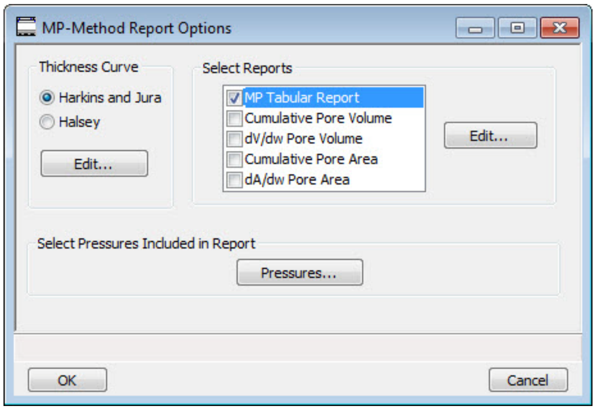


☑ Plot curve
☑ Plot points
☐ Overlay samples
☑ Autoscale x-axis 0.0 to 1.0 Å
☑ Autoscale y-axis 0.0000 to 1,000.0000 cm³/g·Å

Field or Button	Description
Plot curve / Plot points *checkboxes*	Use to plot curves and/or points.
Overlay samples *checkbox*	Use to overlay sample files on the plot.
Autoscale x-axis / Autoscale y-axis *checkboxes*	Select an option to have the x- and/or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges. To enter beginning and ending values manually, deselect these checkboxes.
Cancel *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

MP-Method Report Options

The MP-Method provides pore volume distributions for microporous materials by correlating quantity adsorbed with the thickness of the adsorbed layer as determined from a user-selected thickness curve. In the **Selected Reports** list box, highlight **MP-Method**, then click the **Edit** button.

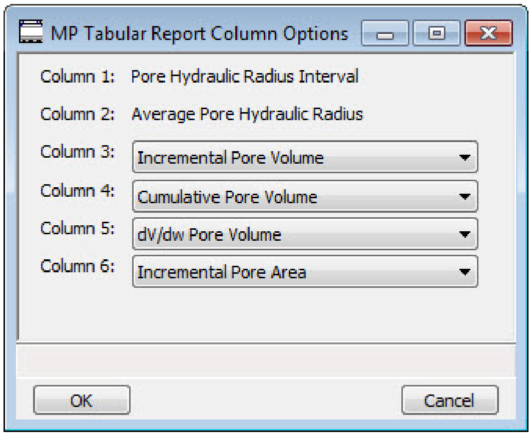


Pore size can be expressed in angstroms or nanometers. Go to *Options > Units* to specify the unit. Refer to UNIT MENU, page **4-1**.

Field or Button	Description
Thickness Curve *group box*	Refer to **Thickness Curve group box**, page **3-39**.
Selected Reports *list box*	Select the reports to generate. Highlight the report and click the **Edit** button to modify report options.
Pressures *button*	Refer to **Pressures button**, page **3-36.**
Cancel *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

Tabular Report Options

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

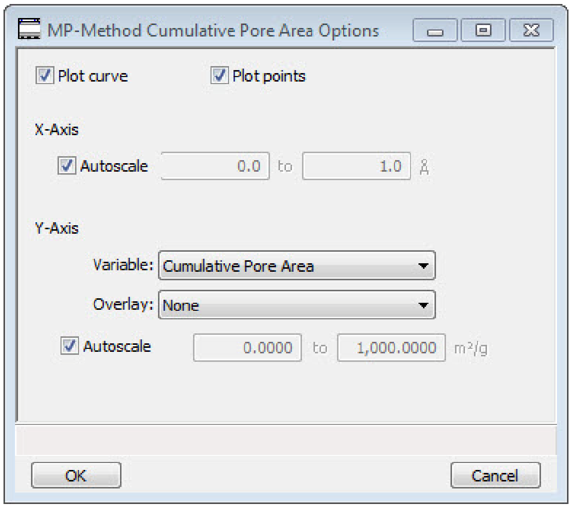


Column 1: Pore Hydraulic Radius Interval
Column 2: Average Pore Hydraulic Radius
Column 3: Incremental Pore Volume
Column 4: Cumulative Pore Volume
Column 5: dV/dw Pore Volume
Column 6: Incremental Pore Area

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

Plot Options

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



☑ Plot curve ☑ Plot points

☑ Autoscale 0.0 to 1.0 Å

Variable: Cumulative Pore Area

Overlay: None

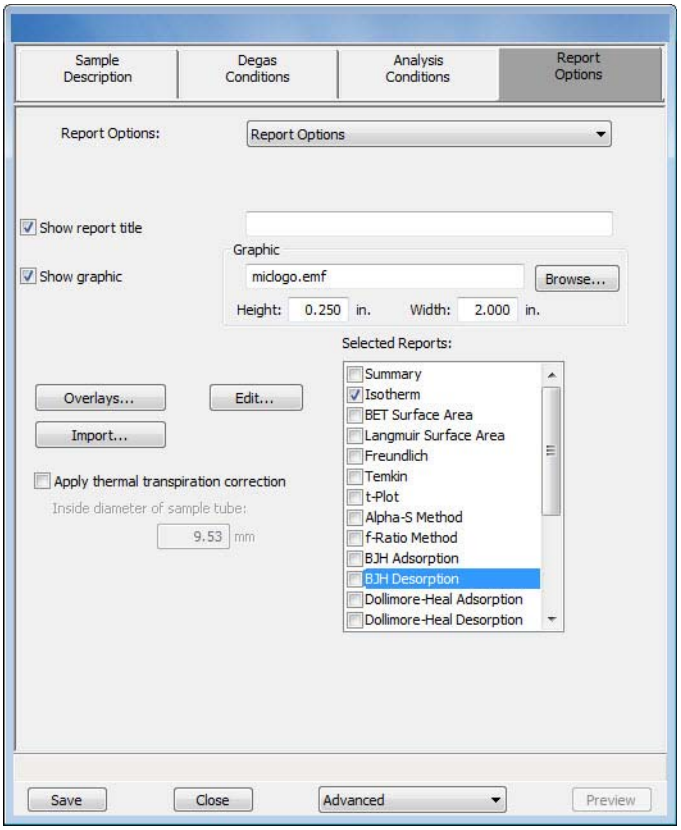
☑ Autoscale 0.0000 to 1,000.0000 m^2/g

Field or Button	Description
Plot curve / Plot points *checkboxes*	Use to plot a curve and/or points.
X-Axis	Use to have the x-axis autoscaled or enter beginning and ending values.
Y-Axis	• **Variable** dropdown list - select a variable**.** • **Overlay** dropdown list - select an option to overlay on the current report. • **Autoscale** checkbox - use to have the y-axis autoscaled or enter beginning and ending values.
Cancel *button* ***OK*** *button*	Refer to **Common Fields and Buttons - File Menu Options**, page **3-1**.

User-Defined Reports

Up to five user-defined reports may be created, each with up to 10 summary reports, 10 tabular reports, and 10 graphical reports. In order to use this feature, a file must be created that contains a Python script that imports a "mic" python module. Refer to **USER-DEFINED REPORTS, PYTHON MODULE**, page **G-1** for an example of the python script and functions for the "mic" python Module.

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



Report Options: Report Options

☑ Show report title

Graphic

☑ Show graphic miclogo.emf

Height: 0.250 in. Width: 2.000 in.

Selected Reports:

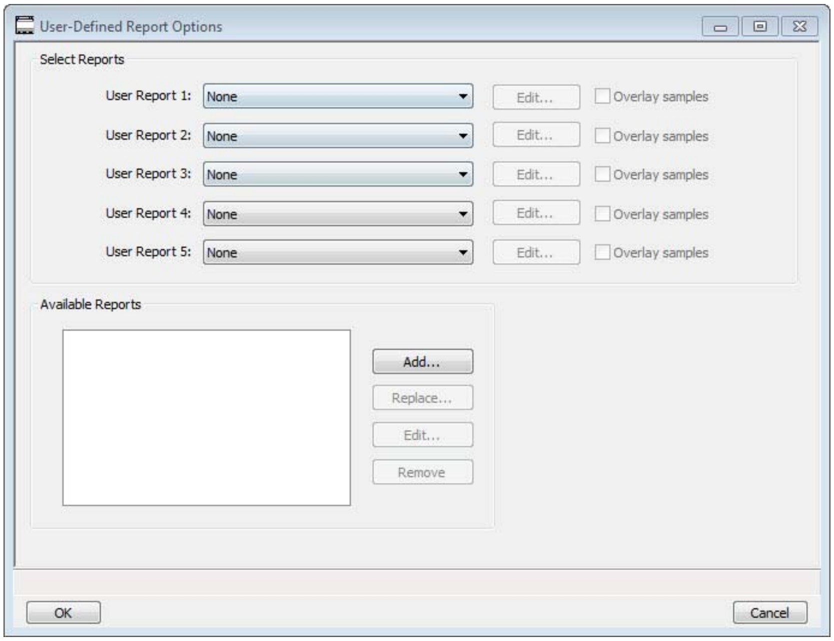
☐ Summary
☑ Isotherm
☐ BET Surface Area
☐ Langmuir Surface Area
☐ Freundlich
☐ Temkin
☐ t-Plot
☐ Alpha-S Method
☐ f-Ratio Method
☐ BJH Adsorption
☐ BJH Desorption
☐ Dollimore-Heal Adsorption
☐ Dollimore-Heal Desorption

☐ Apply thermal transpiration correction

Inside diameter of sample tube: 9.53 mm

Advanced

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



User Report 1: None

User Report 2: None

User Report 3: None

User Report 4: None

User Report 5: None

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

Options Report

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

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

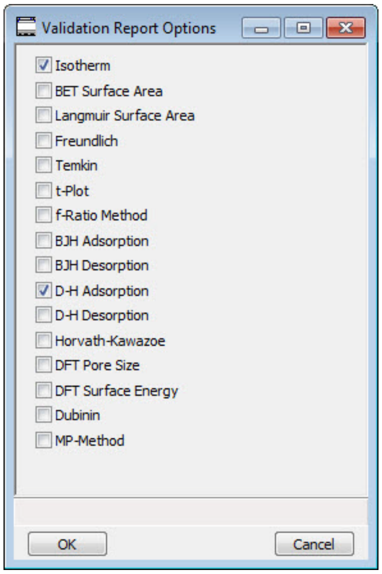
Sample Log Report

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

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

Validation Report

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



☑ Isotherm
☐ BET Surface Area
☐ Langmuir Surface Area
☐ Freundlich
☐ Temkin
☐ t-Plot
☐ f-Ratio Method
☐ BJH Adsorption
☐ BJH Desorption
☑ D-H Adsorption
☐ D-H Desorption
☐ Horvath-Kawazoe
☐ DFT Pore Size
☐ DFT Surface Energy
☐ Dubinin
☐ MP-Method

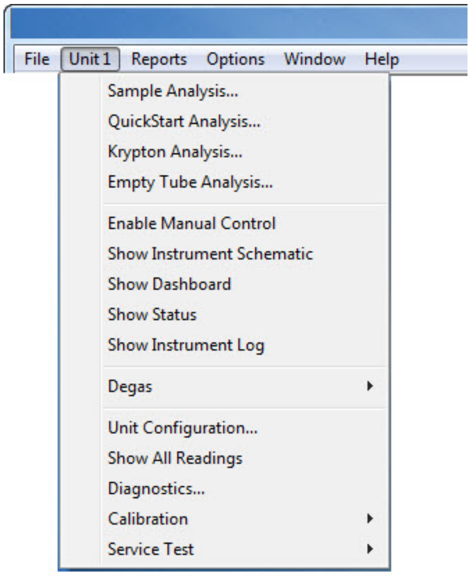
4. UNIT MENU

Introduction

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

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

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



Common Fields and Buttons - Unit Menu Options

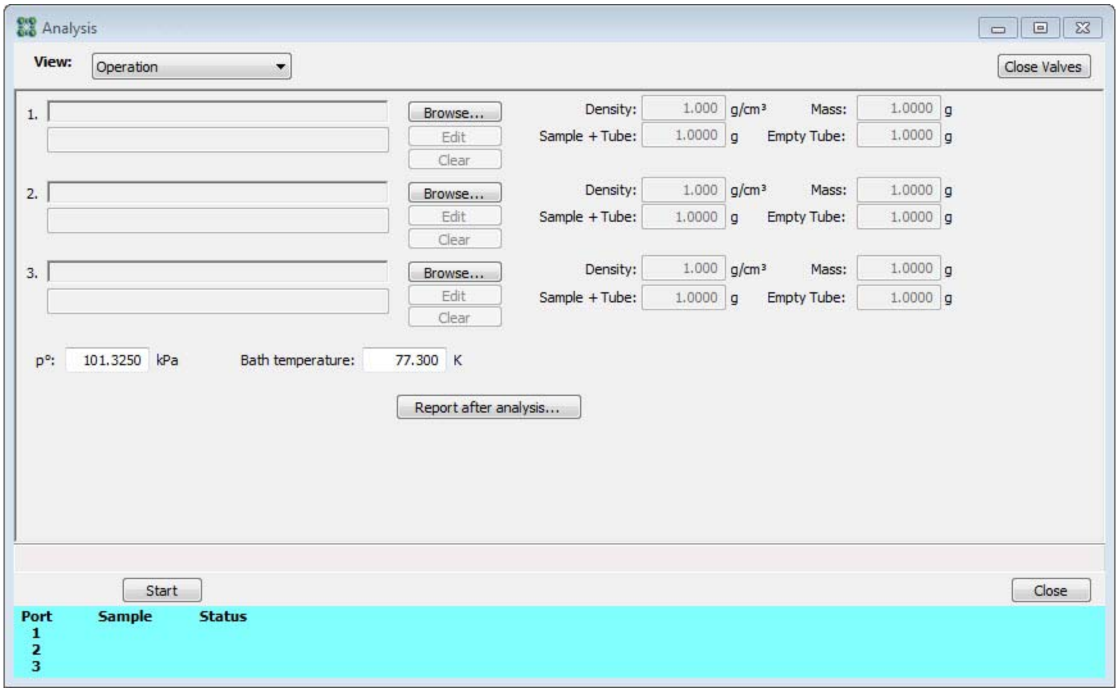
The following fields and buttons are common to many of the Unit menu options. References are made to these fields and buttons throughout this manual.

Field or Button	Description
Browse *button*	Click to search for a file. Select a file from either the **Name** column or from the library and click **Open** or double click the file name to open (or import) the file.
Cancel *button*	Discards any changes made to the screen or cancels the current process. On the **Analysis** window, at the prompt, choose the port(s) to cancel.
Close *button*	Click to close the active window.
Edit *button*	On the analysis window, click to edit the sample information file. Refer to **Sample Information Files**, page **3-9**.
OK *button*	Click to save and close the active window.
Start *button*	Click to start an analysis or calibration procedure.

Sample Analysis

Unit [n] > Sample Analysis

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



View: Operation

1. Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 1.0000 g Empty Tube: 1.0000 g

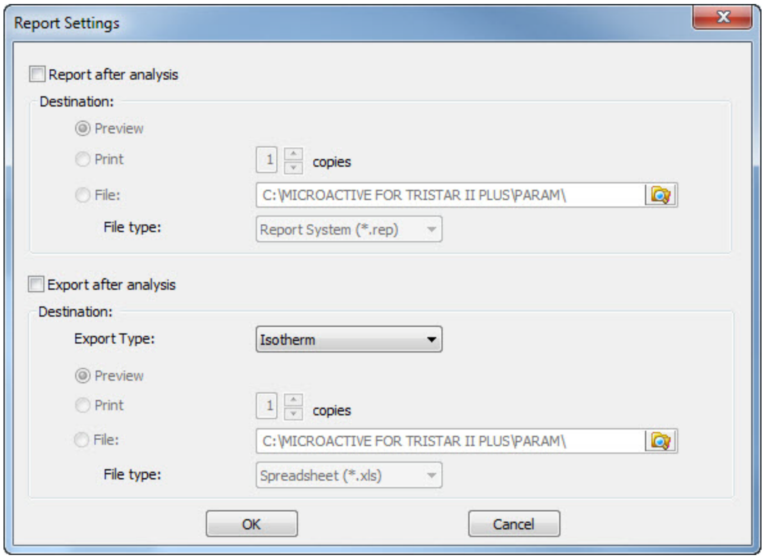
2. Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 1.0000 g Empty Tube: 1.0000 g

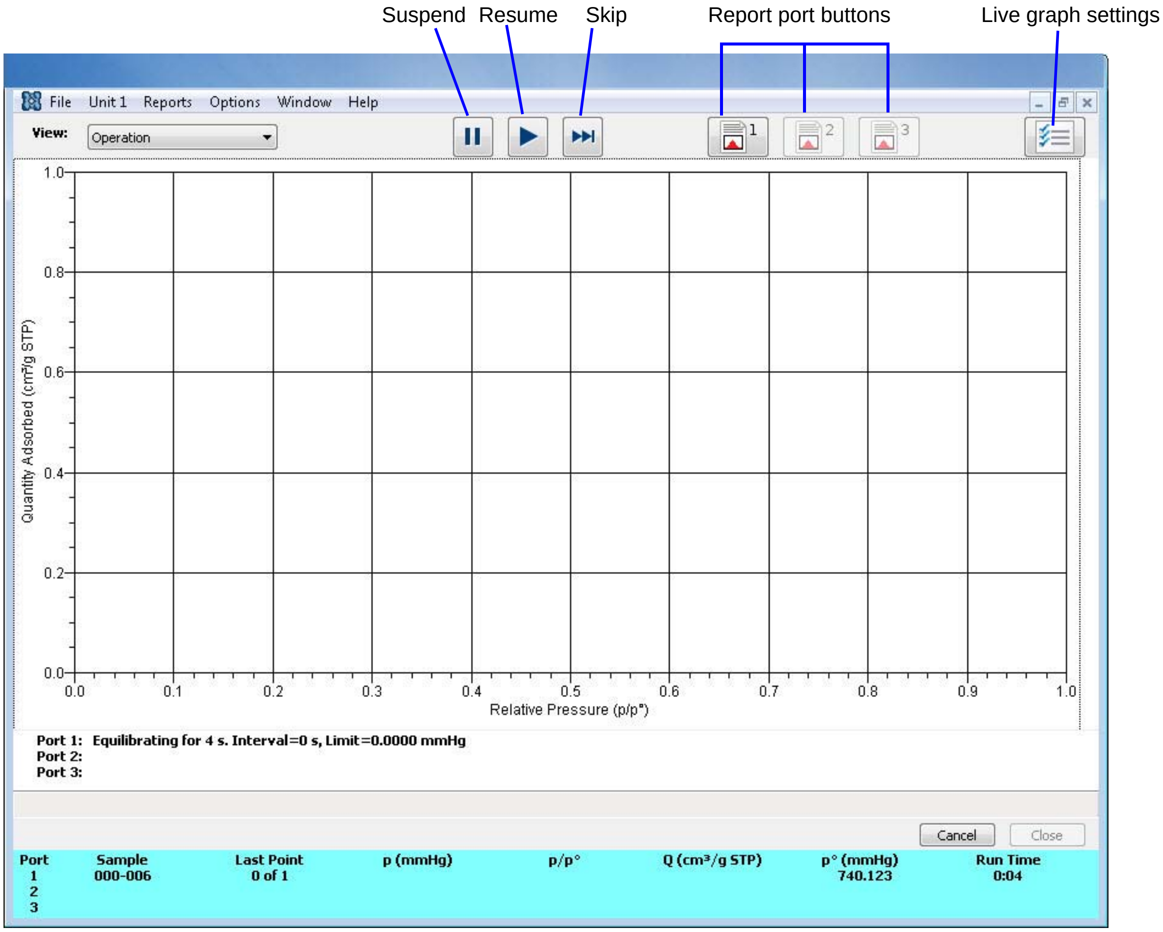
3. Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 1.0000 g Empty Tube: 1.0000 g

p°: 101.3250 kPa Bath temperature: 77.300 K

Port	Sample	Status
1		
2		
3		

Field or Button	Description
***View** dropdown list*	• **Operation** - use to display the current mode of operation. • **Instrument Log** - use to display recent analyses, calibrations, errors or messages. Refer to **Show Instrument Log**, page **4-19.** • **Instrument Schematic** - use to display a schematic of the analyzer system. Refer to **Show Instrument Schematic**, page **4-14**.
***Close Valves** button*	Click to close all valves on the unit.
***Browse** button*	Click to select a sample file to be used for analysis on the associated port. On Port 1, you can select up to three sample files. The files will be loaded into ports 1, 2, and 3 in the order they appear in the file selector.
***Edit** button*	Click to edit the selected sample file.
***Clear** button*	Click to clear all fields for this port.

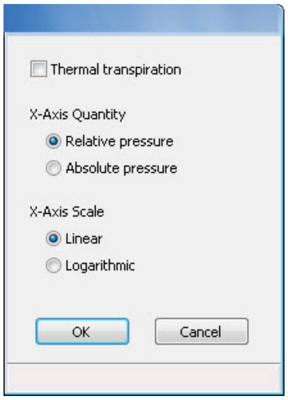
Field or Button	Description *(continued)*
Density / Mass / Sample + Tube / Empty Tube *text boxes*	Enter default values for the sample’s mass and density. Refer to **Mass group box**, page **3-12**.
p^o *text box*	Enabled if **Entered** is selected for the Po measurement for at least one file.
Bath temperature *text box*	Enter the temperature for the analysis bath.
Report after analysis *button*	Click to select a print destination. Report Settings ☐ Report after analysis Destination: ● Preview ○ Print 1 copies ○ File: C:\MICROACTIVE FOR TRISTAR II PLUS\PARAM\ File type: Report System (*.rep) ☐ Export after analysis Destination: Export Type: Isotherm ● Preview ○ Print 1 copies ○ File: C:\MICROACTIVE FOR TRISTAR II PLUS\PARAM\ File type: Spreadsheet (*.xls) OK Cancel • **Report after analysis** checkbox - use to send the report to the screen, printer, or file in either ASCII, .XLS, or REP format. • **Export after analysis** checkbox - use to export Isotherm data to the screen, printer, or file in either ASCII, or .XLS format. Refer to **Destination** group box in **New Sample**, page **3-5**.



Port 1: Equilibrating for 4 s. Interval=0 s, Limit=0.0000 mmHg
Port 2:
Port 3:

Port	Sample	Last Point	p (mmHg)	p/p°	Q (cm^3/g STP)	p° (mmHg)	Run Time
1	000-006	0 of 1				740.123	0:04
2							
3							

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

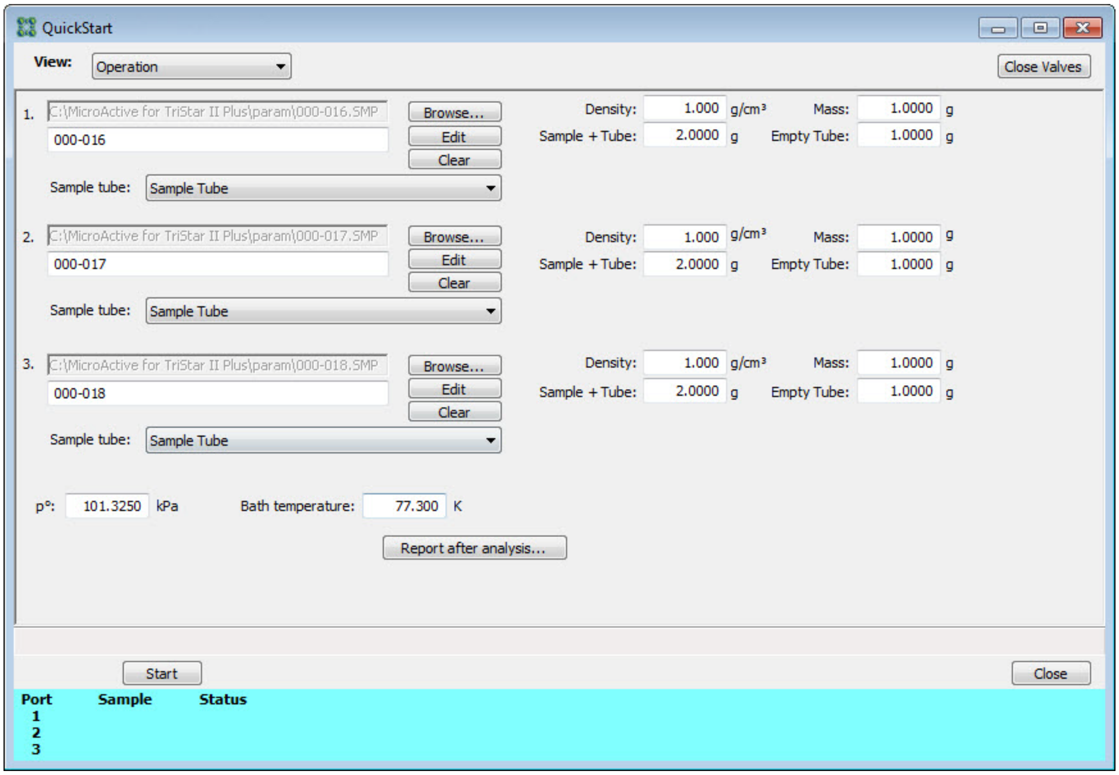
Field or Button	Description *(continued)*
Live Graph Settings *button*	Use to select Thermal transpiration, X-axis Quantity (relative or absolute pressure) and the X-Axis Scale (linear or logarithmic).  ☐ Thermal transpiration X-Axis Quantity ● Relative pressure ○ Absolute pressure X-Axis Scale ● Linear ○ Logarithmic
Status *window*	Displays the last point pressure and relative pressure for each port with varying numbers of digits after the decimal if 10 mmHg transducer is present on that port, as follows: P < 0.1: 6 digits, 0.1 <= P < 10: 4 digits, P >= 10: 2 digits. Relative pressure will show 3 more digits than absolute pressure.
Browse *button* ***Cancel*** *button* ***OK*** *button*	Refer to **Common Fields and Buttons - Unit Menu Options**, page **4-2**.

QuickStart Analysis

Unit [n] > QuickStart Analysis

Use to analyze up to three samples of the same type using the same analysis conditions, adsorptive properties, and report options. The **QuickStart Analysis** option automatically creates a sample information file for each port and assigns the default parameters. Edit the parameters of a file using the **Edit** button or select another file using the **Browse** button.

Additional analyses can be scheduled by clicking **Next** after the completion of the first set of analyses. The **Next** button appears after the first series of analyses is complete. Samples cannot be removed or added to ports until the full set of analyses has completed.



QuickStart

View: Operation

1. C:\MicroActive for TriStar II Plus\param\000-016.SMP
000-016
Sample tube: Sample Tube
Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 2.0000 g Empty Tube: 1.0000 g

2. C:\MicroActive for TriStar II Plus\param\000-017.SMP
000-017
Sample tube: Sample Tube
Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 2.0000 g Empty Tube: 1.0000 g

3. C:\MicroActive for TriStar II Plus\param\000-018.SMP
000-018
Sample tube: Sample Tube
Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 2.0000 g Empty Tube: 1.0000 g

p°: 101.3250 kPa Bath temperature: 77.300 K

Port	Sample	Status
1		
2		
3		

QuickStart Analysis fields and buttons are identical to the **Sample Analysis**, page **4-3** with the following exception:

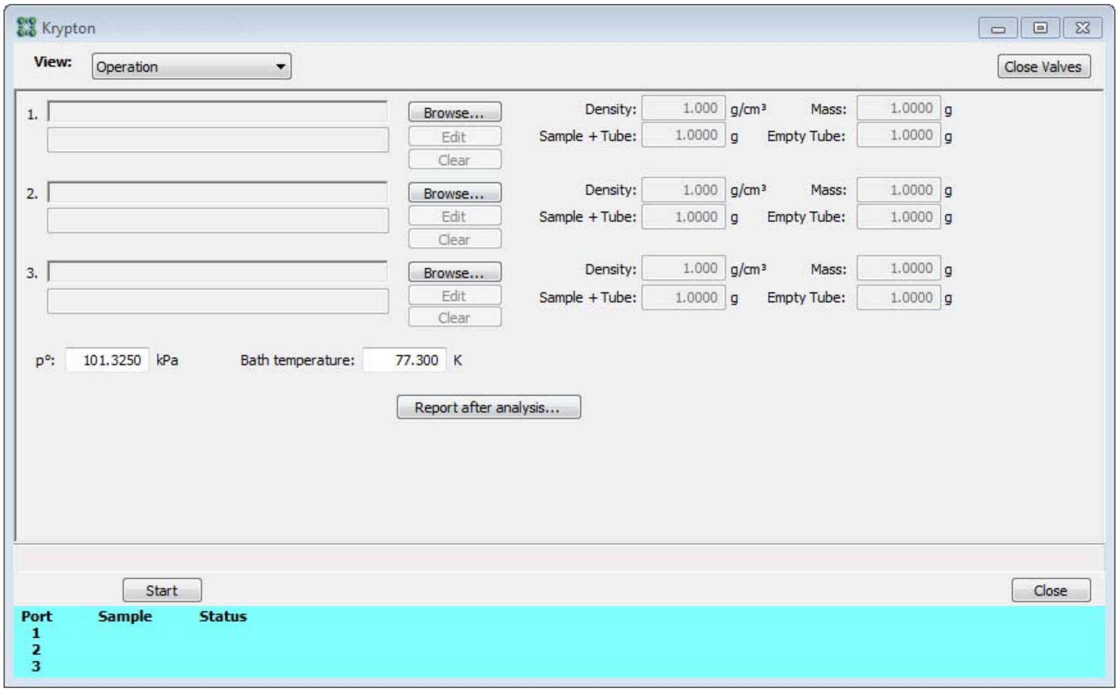
Field or Button	Description
Sample tube *button*	Click the dropdown arrow to select a sample tube file. This option is applicable only if the calculated method for measuring free space is selected for Analysis Conditions.

Krypton Analysis

Unit [n] > Krypton Analysis

Use to perform up to three simultaneous krypton analyses. Use the following guidelines for performing krypton analyses:

- All analyses must use krypton gas. An error message occurs if a gas other than krypton is specified.
- **From Psat tube** must be selected as the **Dosing Method** on Adsorptive Properties. An error message displays if **Normal** is selected as the Dosing Method. Samples cannot be removed from or added to ports until the full set of analyses has completed.



View: Operation

Density: 1.000 g/cm³ Mass: 1.0000 g
Sample + Tube: 1.0000 g Empty Tube: 1.0000 g

p°: 101.3250 kPa Bath temperature: 77.300 K

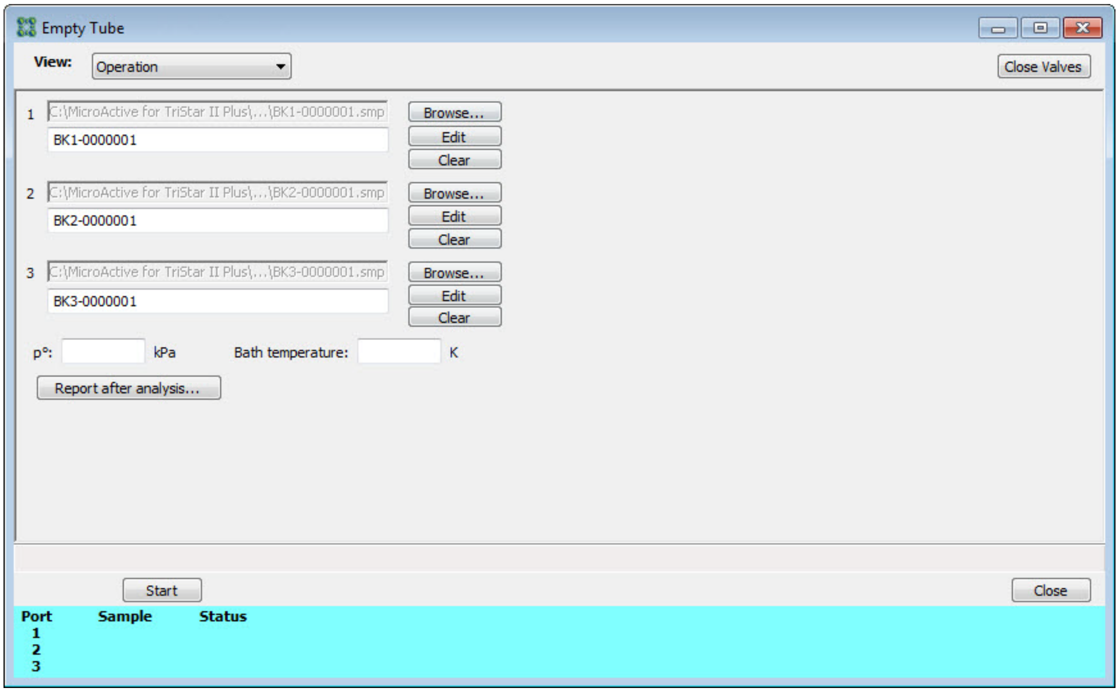
Port	Sample	Status
1		
2		
3		

Krypton Analysis fields and buttons are identical to the **Sample Analysis**, page **4-3**.

Empty Tube Analysis

Unit [n] > Empty Tube Analysis

Use to obtain sample tube parameters when using a calculated free space. An empty tube analysis can also be used for diagnostic purposes.



View: Operation

1 C:\MicroActive for TriStar II Plus\...\BK1-0000001.smp
BK1-0000001

2 C:\MicroActive for TriStar II Plus\...\BK2-0000001.smp
BK2-0000001

3 C:\MicroActive for TriStar II Plus\...\BK3-0000001.smp
BK3-0000001

p°: kPa Bath temperature: K

Port	Sample	Status
1		
2		
3		

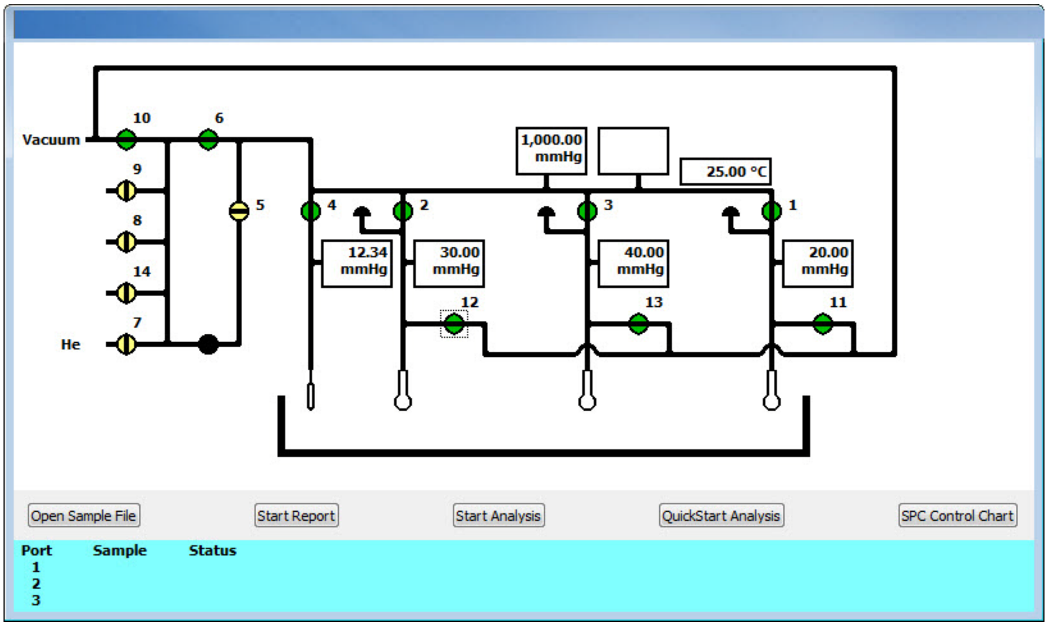
Empty Tube Analysis fields and buttons are identical to the **Sample Analysis**, page **4-3**.

Enable Manual Control

Unit [n] > Enable Manual Control

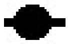
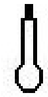
Use to enable the manual control of certain system valves and elevator components. When this option is enabled, a checkmark appears to the left of the menu item.

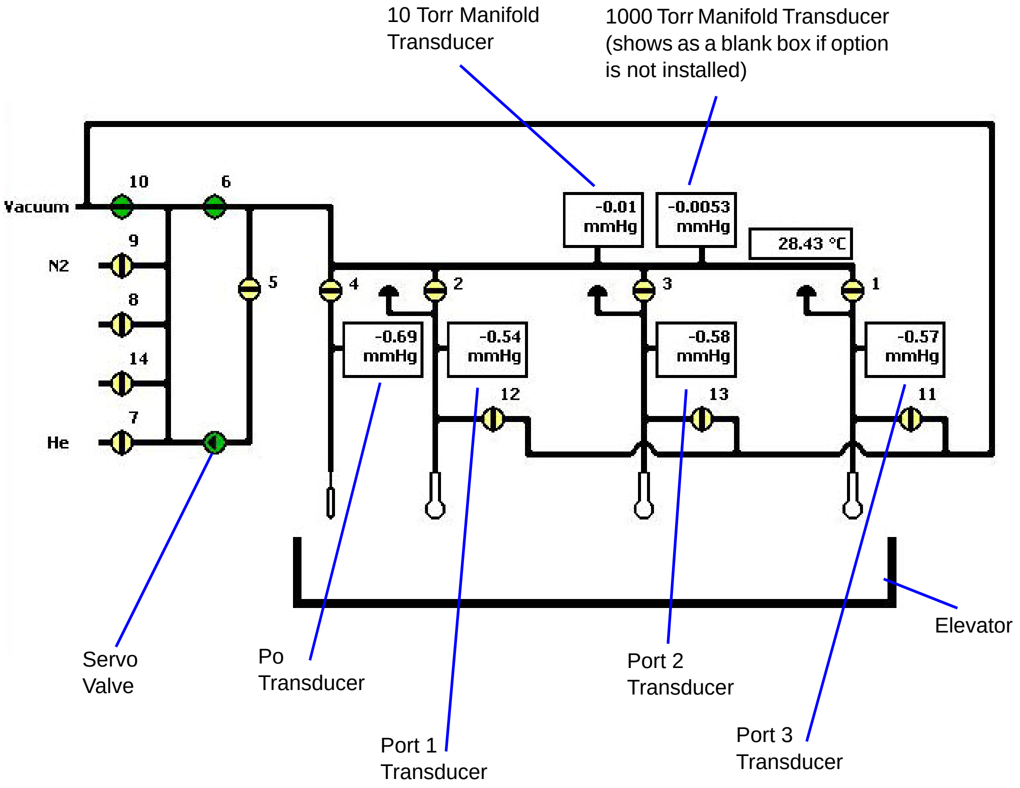
If the following instrument schematic is not immediately visible, go to ***Unit [n] > Show Instrument Schematic.***



Port	Sample	Status
1		
2		
3		

Instrument Schematic Components

Component	Description
	**Valve** - open. The icon color changes to green when open.
	**Valve** - closed. The icon color changes to yellow when closed.
	**Servo Valve** - open
	**Servo Valve** - closed
	Elevator
	**Sample tube** - cannot be manually controlled.



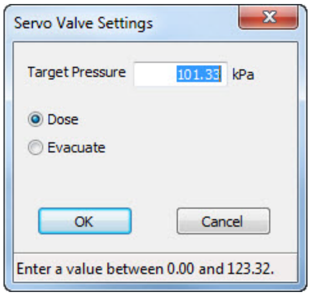
System Valves

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

Valves	Description
1 - 3	Sample ports
4	Po port
5	Servo Valve
6	Isolation Valve
7, 8, 9, 14	Inlet Valves
10	Vacuum
11-13	Unrestricted vacuum for sample ports
Unmarked	Servo

Instrument Schematic Shortcut Menus

Each manually controlled component has a shortcut menu displaying the operations available for that particular component, such as, Open, Close, Pulse. To access the shortcut menu, hover the mouse cursor over the component and right click. Applicable only if ***Unit [n] > Enable Manual Control*** is selected..

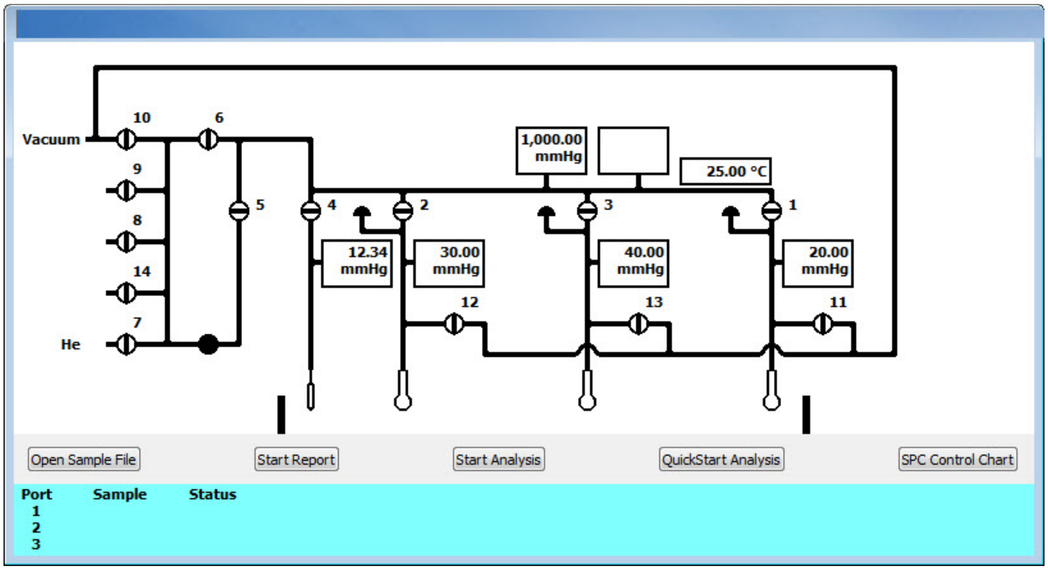
Shortcut Icon	Description
☝	When the cursor changes to this icon, right click to display options for the selected component: **Open** - opens the selected valve. The valve symbol changes to green. An alternate method is to either double click the valve or select the valve and press the keyboard spacebar to turn it off/on. **Close** - closes the selected valve. The valve symbol changes to yellow. An alternate method is to either double click the valve or select the valve and press the keyboard spacebar to turn it off/on. **Pulse** - use to quickly turn the valve on and off allowing the operation to proceed in small increments. • For Servo Valve: **Set** - use to set the servo valve target pressure and to Dose or Evacuate.  Target Pressure 101.33 kPa ● Dose ○ Evacuate Enter a value between 0.00 and 123.32. **Open** - opens the servo valve. The valve symbol changes to green. **Close** - closes the servo valve. The valve symbol changes to solid black.
⇕	When the cursor changes to this icon, right click to display options for the elevator: • **Raise** - raises the elevator. Select **Raise** and press the keyboard space bar to raise the elevator. • **Lower** - lowers the elevator. Select **Lower** and press the keyboard space bar to lower the elevator. • **Stop** - use to stop the elevator from raising or lowering.

Show Instrument Schematic

Unit [n] - Show Instrument Schematic

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

Refer to **Instrument Schematic Components**, page **4-11** for details on this schematic.



Port	Sample	Status
1		
2		
3		

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

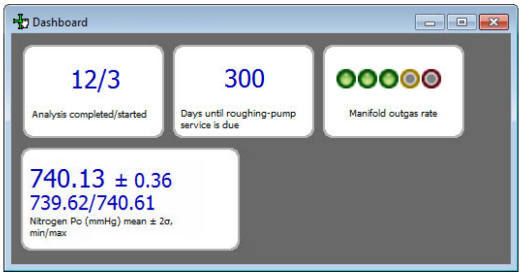
Show Dashboard

Unit [n] > Show Dashboard

The dashboard displays the following:

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

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



12/3
Analysis completed/started

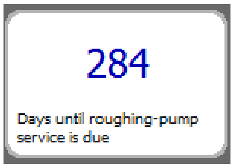
300
Days until roughing-pump service is due

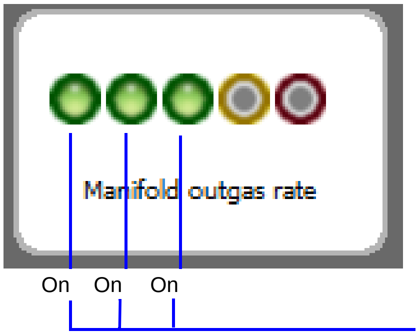
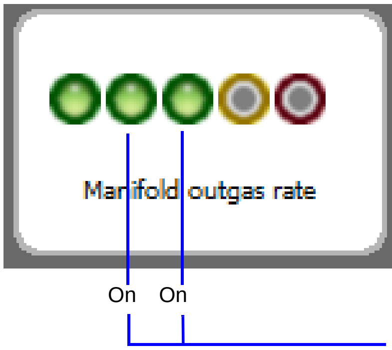
●●●●●
Manifold outgas rate

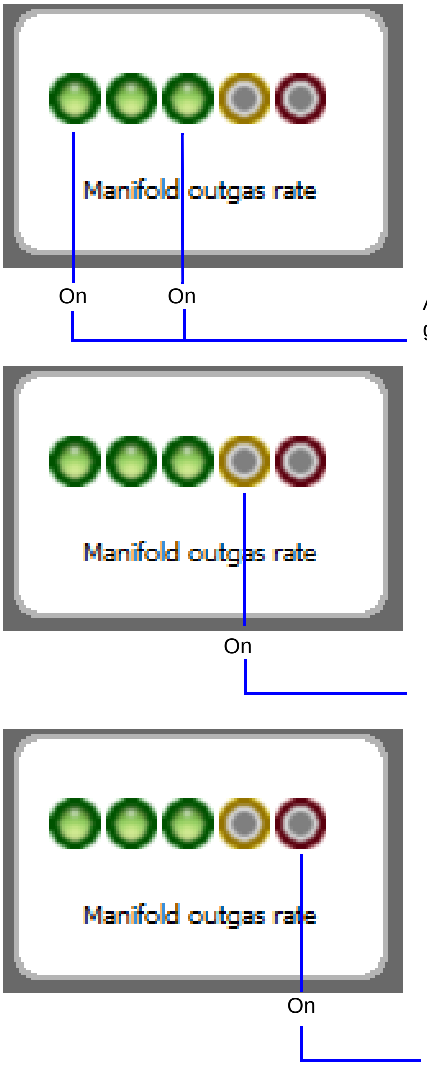
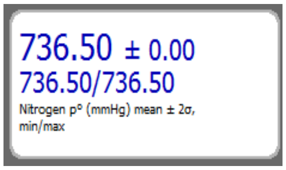
740.13 ± 0.36
739.62/740.61
Nitrogen Po (mmHg) mean ± 2σ, min/max

Red numbers on the dashboard require attention.

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

Gauge	Description
Analyses completed/started  26/47 Analysis completed/started	Displays *N/M* where *N* is the number of analysis that have finished data collection and *M* is the number of analyses that have been started. Analyses canceled or terminated by errors before the termination stage starts are not counted as completed.
Days until roughing-pump service is due  284 Days until roughing-pump service is due	Yearly maintenance is recommended. The number of days until the anniversary of the last pump maintenance are shown. The displayed value is updated at least once per day and when the maintenance time is reset. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.

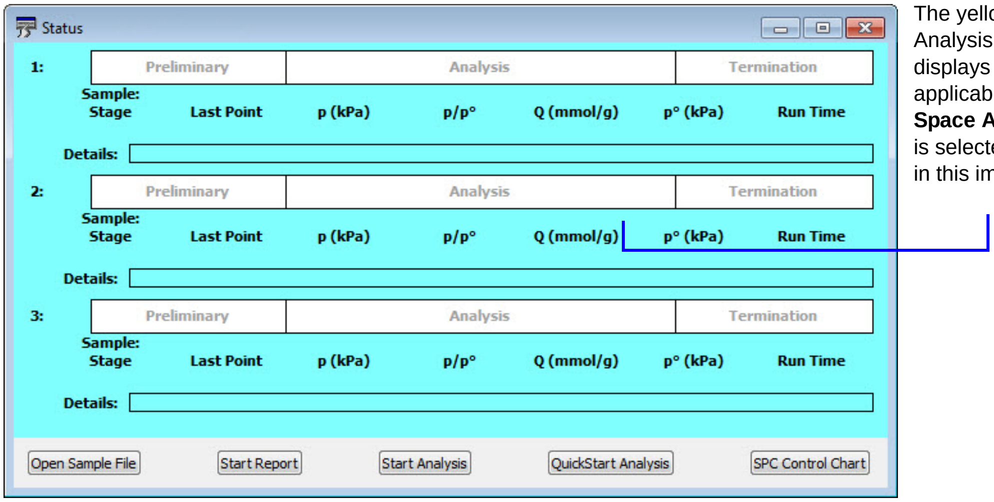
Gauge	Description *(continued)*
Manifold outgas rate	Provides the qualitative indication of the outgas rate in the dosing manifold. LED images constitute a bidirectional bar graph of the outgas rate. The gauge is updated after each outgas rate measurement. To reset these numbers right click on the window then click the **Reset**.  The three green LEDs are lit if the outgas rate is below 30% of the outgas rate limit.  At 30%, the left LED turns off.

Gauge	Description *(continued)*
Manifold outgas rate *(continued)*	 At 60%, the center green LED turns off. At 90%, three green LEDs turn off and the yellow LED is turned on. At 110% and above, only the red LED is lit and attention is required.
Nitrogen Po  736.50 ± 0.00 736.50/736.50 Nitrogen p° (mmHg) mean ± 2σ, min/max	Displays statistics of the saturation pressures measured with nitrogen gas at liquid nitrogen temperatures. The mean, two-sigma, minimum, and maximum values display. The gauge is updated when a Po is logged with nitrogen as the adsorptive and a bath temperature of 77±2 K. To reset these numbers right click on the window then click the **Reset**

Show Status

Unit [n] > Show Status

Use to show the current status for each port.



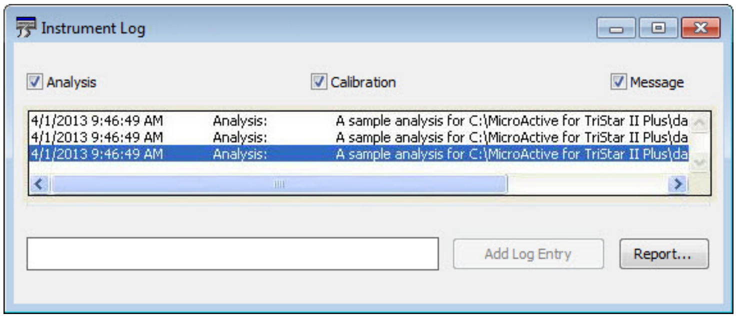
The yellow Post Analysis Free Space displays when applicable and if **Free Space After Analysis** is selected. (Not shown in this image.)

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

Show Instrument Log

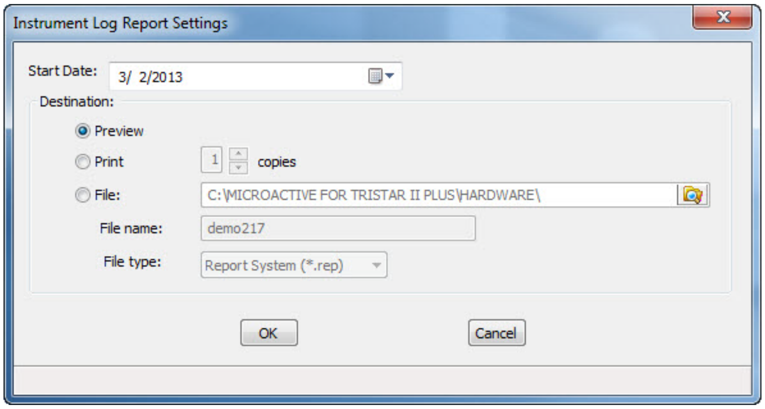
Unit [n] > Show Instrument Log

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



```
4/1/2013 9:46:49 AM      Analysis:        A sample analysis for C:\MicroActive for TriStar II Plus\da
4/1/2013 9:46:49 AM      Analysis:        A sample analysis for C:\MicroActive for TriStar II Plus\da
4/1/2013 9:46:49 AM      Analysis:        A sample analysis for C:\MicroActive for TriStar II Plus\da
```

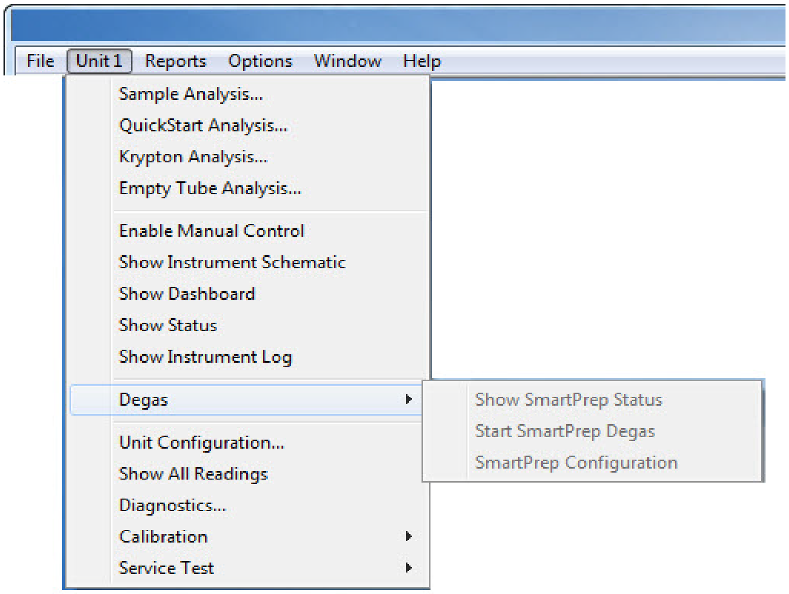
Field or Button	Description
Analysis ***Calibration*** ***Message***	Select the logs to display.
Include Message Number	Use to generate a log report containing all the instances of one message number making it easier to look at the history of a particular quantity located in the log.
Add Log Entry *button*	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.

Field or Button	Description *(continued)*
***Report** button*	Click to display the **Instrument Log Report Settings** window to specify report output options.  Instrument Log Report Settings Start Date: 3/ 2/2013 Destination: ● Preview ○ Print 1 copies ○ File: C:\MICROACTIVE FOR TRISTAR II PLUS\HARDWARE\ File name: demo217 File type: Report System (*.rep) • **Start Date** - click to display a calendar and select the start date for the report. • **Destination** group box: **Preview** - sends the report to the screen. Click **Print** on the report screen to send the file to the printer. **Print** - sends the report to the default printer. **Copies** - select the number of copies to print. This field is only enabled when **Print** is selected. **File** - saves the report as a file. Click the **Browse** icon to the right of the text field to select the directory where the new report file will be stored. Enter the new file name in the **File name** text box. **File Type** - use to save the new file with a .TXT, .XLS or .REP file extension. This field is only enabled when **File** is selected. – **.REP** (Report system) - saves the report in a format that can be opened with any MicroActive program. – **.TXT** (ASCII text) - saves the report as a common machine language file. – **.XLS** (Spreadsheet file) - saves the report in a spreadsheet format.

Degas

If a SmartPrep is not connected to the analyzer, the menu options in this section are disabled.

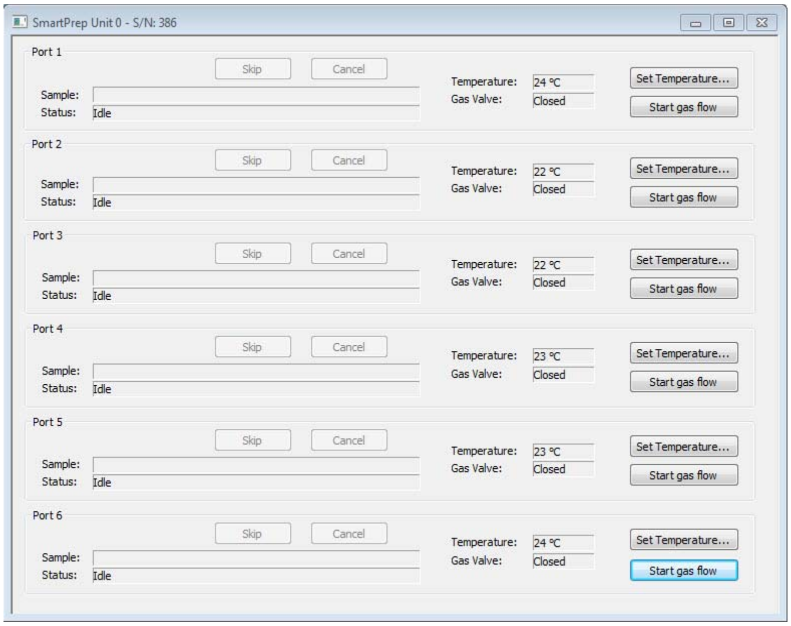
Unit > Degas



Show SmartPrep Status

Unit > Degas > Show SmartPrep Statu

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



SmartPrep Unit 0 - S/N: 386

Port 1
Sample:
Status: Idle
Temperature: 24 °C
Gas Valve: Closed

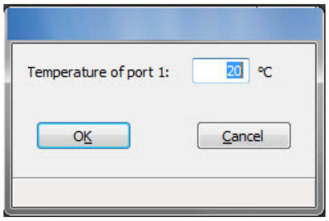
Port 2
Sample:
Status: Idle
Temperature: 22 °C
Gas Valve: Closed

Port 3
Sample:
Status: Idle
Temperature: 22 °C
Gas Valve: Closed

Port 4
Sample:
Status: Idle
Temperature: 23 °C
Gas Valve: Closed

Port 5
Sample:
Status: Idle
Temperature: 23 °C
Gas Valve: Closed

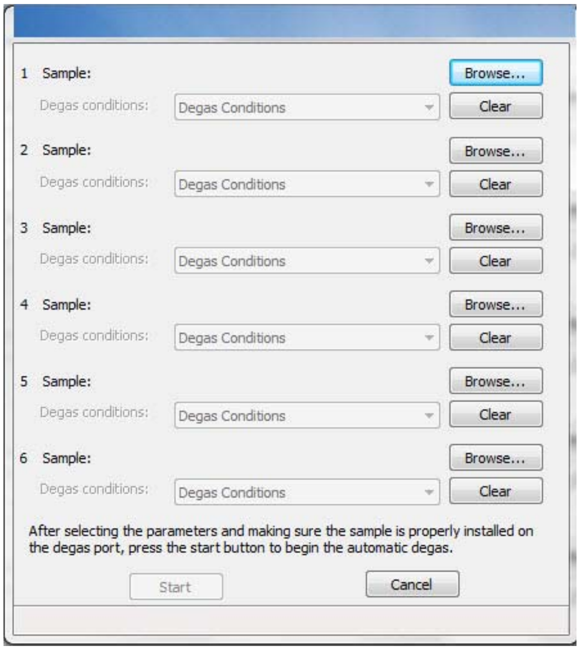
Port 6
Sample:
Status: Idle
Temperature: 24 °C
Gas Valve: Closed

Field or Button	Description
Skip *button*	Use to skip the degassing of the selected sample.
Cancel *button*	Use to cancel the degassing of the selected sample.
Set Temperature *button*	Use to set the temperature of the selected port.  Temperature of port 1: 20 °C
Stop Gas Flow *button*	Stops the gas flow to the selected port.

Start SmartPrep Degas

Unit *>* ***Degas*** *>* ***Start SmartPrep Degas***

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



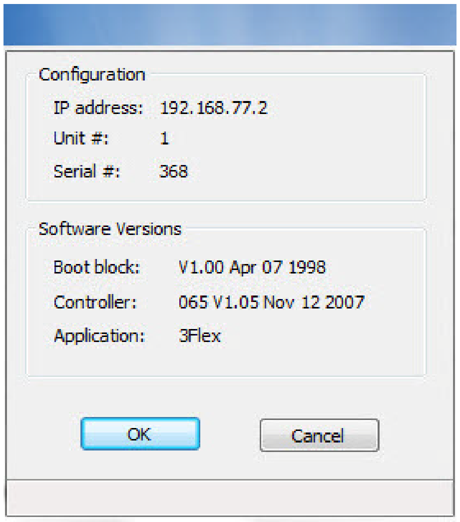
After selecting the parameters and making sure the sample is properly installed on the degas port, press the start button to begin the automatic degas.

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

SmartPrep Configuration

Unit [n] > Degas > SmartPrep Configuration

Displays the SmartPrep configuration and software versions.



Configuration

IP address: 192.168.77.2

Unit #: 1

Serial #: 368

Software Versions

Boot block: V1.00 Apr 07 1998

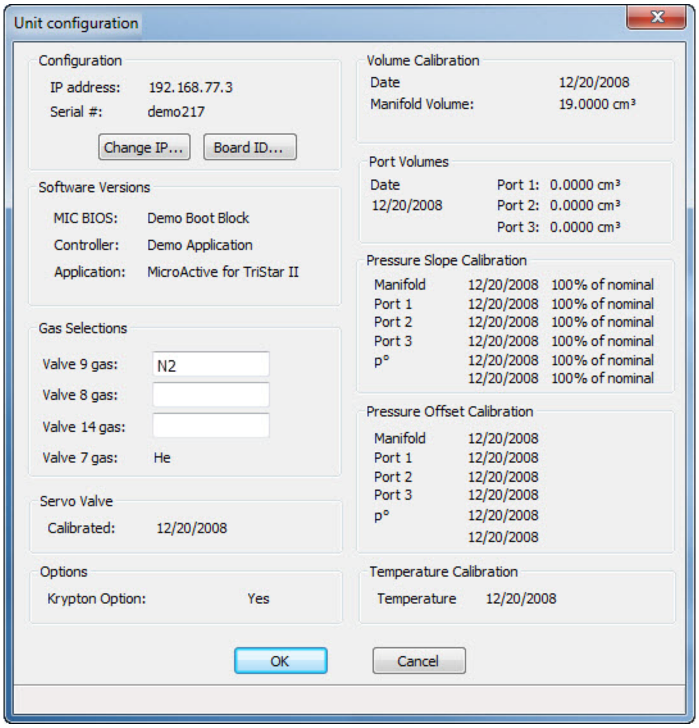
Controller: 065 V1.05 Nov 12 2007

Application: 3Flex

Unit Configuration

Unit [n] > Unit Configuration

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



Configuration
IP address: 192.168.77.3
Serial #: demo217

Software Versions
MIC BIOS: Demo Boot Block
Controller: Demo Application
Application: MicroActive for TriStar II

Gas Selections
Valve 9 gas: N2
Valve 8 gas:
Valve 14 gas:
Valve 7 gas: He

Servo Valve
Calibrated: 12/20/2008

Options
Krypton Option: Yes

Volume Calibration
Date 12/20/2008
Manifold Volume: 19.0000 cm³

Port Volumes
Date 12/20/2008
Port 1: 0.0000 cm³
Port 2: 0.0000 cm³
Port 3: 0.0000 cm³

Pressure Slope Calibration

Manifold	12/20/2008	100% of nominal
Port 1	12/20/2008	100% of nominal
Port 2	12/20/2008	100% of nominal
Port 3	12/20/2008	100% of nominal
p°	12/20/2008	100% of nominal
	12/20/2008	100% of nominal

Pressure Offset Calibration

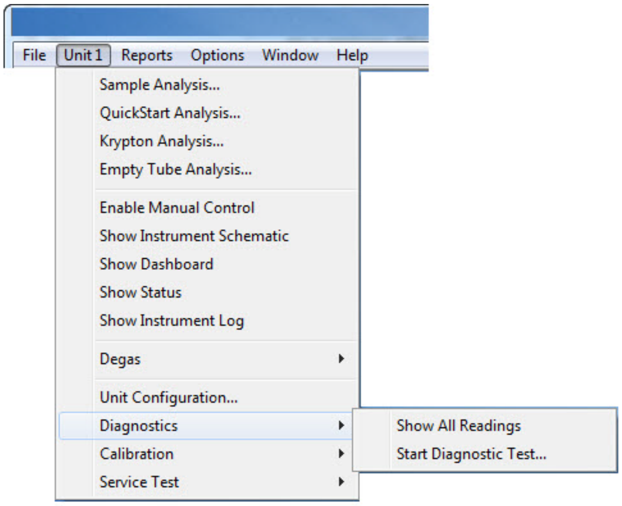
Manifold	12/20/2008
Port 1	12/20/2008
Port 2	12/20/2008
Port 3	12/20/2008
p°	12/20/2008
	12/20/2008

Temperature Calibration
Temperature 12/20/2008

Field or Button	Description
Configuration *group box*	Displays the IP address used by the analysis program and the serial number of the selected analyzer. • **Change IP -** click to display the **Unit IP Setup** window. The IP address and Subnet mask assigned during installation display. Do not edit these fields unless instructed by a Micromeritics service representative. • **Board ID -** click to read the board ID. The parameters on this window cannot be edited.
Software Versions *group box*	Displays the software versions of the MIC BIOS, controller, and analysis program.
Gas Selections *group box*	In the text boxes, enter the mnemonics for the analysis gases attached to inlet valves.
Options	Displays options installed on the instrument.

Diagnostics

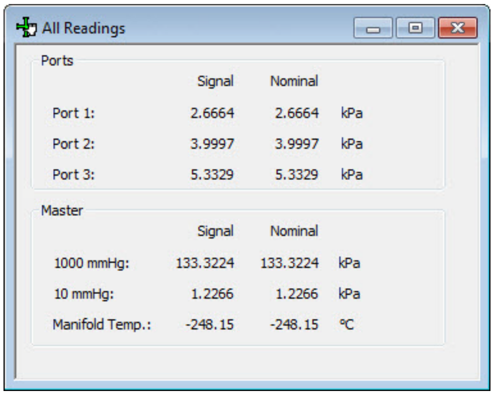
Unit [n] > Diagnostics



Show All Readings

Unit [n] > Diagnostics > Show All Readings

The All Readings screen displays the calibrated and nominal readings of all sensors in the system.



Ports

	Signal	Nominal	
Port 1:	2.6664	2.6664	kPa
Port 2:	3.9997	3.9997	kPa
Port 3:	5.3329	5.3329	kPa

Master

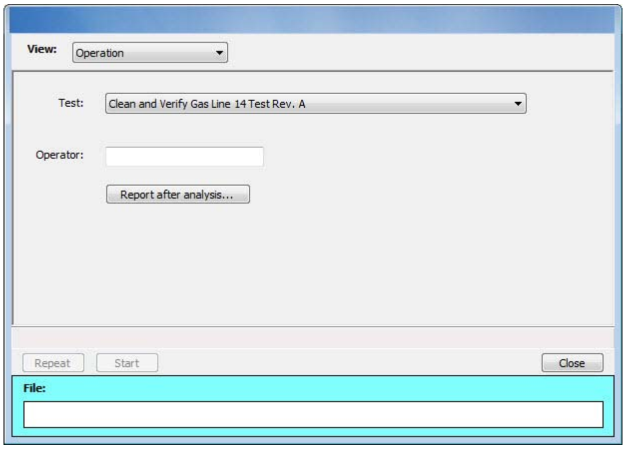
	Signal	Nominal	
1000 mmHg:	133.3224	133.3224	kPa
10 mmHg:	1.2266	1.2266	kPa
Manifold Temp.:	-248.15	-248.15	°C

Diagnostic Test

Unit [n] > Diagnostics > Start Diagnostic Test.

Use to clean and verify gas lines when connecting or changing a gas. Refer to **Cleaning and Verifying the Gas Line**, page **7-21** for detailed instructions. Also used to perform diagnostic tests requested by a service representative.

This option is typically used and/or requested by a service representative. These tests create and save files to the **TriStar II 3020\Service\USERDIAG** directory. The service representative may request an emailed or faxed copy of the file for diagnostic purposes..


View: Operation

Test: Clean and Verify Gas Line 14 Test Rev. A

Operator:

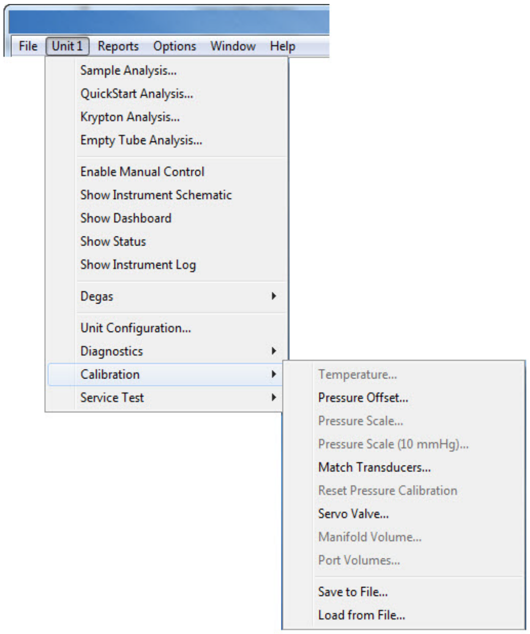
Field or Button	Description
***View** dropdown list*	• **Operation -** use to display the current mode of operation. • **Instrument Log -** use to display recent analyses, calibrations, errors or messages. Refer to **Show Instrument Log**, page **6-14.** • **Instrument Schematic -** use to display a schematic of the analyzer system. **Show Instrument Schematic**, page **6-12**.
***Test** dropdown list*	Select the diagnostic test to be performed. • Degas Heat Test Rev. *[n]* • Clean and Verify Gas Line # Test Rev. *[n]* • Manifold Heat Test Rev. *[n]* • Ports Leak Test Rev. *[n]* • System Operation Verification Rev. C
Operator	Enter information to identify the person running the service test.
Estimated time (min.)	Approximate time for test completion.

Field or Button	Description *(continued)*
Report after test	Select to automatically generate reports to the selected destination when the test is complete.
***Repeat** button*	Repeats the selected diagnostic test.
***Next** button*	Starts the next test.
***Start** button*	Starts the selected diagnostic test.
***Close** button*	Closes the window.

Calibration

Unit [n] > Calibration

Use to perform system calibrations. Disabled calibration options can be accessed only with the assistance of an authorized Micromeritics service representative.



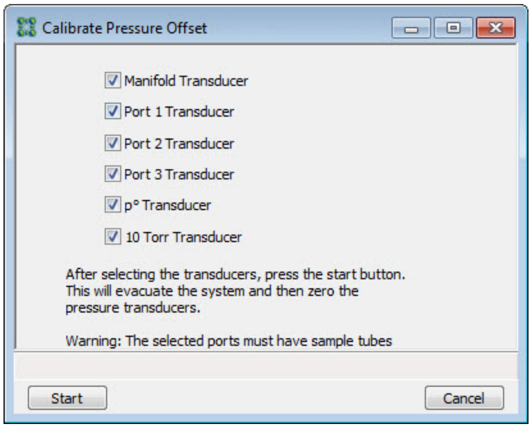
Information on the following items are not available in this manual. They are enabled when in Service Test Mode only.

- Temperature
- Pressure Scale
- Reset Pressure Calibration
- Manifold Volume
- Port Volumes

Pressure Offset

Unit [n] > Calibration > Pressure Offset

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



☑ Manifold Transducer
☑ Port 1 Transducer
☑ Port 2 Transducer
☑ Port 3 Transducer
☑ p° Transducer
☑ 10 Torr Transducer

After selecting the transducers, press the start button. This will evacuate the system and then zero the pressure transducers.

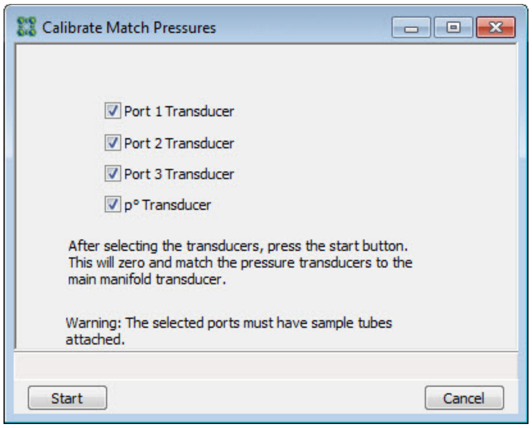
Warning: The selected ports must have sample tubes

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

Match Transducers

Unit [n]** > **Calibration** > **Match Transducers

Use to evacuate the system and zero the pressure transducers, then adjust the scale to match them to the manifold transducer near full scale pressure. In order to perform this procedure, sample tubes must be attached to each port.



☑ Port 1 Transducer
☑ Port 2 Transducer
☑ Port 3 Transducer
☑ p° Transducer

After selecting the transducers, press the start button. This will zero and match the pressure transducers to the main manifold transducer.

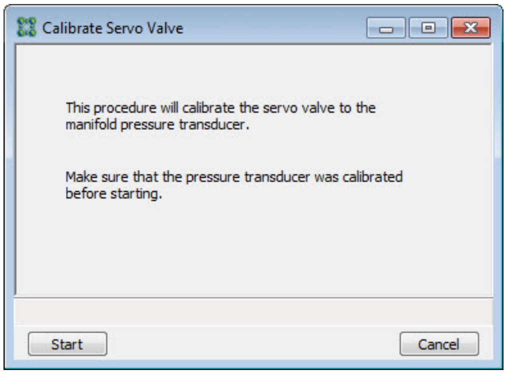
Warning: The selected ports must have sample tubes attached.

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

Servo Valve

Unit [n]* > *Calibration* > *Servo Valve

Use to calibrate the servo valve to the manifold pressure transducer. The servo valve should always be recalibrated after a pressure calibration has been performed. The pressure transducer should be calibrated before starting this calibration procedure. Refer to **Servo Valve**, page **7-27**.



This procedure will calibrate the servo valve to the manifold pressure transducer.

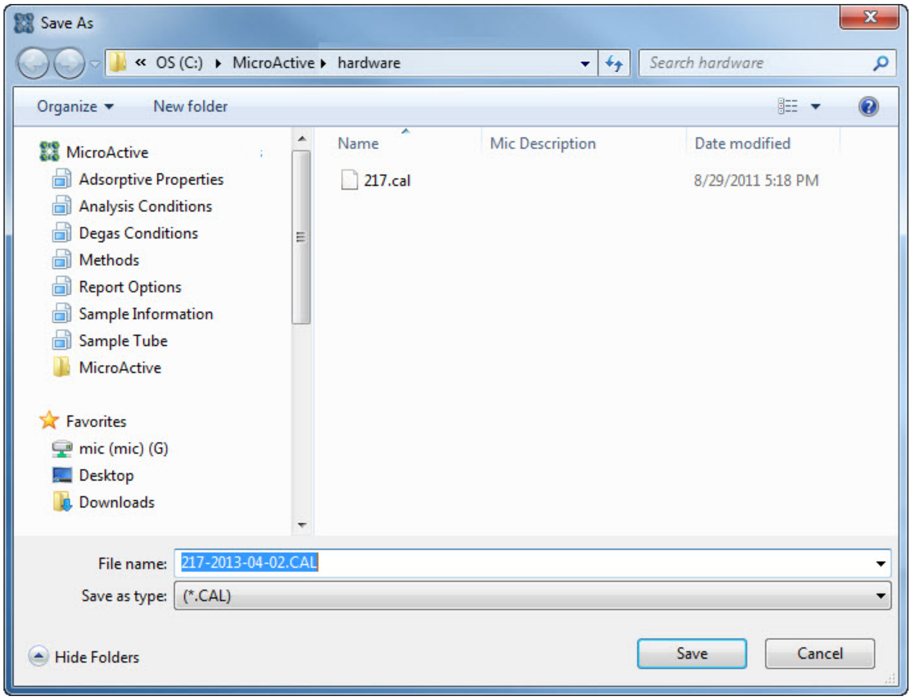
Make sure that the pressure transducer was calibrated before starting.

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

Save to File

Unit [n] > Calibration > Save to File

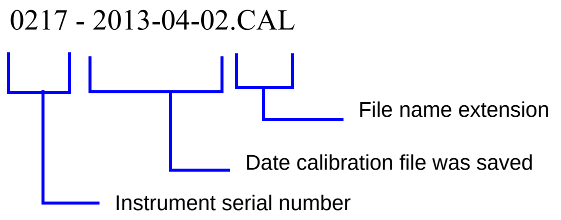
Use to save the current calibration settings to a backup file which can later be reloaded using ***Unit [n] > Calibration > Load from File*** menu option.



Name	Mic Description	Date modified
217.cal		8/29/2011 5:18 PM

File name: 217-2013-04-02.CAL

Save as type: (*.CAL)

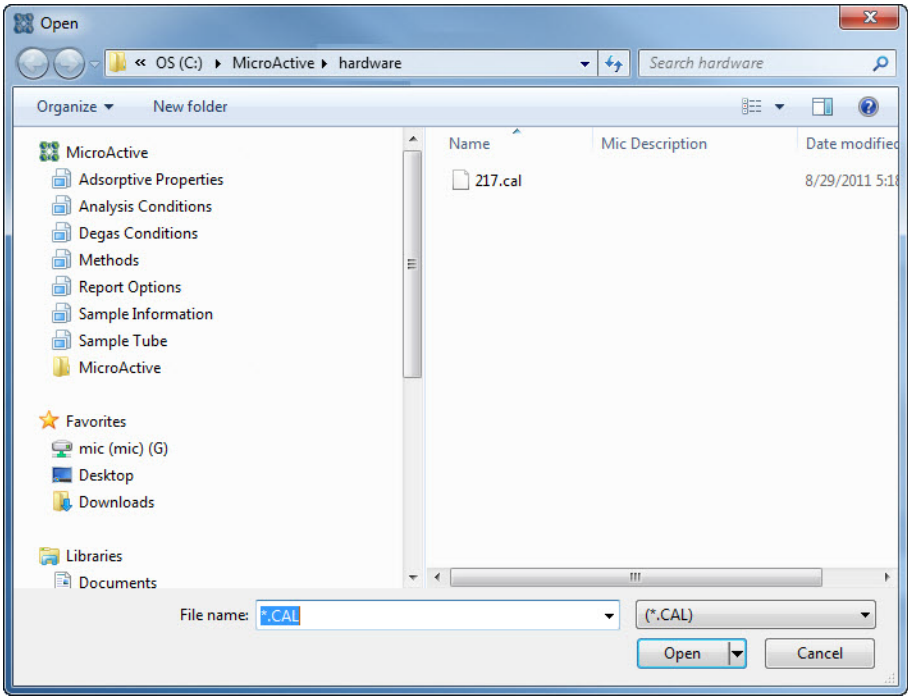
Field or Button	Description
File name text box	The default file naming convention for calibration files can be used or the filename can be changed. The default file name is interpreted as: 

Load from File

Unit [n] > Calibration > Load from File

Use to load a previously saved calibration file.

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



File name: *.CAL

(*.CAL)

Changing the calibration may affect the instrument’s performance.

Service Test

Unit [n] > Service Test

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

This option is enabled only when the analysis program is operating in Service Test mode. Refer to **Service Test Mode**, page **6-5**.

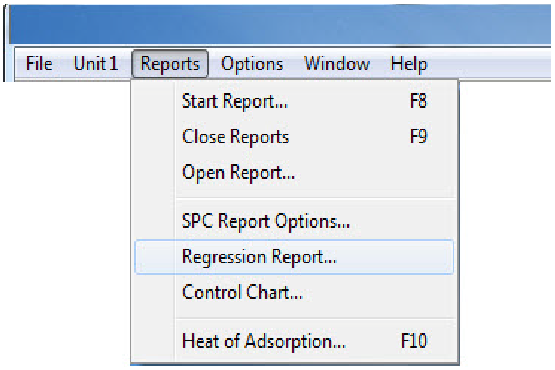
5. REPORTS MENU

Introduction

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

Reports can be generated for data:

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



Common Fields and Buttons - Reports Menu Options

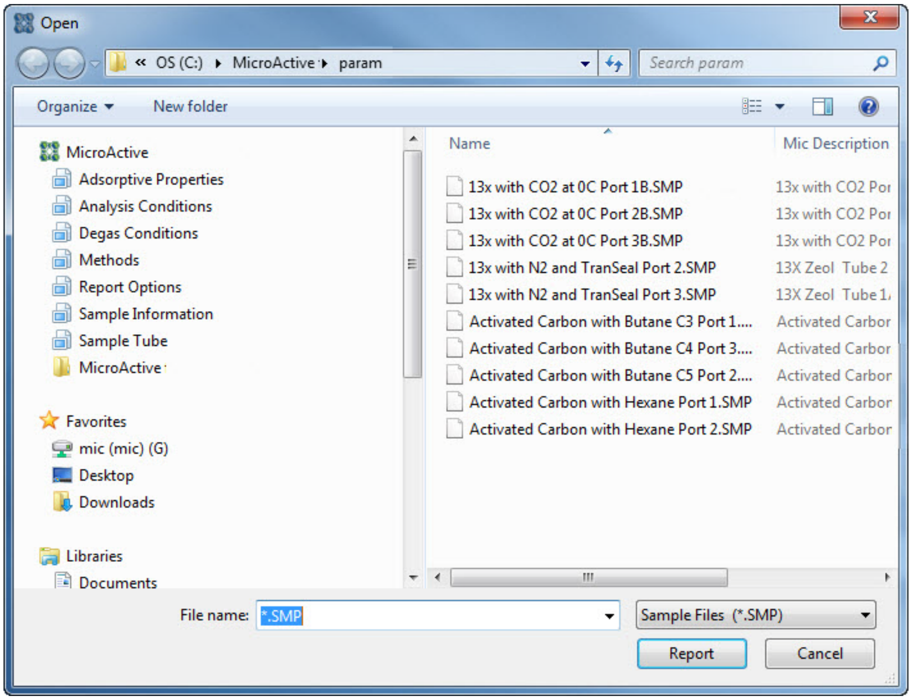
The following fields and buttons are common to many of the report files. References are made to these fields and buttons throughout this chapter.

Field or Button	Description
Autoscale *checkbox*	When enabled on report parameters screens, allows the x- and y-axes to be scaled automatically. **Autoscale** means that the x- and y- ranges will be set so that all the data is shown. If **Autoscale** is not selected, the entered range is used.
Axis Range	On report parameters screens, the **From / To** fields are enabled when **Autoscale** options are not selected. Enter the starting and ending values for the x- and/or y-axes.
Browse *button*	Click to locate another file.
Cancel *button*	Closes the window and cancels any unsaved changes.

Field or Button	Description *(continued)*
Destination *group box*	• **Preview** - sends the file to the screen. Click **Print** on the report screen to send the file to the printer. • **Print** - sends the file to the default printer. • **Copies** - select the number of copies to print. This field is only enabled when **Print** is selected.
Destination *group box (continued)*	• **File Type** - use to save the new file with a .TXT, .XLS or .REP file extension. This field is only enabled when **File** is selected. – **.REP** (Report system) - saves the report in a format that can be opened with any MicroActive program. – **.TXT** (ASCII text) - saves the report as a common machine language file. – **.XLS** (Spreadsheet file) - saves the report in a spreadsheet format.
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. Multiple files can be selected by holding down the **Ctrl** key on the keyboard while selecting multiple files.
From / To *text boxes*	Enter the **From** and **To** range for autoscaling the x- and/or y-axes.
Name column	Displays a list of files in the selected directory.
OK *button*	Click to save and close the active window.
Open *button*	Select a file from either the **Name** column or from the library. Click **Open** to open the file.
Report *button*	Click to generate the report.
Save *button*	Saves the active file.

Start Report

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



Name	Mic Description
13x with CO2 at 0C Port 1B.SMP	13x with CO2 Por
13x with CO2 at 0C Port 2B.SMP	13x with CO2 Por
13x with CO2 at 0C Port 3B.SMP	13x with CO2 Por
13x with N2 and TranSeal Port 2.SMP	13X Zeol Tube 2
13x with N2 and TranSeal Port 3.SMP	13X Zeol Tube 1,
Activated Carbon with Butane C3 Port 1....	Activated Carbor
Activated Carbon with Butane C4 Port 3....	Activated Carbor
Activated Carbon with Butane C5 Port 2....	Activated Carbor
Activated Carbon with Hexane Port 1.SMP	Activated Carbor
Activated Carbon with Hexane Port 2.SMP	Activated Carbor

File name: *.SMP

Sample Files (*.SMP)

Use to generate a report on a sample analysis.

Close Reports

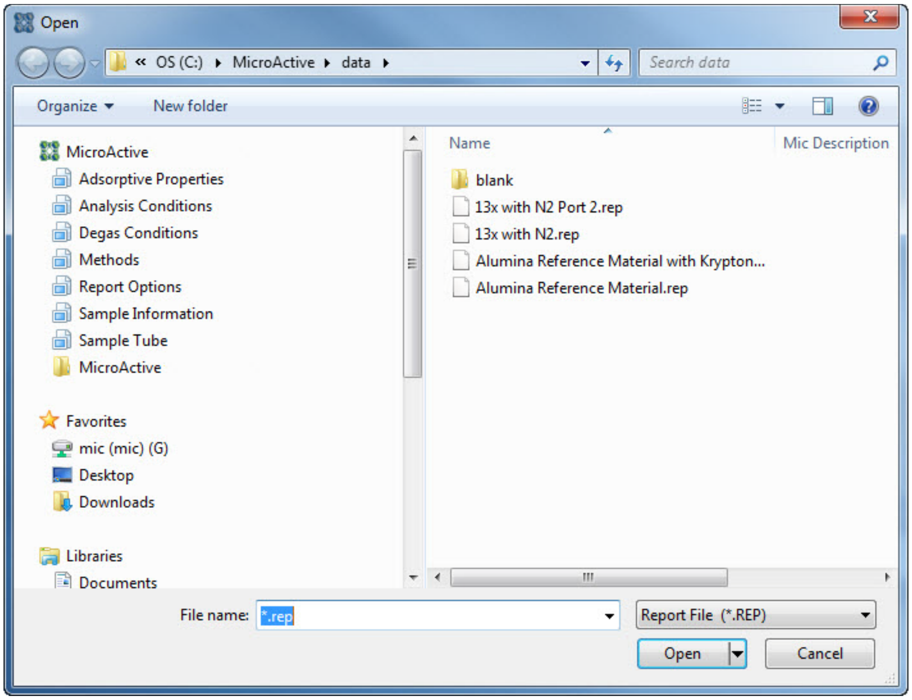
Reports > ***Close Reports*** (or use the **F9** keyboard shortcut)

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

Open Report

***Reports** > **Open Report** > [file}*

Use to open a saved report.



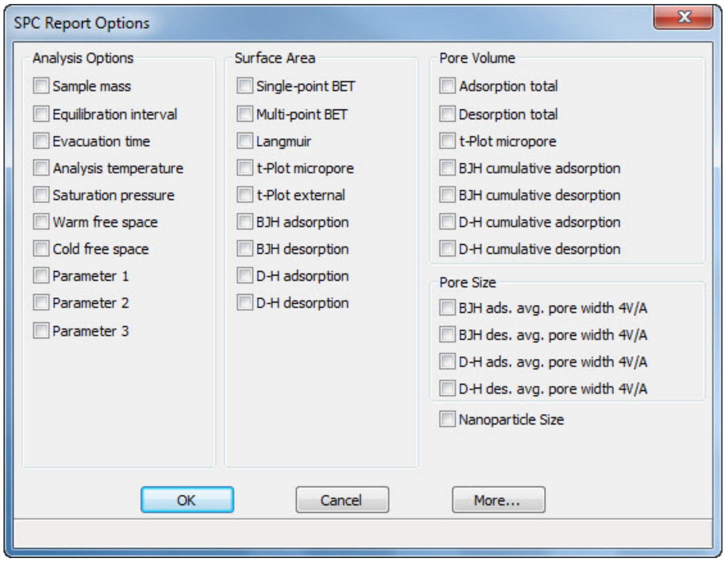
File name: *.rep

SPC Report Options

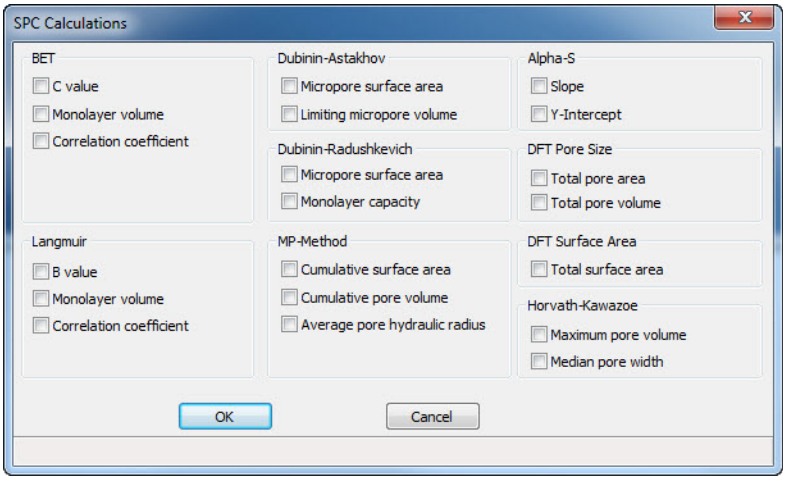
Reports* > *SPC Report Options

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

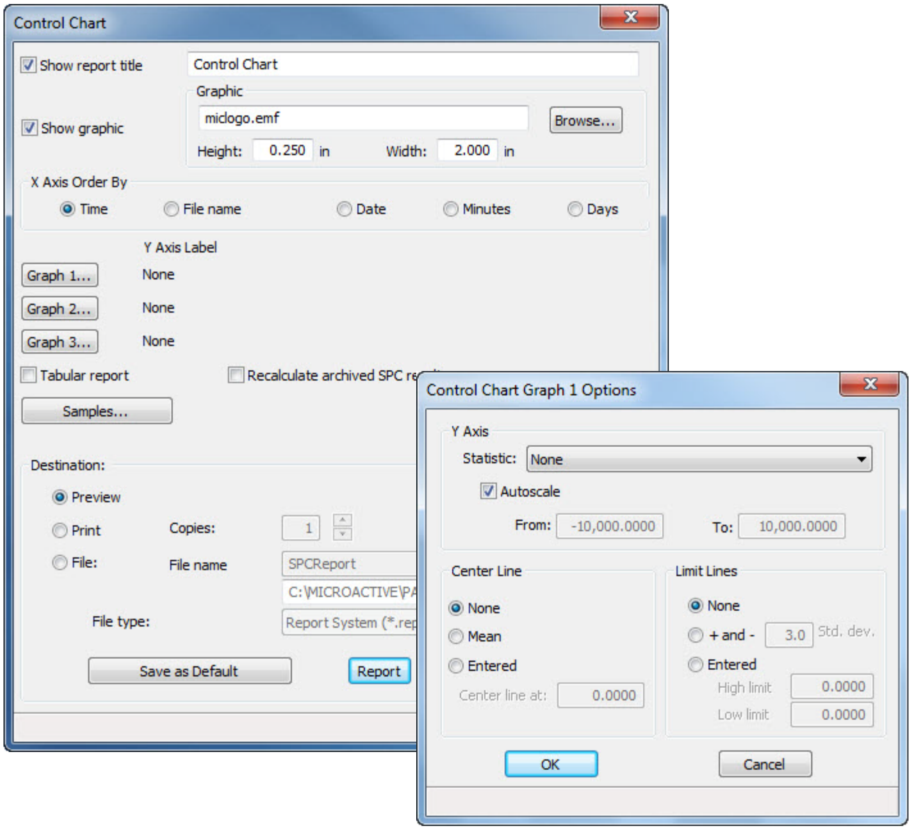
The selected items appear as options on the ***Reports* > *Regression Report*** screen as selections in the dropdown boxes and are used in graph selection in ***Reports* > *Control Chart.***



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



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



Control Chart

☑ Show report title: Control Chart

☑ Show graphic

Graphic: miclogo.emf

Height: 0.250 in

Width: 2.000 in

X Axis Order By: ● Time ○ File name ○ Date ○ Minutes ○ Days

Y Axis Label

Graph 1... None

Graph 2... None

Graph 3... None

☐ Tabular report

☐ Recalculate archived SPC re

Destination: ● Preview ○ Print ○ File:

Copies: 1

File name: SPCReport

C:\MICROACTIVE\PA

File type: Report System (*.rep

Control Chart Graph 1 Options

Y Axis

Statistic: None

☑ Autoscale

From: -10,000.0000

To: 10,000.0000

Center Line: ● None ○ Mean ○ Entered

Center line at: 0.0000

Limit Lines: ● None ○ + and - 3.0 Std. dev. ○ Entered

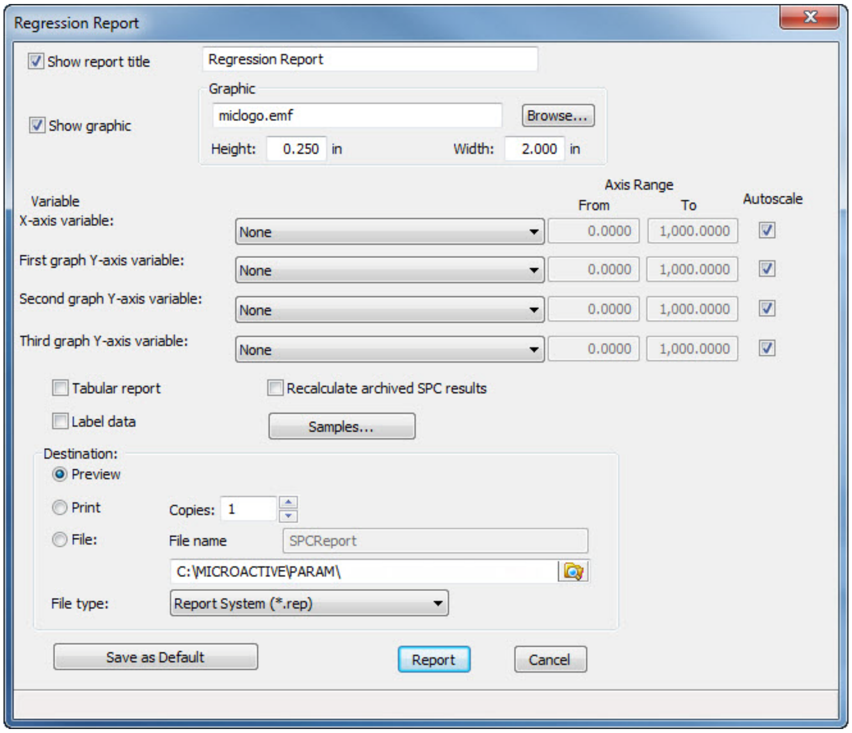
High limit: 0.0000

Low limit: 0.0000

Regression Report

Reports > Regression Report

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



Regression Report

☑ Show report title: Regression Report

Graphic

☑ Show graphic: miclogo.emf

Height: 0.250 in Width: 2.000 in

Variable		Axis Range From	Axis Range To	Autoscale
X-axis variable:	None	0.0000	1,000.0000	☑
First graph Y-axis variable:	None	0.0000	1,000.0000	☑
Second graph Y-axis variable:	None	0.0000	1,000.0000	☑
Third graph Y-axis variable:	None	0.0000	1,000.0000	☑

☐ Tabular report

☐ Recalculate archived SPC results

☐ Label data

Destination:

● Preview

○ Print Copies: 1

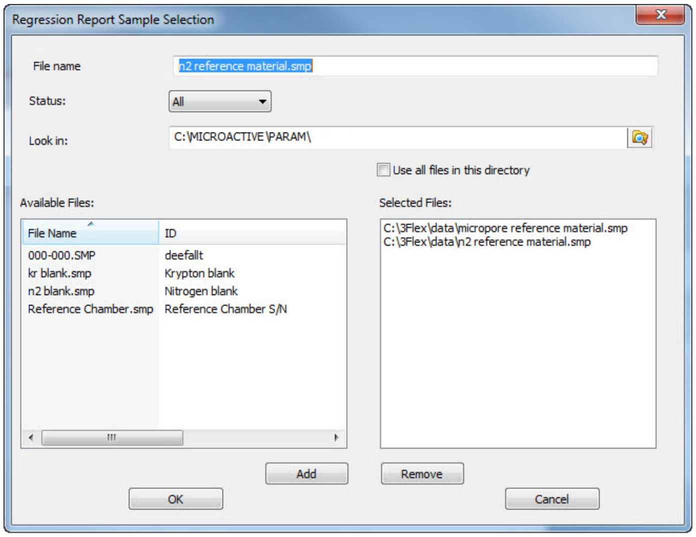
○ File: File name SPCReport

C:\MICROACTIVE\PARAM\

File type: Report System (*.rep)

Field or Button	Description
Show report title *text box*	Select and enter a report title to appear on the report header.
Show graphic *text box*	Use to show a graphic on the report header. Click the **Browse** button 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.
X- and Y-Axis Variable *dropdown lists*	Use to designate the x- and y-axes variables. The variables in the dropdown lists are those selected in the ***Reports > SPC Report Options*** window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.
Axis Range *text boxes*	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if **Autoscale** is selected.

Field or Button	Description *(continued)*
***Autoscale** checkboxes*	When enabled, allows the x- and y-axes to be scaled automatically.
***Tabular report** checkbox*	Use to generate a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
***Label data** checkbox*	Use to label the points on the plot to correspond with the values in the sample files.
***Recalculate archived SPC results** checkbox*	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.

<table>
<tr><th>Field or Button</th><th>Description (continued)</th></tr>
<tr><td>Samples button</td><td>Click to select sample files for report generation. Multiple files can be selected by holding down the Ctrl key on the keyboard while selecting the files.


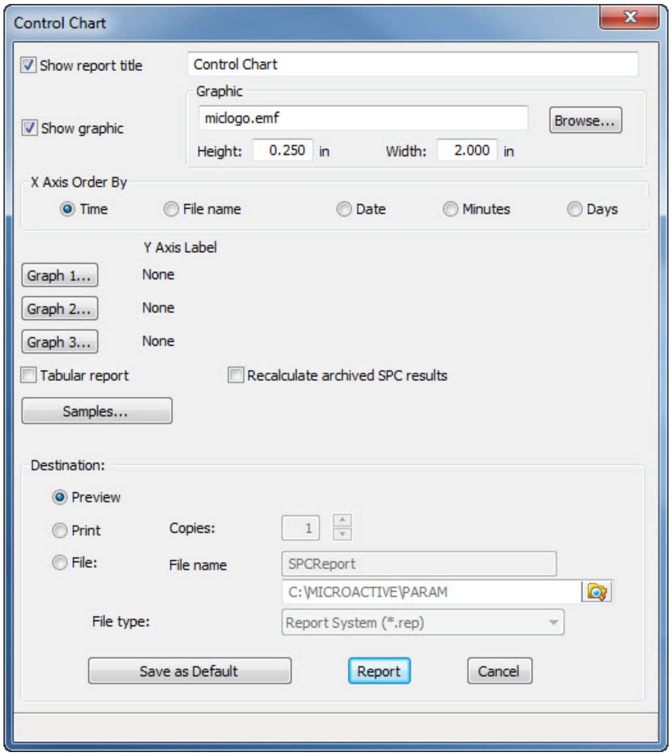
File name: n2 reference material.smp
Status: All
Look in: C:\MICROACTIVE\PARAM\
☐ Use all files in this directory
Available Files:
<table>
<tr><th>File Name</th><th>ID</th></tr>
<tr><td>000-000.SMP</td><td>deefallt</td></tr>
<tr><td>kr blank.smp</td><td>Krypton blank</td></tr>
<tr><td>n2 blank.smp</td><td>Nitrogen blank</td></tr>
<tr><td>Reference Chamber.smp</td><td>Reference Chamber S/N</td></tr>
</table>
Selected Files:
C:\3Flex\data\micropore reference material.smp
C:\3Flex\data\n2 reference material.smp

Use all files in directory- select to include all files in the report.
Available Files - contains files located in the directory specified in the Look In text box.
Selected Files - files added from the Available Files list box.
Add / Remove buttons - select a file in the Available Files list box and click Add to move the file to the Selected Files list box. Or select a file in the Selected Files list box and click Remove to move the file back to the Available Files list box. Or double-click the file name to move the file from one list box to the other.
</td></tr>
<tr><td>Report button</td><td>Click to view the report for the selected items.</td></tr>
<tr><td>Save as Default button</td><td>Click to save selected report options as default report settings.</td></tr>
<tr><td>Browse button
Cancel button
Destination group box
From / To text boxes
OK button</td><td>Refer to Common Fields and Buttons - Reports Menu Options, page 5-1.</td></tr>
</table>

Control Chart

Reports* > *Control Chart

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



☑ Show report title: Control Chart

Graphic

☑ Show graphic: miclogo.emf

Height: 0.250 in Width: 2.000 in

X Axis Order By: ● Time ○ File name ○ Date ○ Minutes ○ Days

Y Axis Label

Graph 1... None

Graph 2... None

Graph 3... None

☐ Tabular report ☐ Recalculate archived SPC results

Destination:

● Preview

○ Print Copies: 1

○ File: File name: SPCReport

C:\MICROACTIVE\PARAM

File type: Report System (*.rep)

Field or Button	Description
***Show report title** text box*	Select and enter a report title to appear on the report header.
***Show graphic** text box*	Use to show a graphic on the report header. Click the **Browse** button 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.

<table>
<tr><th>Field or Button</th><th>Description (continued)</th></tr>
<tr><td>

X Axis Order by *group box*

</td><td>

Select the order in which x-axis statistics are placed.

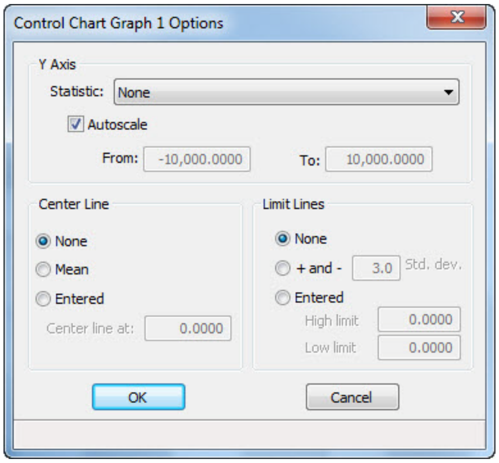
- **Time -** sorts by the time the files were analyzed.
- **File name -** sorts in alphanumeric order.
- **Date** - sorts by the date the files were analyzed.
- **Minutes -** sorts by the minutes elapsed from the first file placed on the list, which is the earliest-analyzed file.
- **Days** - sorts by the number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.

</td></tr>
<tr><td>

Graph [n] *buttons*

</td><td>

Click to define the y-axis of each graph.



Statistic: None
☑ Autoscale
From: -10,000.0000 To: 10,000.0000

Center Line: ● None ○ Mean ○ Entered
Center line at: 0.0000

Limit Lines: ● None ○ + and - 3.0 Std. dev. ○ Entered
High limit 0.0000
Low limit 0.0000

- **Y-Axis** group box -

  Statistic dropdown list - displays the SPC variables selected at ***Reports > SPC Report Options*** window. The selected variable will be plotted against time. This selection also becomes the y-axis label.

  Autoscale checkbox - allows the y-axis to be scaled automatically. To specify a range, deselect this option and enter a range in the **From** and **To** fields.
- **Center Line** group box - displays placement options for the center line in the graph. Choose **Entered** to specify placement of the line.
- **Limit Lines** group box - displays limiting lines options. Lines can be placed at some multiple of the standard deviation or at specified positions (**Entered**). When **Entered** is selected, enter the **High limit** and **Low limit** fields with appropriate values.

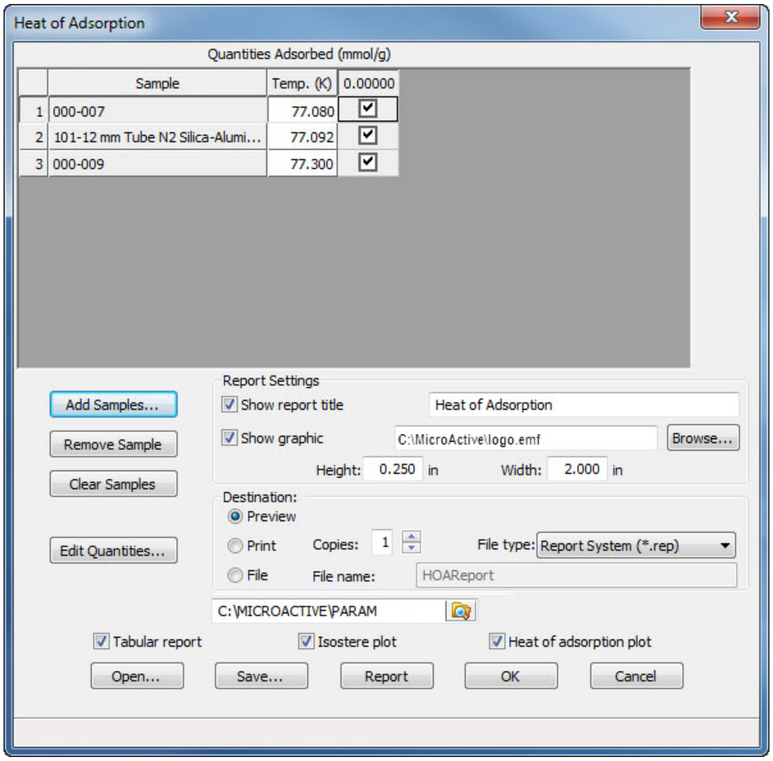
</td></tr>
</table>

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

Heat of Adsorption Report

Reports > Heat of Adsorption

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



Quantities Adsorbed (mmol/g)

	Sample	Temp. (K)	0.00000
1	000-007	77.080	☑
2	101-12 mm Tube N2 Silica-Alumi...	77.092	☑
3	000-009	77.300	☑

Report Settings

☑ Show report title: Heat of Adsorption

☑ Show graphic: C:\MicroActive\logo.emf

Height: 0.250 in Width: 2.000 in

Destination:

● Preview

○ Print Copies: 1 File type: Report System (*.rep)

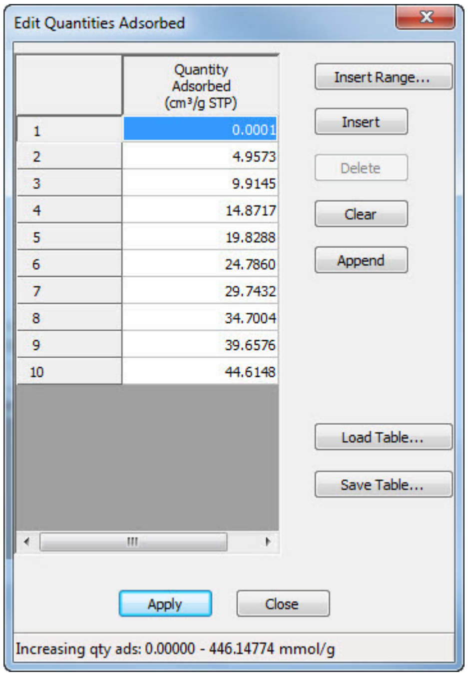
○ File File name: HOAReport

C:\MICROACTIVE\PARAM

☑ Tabular report ☑ Isostere plot ☑ Heat of adsorption plot

Field or Button	Description
Table	Contains files added by using the **Add Samples** button and provides the quantity adsorbed.
Add Samples *button*	Click to add a sample file to the table. 1. Click the **Add Samples** button. 2. Double-click the file in the **Name** column or select the file name and click **Open**.
Remove Sample *button*	Click to remove the selected sample from the list.

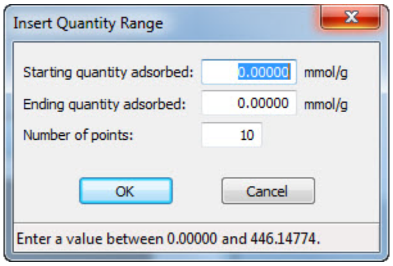
<table>
<tr><th>Field or Button</th><th>Description (continued)</th></tr>
<tr><td>Clear Samples button</td><td>Click to remove all entries from the table.</td></tr>
<tr><td>Edit Quantities button</td><td>
Use to specify the range of surface coverage to include in the report.



<table>
<tr><th></th><th>Quantity Adsorbed (cm³/g STP)</th></tr>
<tr><td>1</td><td>0.0001</td></tr>
<tr><td>2</td><td>4.9573</td></tr>
<tr><td>3</td><td>9.9145</td></tr>
<tr><td>4</td><td>14.8717</td></tr>
<tr><td>5</td><td>19.8288</td></tr>
<tr><td>6</td><td>24.7860</td></tr>
<tr><td>7</td><td>29.7432</td></tr>
<tr><td>8</td><td>34.7004</td></tr>
<tr><td>9</td><td>39.6576</td></tr>
<tr><td>10</td><td>44.6148</td></tr>
</table>

Increasing qty ads: 0.00000 - 446.14774 mmol/g

- **Insert Range** button - click to specify the starting and ending quantities adsorbed and number of points to insert.



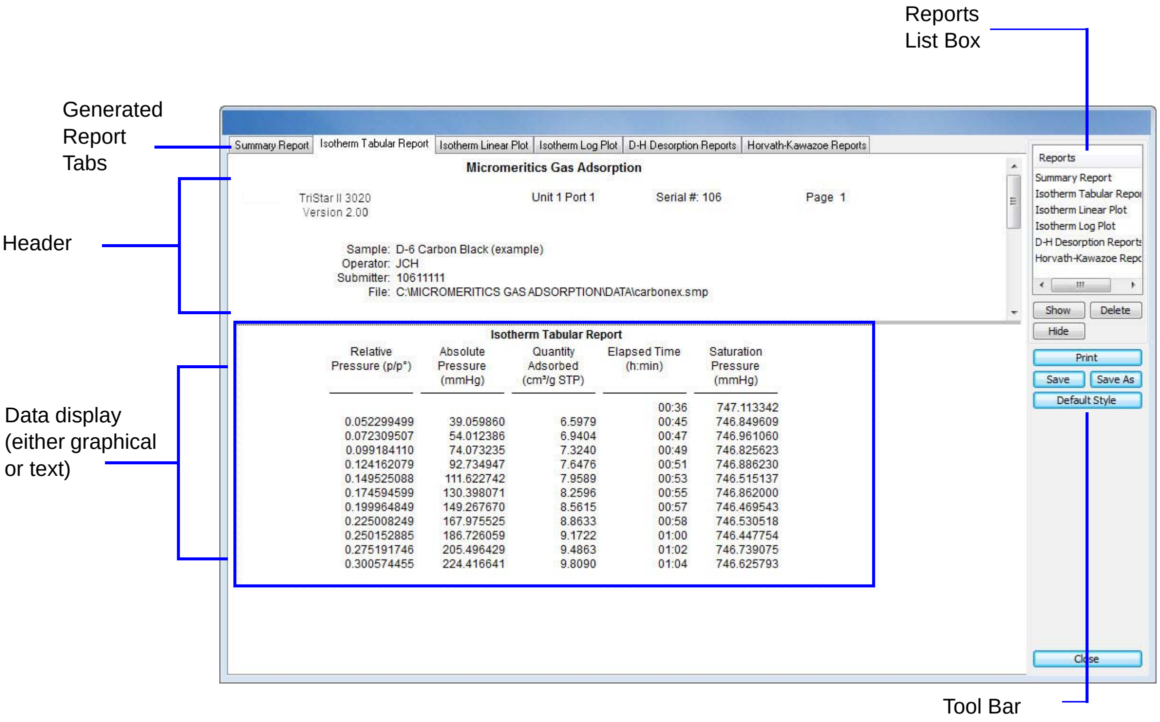
Starting quantity adsorbed: 0.00000 mmol/g
Ending quantity adsorbed: 0.00000 mmol/g
Number of points: 10

Enter a value between 0.00000 and 446.14774.

- **Insert** button - insert a row above the selected row.
- **Delete** button - deletes the selected row.
- **Clear** button - clears the entire table of all entries except one.
- **Append** button - inserts one row at the end of the table.
- **Load Table** button - click to import values from another file.
- **Save Table** button - save the current table as a. QNT file.
- **Apply** button - click to apply all table changes.
- **Close** button - click to close the table without saving changes.
</td></tr>
<tr><td>Tabular report checkbox</td><td>Refer to Regression Report, page 5-7.</td></tr>
</table>

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

Report Features and Shortcuts



Micromeritics Gas Adsorption

TriStar II 3020
Version 2.00
Unit 1 Port 1
Serial #: 106
Page 1

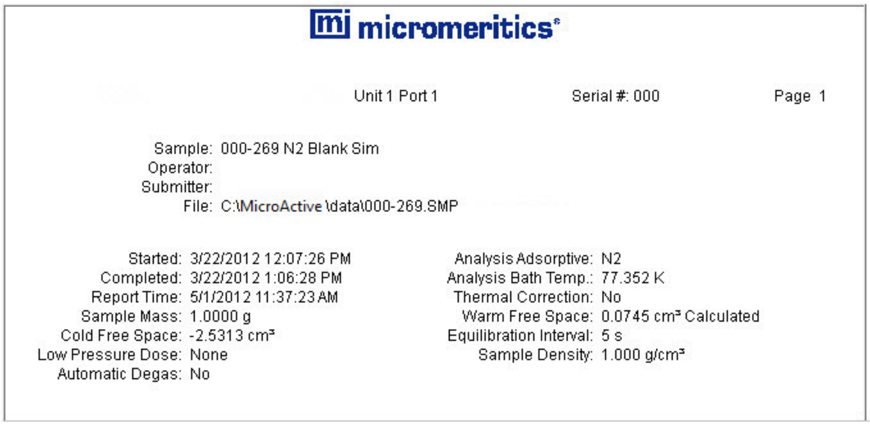
Sample: D-6 Carbon Black (example)
Operator: JCH
Submitter: 10611111
File: C:\MICROMERITICS GAS ADSORPTION\DATA\carbonex.smp

Isotherm Tabular Report

Relative Pressure (p/p°)	Absolute Pressure (mmHg)	Quantity Adsorbed (cm³/g STP)	Elapsed Time (h:min)	Saturation Pressure (mmHg)
			00:36	747.113342
0.052299499	39.059860	6.5979	00:45	746.849609
0.072309507	54.012386	6.9404	00:47	746.961060
0.099184110	74.073235	7.3240	00:49	746.825623
0.124162079	92.734947	7.6476	00:51	746.886230
0.149525088	111.622742	7.9589	00:53	746.515137
0.174594599	130.398071	8.2596	00:55	746.862000
0.199964849	149.267670	8.5615	00:57	746.469543
0.225008249	167.975525	8.8633	00:58	746.530518
0.250152885	186.726059	9.1722	01:00	746.447754
0.275191746	205.496429	9.4863	01:02	746.739075
0.300574455	224.416641	9.8090	01:04	746.625793

Report Header

All reports contain a header displaying file statistics.



Unit 1 Port 1   Serial #: 000   Page 1

Sample: 000-269 N2 Blank Sim
Operator:
Submitter:
File: C:\MicroActive \data\000-269.SMP

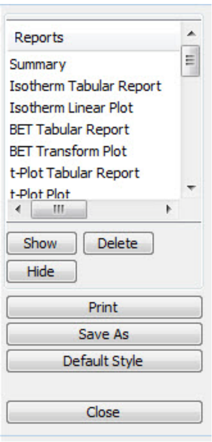
Started: 3/22/2012 12:07:26 PM
Completed: 3/22/2012 1:06:28 PM
Report Time: 5/1/2012 11:37:23 AM
Sample Mass: 1.0000 g
Cold Free Space: -2.5313 cm³
Low Pressure Dose: None
Automatic Degas: No

Analysis Adsorptive: N2
Analysis Bath Temp.: 77.352 K
Thermal Correction: No
Warm Free Space: 0.0745 cm³ Calculated
Equilibration Interval: 5 s
Sample Density: 1.000 g/cm³

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

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

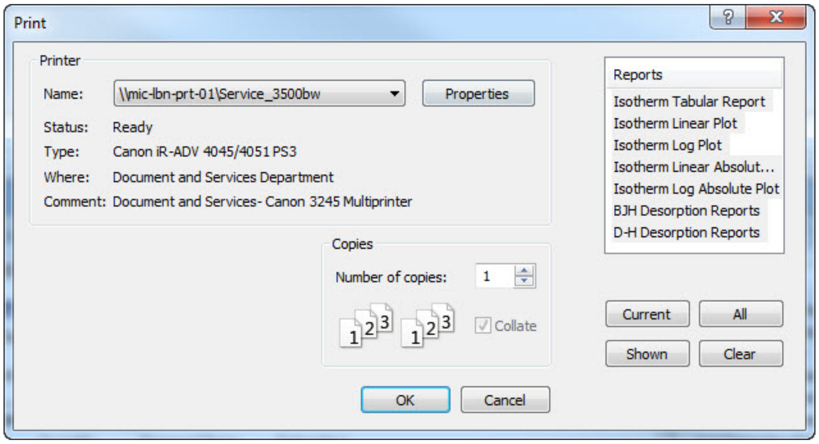
Report Tool Bar

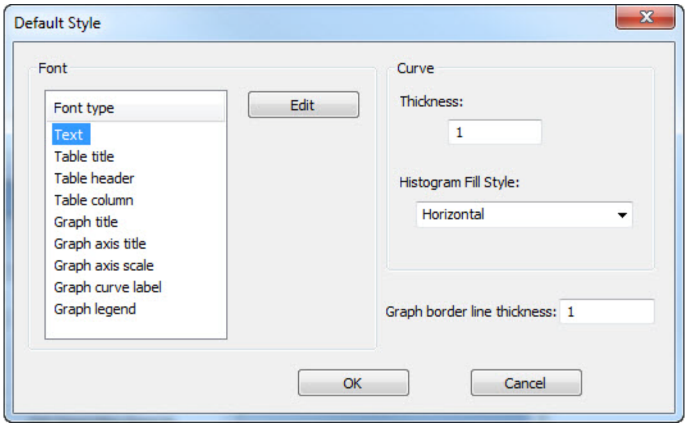
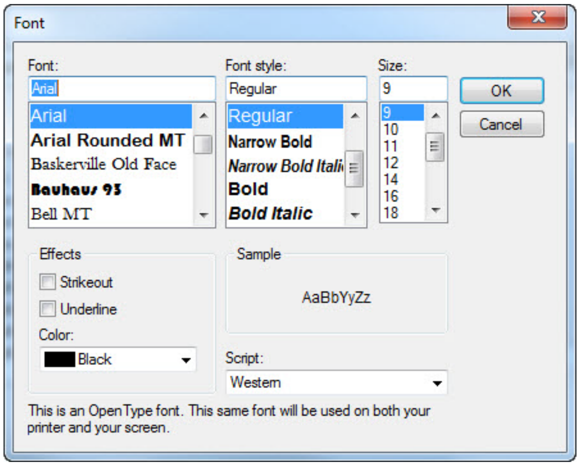


The **Report** window has a tool bar and selectable tabs across the top of the report header. To view a specific report, select its tab or select the report in the Reports list box and click ***Show***.

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

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

Field or Button	Description *(continued)*
***Print** button*	Displays the **Print** window for report output.  Name: \\mic-lbn-prt-01\Service_3500bw Status: Ready Type: Canon iR-ADV 4045/4051 PS3 Where: Document and Services Department Comment: Document and Services- Canon 3245 Multiprinter Number of copies: 1 ☑ Collate • **Name** dropdown list and **Properties** button - select the printer and click the **Properties** button to change printer setup, etc. • **Copies** group box - select the number of copies and collate option. • **Current** button - selects the active report (or selected tab). • **All** button - selects all reports in the Reports list box. • **Shown** button - selects only the reports not hidden. • **Clear** button - clears all selections. • **OK** button - prints the selected report to the printer indicated. • **Cancel** button - closes the **Print** window.
***Save** button*	Saves all reports of the active file using the sample file name with a .REP file extension.
***Save As** button*	Saves all selected reports to the indicated file format: • **.REP** (Report system) - saves the report in a format that can be opened with any MicroActive program. • **.TXT** (ASCII text) - saves the report as a common machine language file. • **.XLS** (Spreadsheet file) - saves the report in a spreadsheet format.

Field or Button	Description *(continued)*
***Default Style** button*	Click to specify default report parameters for fonts and curve properties.  Thickness: 1 Histogram Fill Style: Horizontal Graph border line thickness: 1 - **Font** group box - **Font Type** list box - allows font type and attributes to be edited for the selected item. Select an item in the list, click **Edit**, and select from various font options. Click **OK** when done.  Font: Arial Font style: Regular Size: 9 ☐ Strikeout ☐ Underline Color: Black Script: Western This is an OpenType font. This same font will be used on both your printer and your screen.

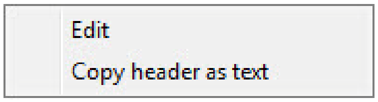
Field or Button	Description *(continued)*
***Default Style** button (continued)*	- **Curve** group box - **Thickness** text box - enter a thickness number for the curve. **Histogram Fill** dropdown list - select a histogram fill option from the list. - **Graph border line thickness** text box - enter a thickness number for the graph border. - **Load** button - click to load the last saved default settings. - **Save** button - click to save the changes as the new default settings. - **Close** button - click to close the window and save the changes for the current report.

Report Shortcut Menus

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

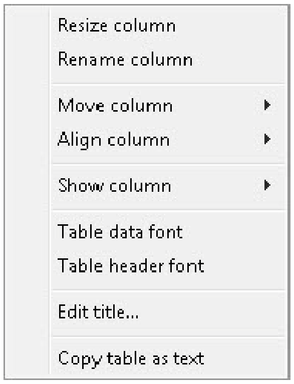
Report Header Shortcuts

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



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

Tabular Reports Shortcuts

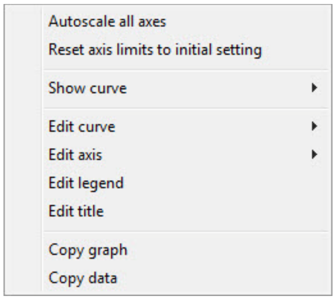


Display tabular report shortcuts by right-clicking in the body of the tabular report. Column shortcuts require right-clicking on the column to be modified.

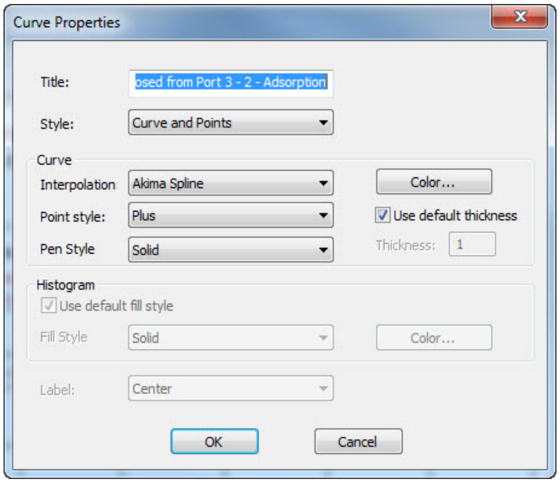
Option	Description
Resize column	Right-click on the column to be resized. Select **Resize Column** on the shortcut menu and enter the new column width in inches.
Rename column	Right-click on the column to be renamed. Select **Rename Column** on the shortcut menu and enter the new column name.
Move column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

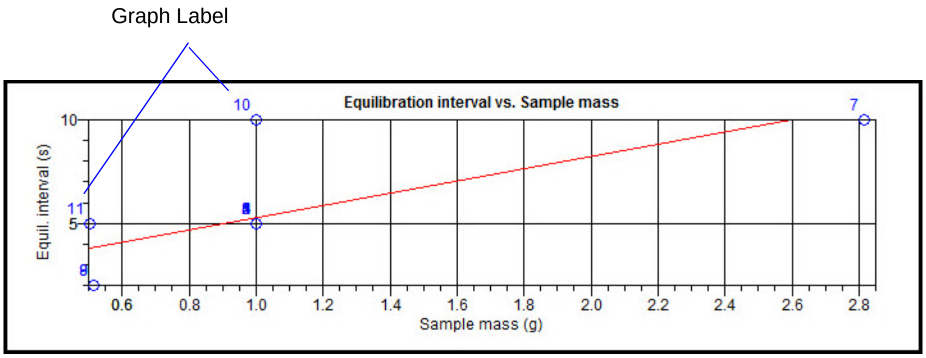
Option	Description *(continued)*
Copy table as text	Use to copy the report contents to the clipboard as tab delimited text. It can then be pasted into another document.

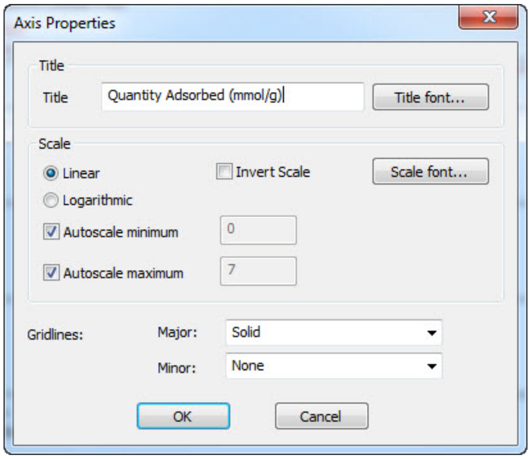
Graph Shortcuts

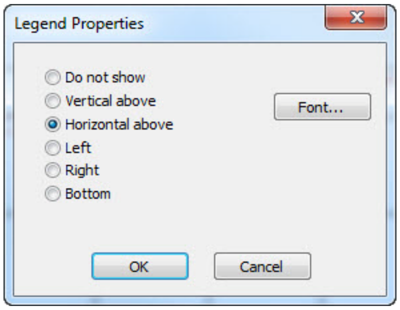
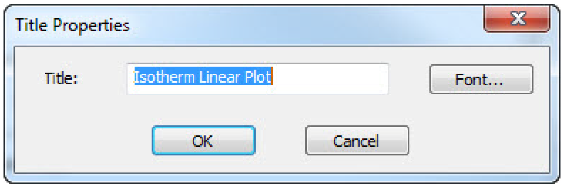


Display graph report shortcuts by right-clicking in the body of the graph report.

Option	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
Show curve	Displays a list of all curves. Click a column to add a checkmark and show the curve or remove the checkmark to hide the curve.
Edit curve	Use to edit selected curve properties.  Curve Properties Title: osed from Port 3 - 2 - Adsorption Style: Curve and Points Curve Interpolation: Akima Spline Point style: Plus Pen Style: Solid ☑ Use default thickness Thickness: 1 Histogram ☑ Use default fill style Fill Style: Solid Label: Center • **Title** text box - use to change the title of the selected curve.

Option	Description *(continued)*
Edit curve *(continued)*	• **Style** dropdown list - use to select another style for the collected data curve. • **Curve** group box - options are disabled if **Histogram** is selected in the **Style** dropdown list. Use to change the interpolation, point style and pen style for the selected curve. **Color** button - click to change the curve color. **Use default thickness** checkbox - select to use the default curve thickness. Deselect the checkbox and enter a new thickness number in the **Thickness** text box. • **Histogram** group box - enabled only if **Histogram** is selected in the **Style** dropdown list. Use to specify the type of fill, fill color and label position for the selected curve. **Label** dropdown list - select where the graph point labels will display (left, right, center, etc.) on the SPC report.
	

Option	Description *(continued)*
Edit axis	Use to edit the selected axis properties.  Title: Quantity Adsorbed (mmol/g) ● Linear ☐ Invert Scale ○ Logarithmic ☑ Autoscale minimum 0 ☑ Autoscale maximum 7 Major: Solid Minor: None • **Title** group box - use to edit the selected axis label. **Title** text box - use to modify the label of the selected axis. **Title font** button - use to modify the font for the selected axis label. Deselect the **Use default font** to enable font options. Select the font attributes and click **OK**. • **Scale** group box - use to change the graph display. **Linear / Logarithmic** - select the option to scale the graph as linear or logarithmic. **Autoscale minimum / maximum** - select the **Autoscale** checkbox to enable the option. To manually specify minimum / maximum, deselect the **Autoscale** checkbox and enter the new amount in the text box. **Invert scale** checkbox - use to invert the scale. **Scale font** button - use to modify the font for the scale label. Deselect the **Use default font** to enable font options. Select the font attributes and click **OK**. **Gridlines Major / Minor** dropdown lists - use to change how to display major / minor gridlines.

Option	Description *(continued)*
Edit legend	Use to change the legend location and font. Click **Font** to modify legend fonts. Deselect the **Use default font** to enable font options.  Legend Properties ○ Do not show ○ Vertical above ● Horizontal above ○ Left ○ Right ○ Bottom
Edit title	Use to change the graph title and font. Deselect the **Use default font** to enable font options.  Title Properties Title: Isotherm Linear Plot
Copy Graph	Copies the graph to the clipboard. It can then be pasted into other applications.
Copy Data	Copies the report data to the clipboard. It can then be pasted into other applications as tab-delimited columns of text.

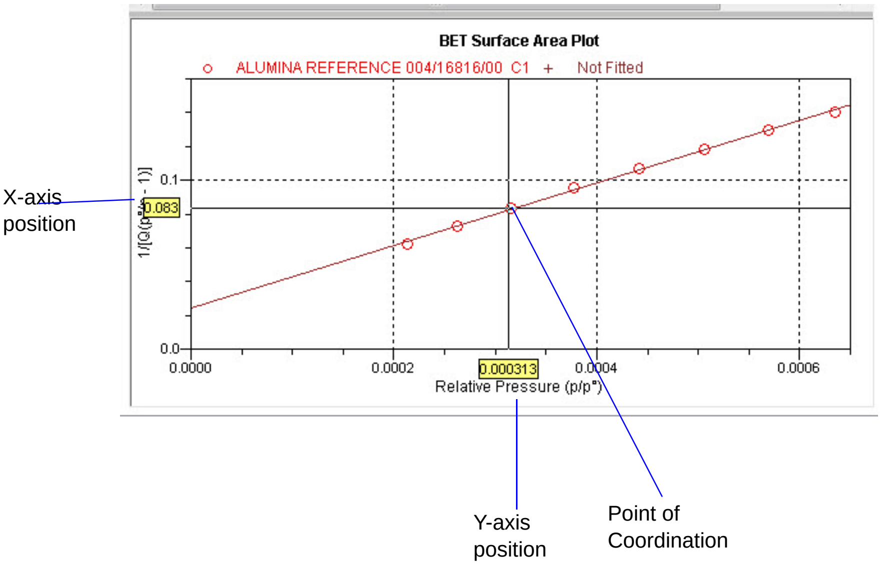
Other On-Screen Features

Zoom Feature

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

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

Axis Cross-Hair



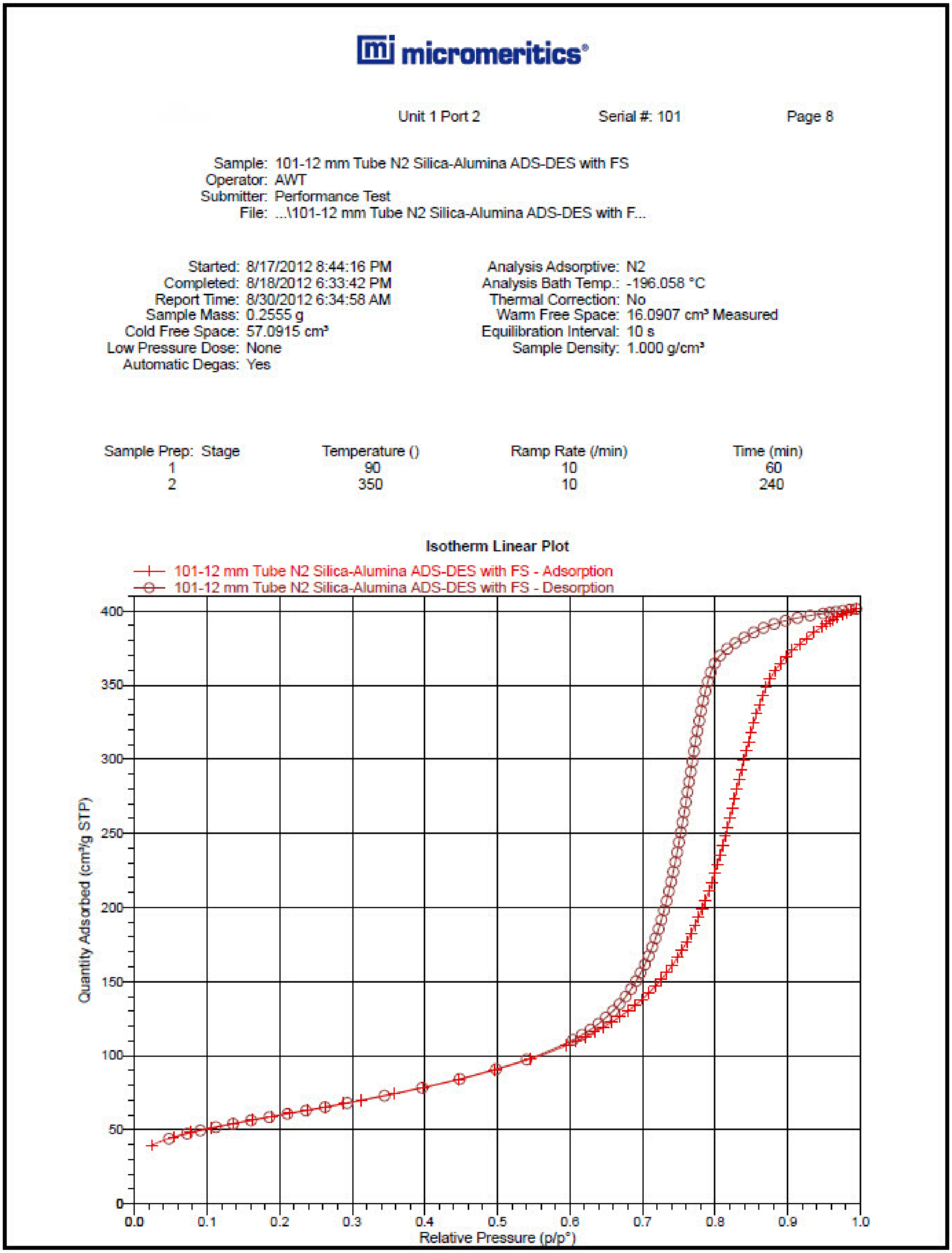
The cross-hair feature displays axis coordinates. To use this feature:

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

Report Examples

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

Isotherm Linear Plot



micromeritics®

Unit 1 Port 2 Serial #: 101 Page 8

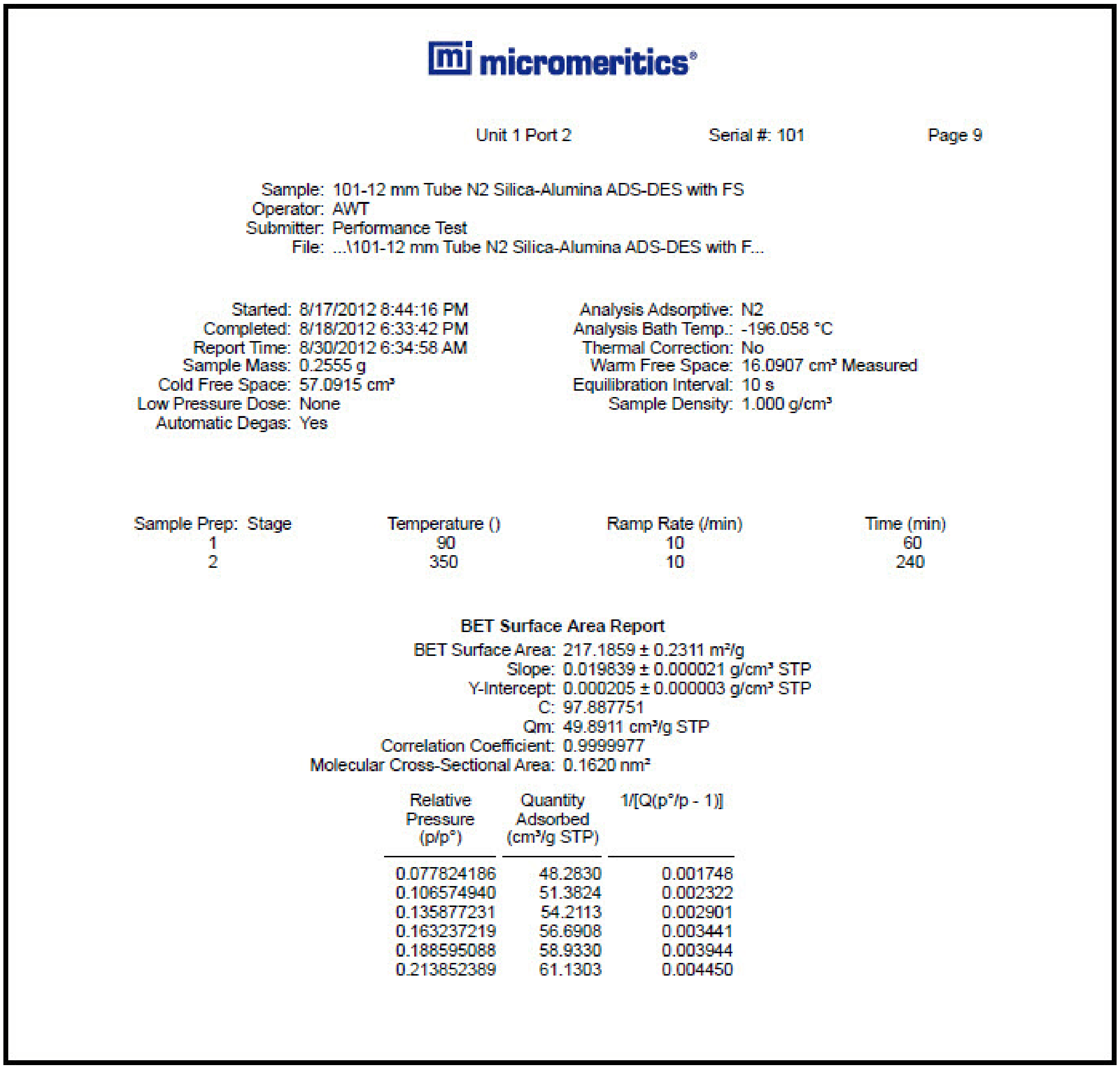
Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

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

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

Isotherm Linear Plot

BET Surface Area Report



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Unit 1 Port 2 Serial #: 101 Page 9

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

Started: 8/17/2012 8:44:16 PM
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 cm^3
Low Pressure Dose: None
Automatic Degas: Yes

Analysis Adsorptive: N2
Analysis Bath Temp.: -196.058 °C
Thermal Correction: No
Warm Free Space: 16.0907 cm^3 Measured
Equilibration Interval: 10 s
Sample Density: 1.000 g/cm^3

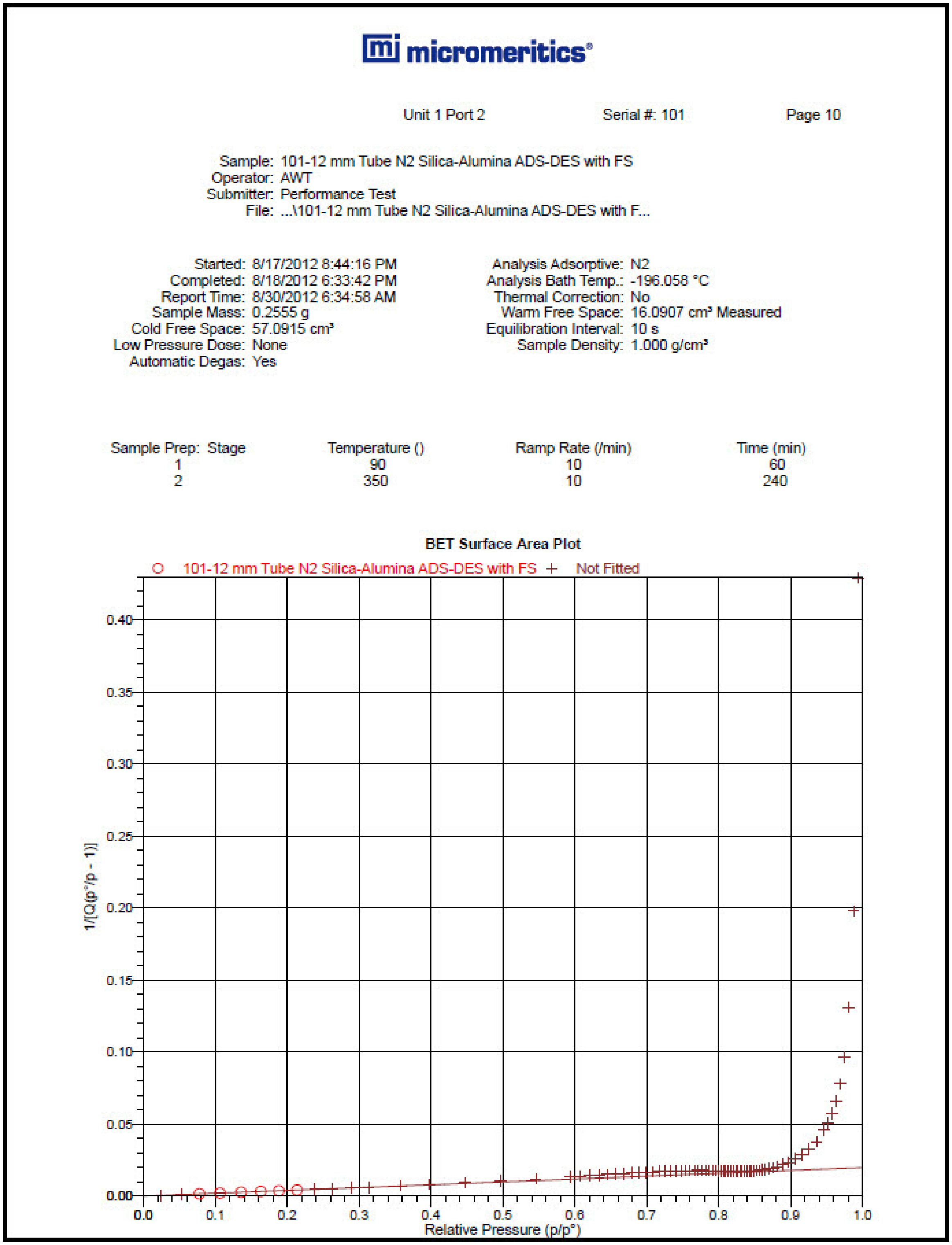
Sample Prep: Stage	Temperature ()	Ramp Rate (/min)	Time (min)
1	90	10	60
2	350	10	240

BET Surface Area Report

BET Surface Area: 217.1859 ± 0.2311 m^2/g
Slope: 0.019839 ± 0.000021 g/cm^3 STP
Y-Intercept: 0.000205 ± 0.000003 g/cm^3 STP
C: 97.887751
Qm: 49.8911 cm^3/g STP
Correlation Coefficient: 0.9999977
Molecular Cross-Sectional Area: 0.1620 nm^2

Relative Pressure (p/p°)	Quantity Adsorbed (cm^3/g STP)	$1/[Q(p^\circ/p - 1)]$
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



Unit 1 Port 2 Serial #: 101 Page 10

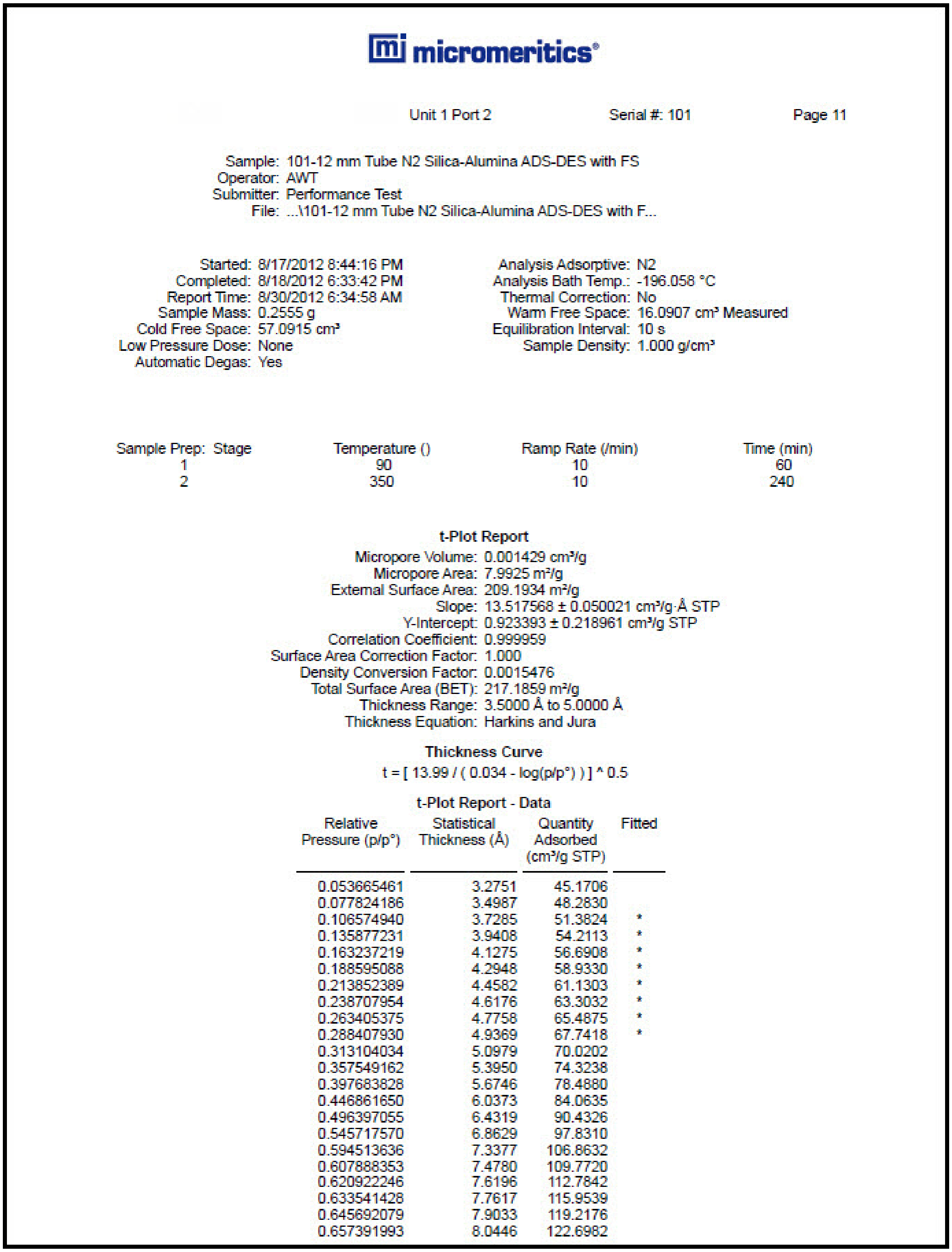
Sample: 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS
Operator: AWT
Submitter: Performance Test
File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

Started: 8/17/2012 8:44:16 PM
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



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Unit 1 Port 2 Serial #: 101 Page 11

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

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

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

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

t-Plot Report

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

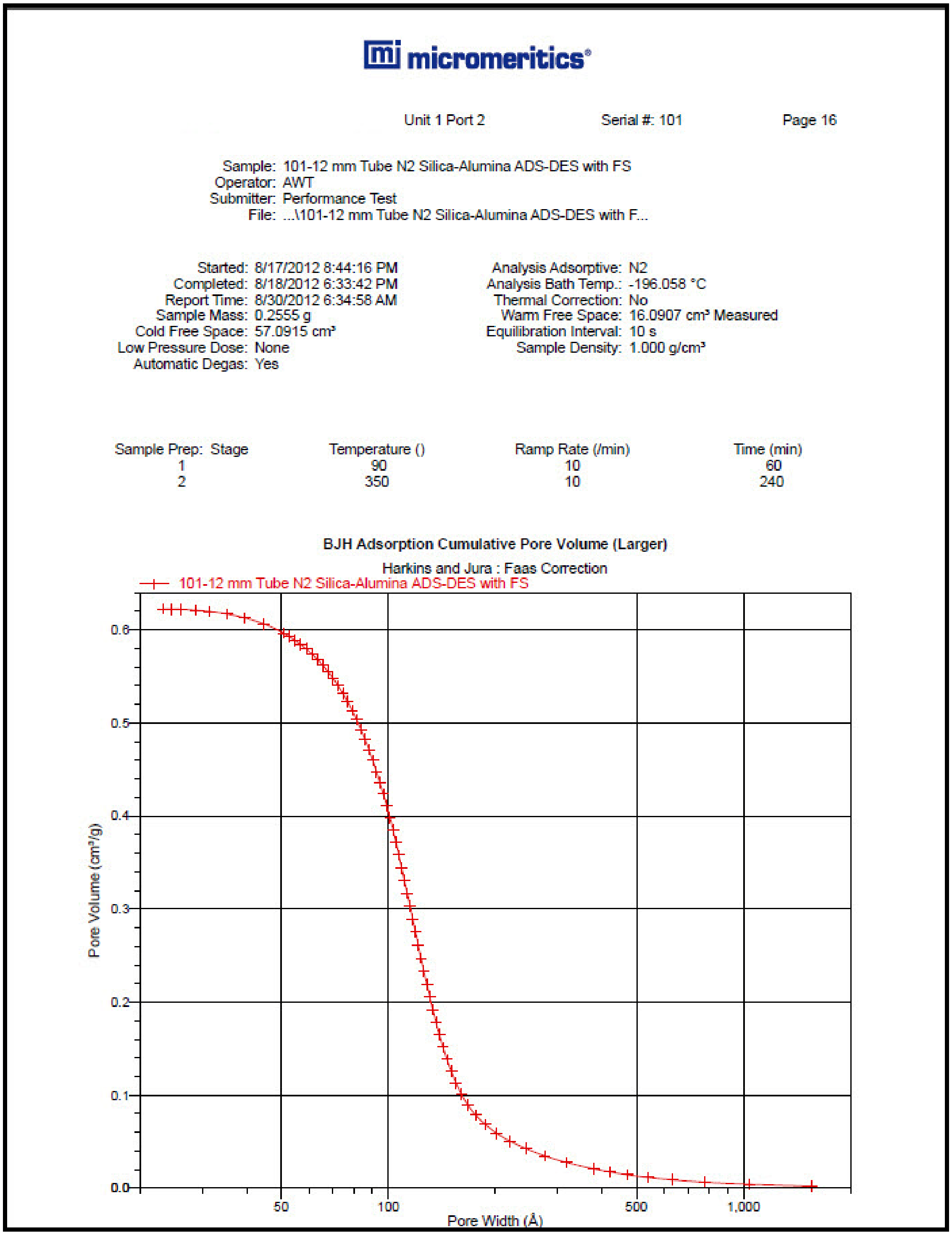
Thickness Curve

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

t-Plot Report - Data

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

BJH Adsorption: Cumulative Pore Volume



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Unit 1 Port 2 Serial #: 101 Page 16

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

Started: 8/17/2012 8:44:16 PM
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

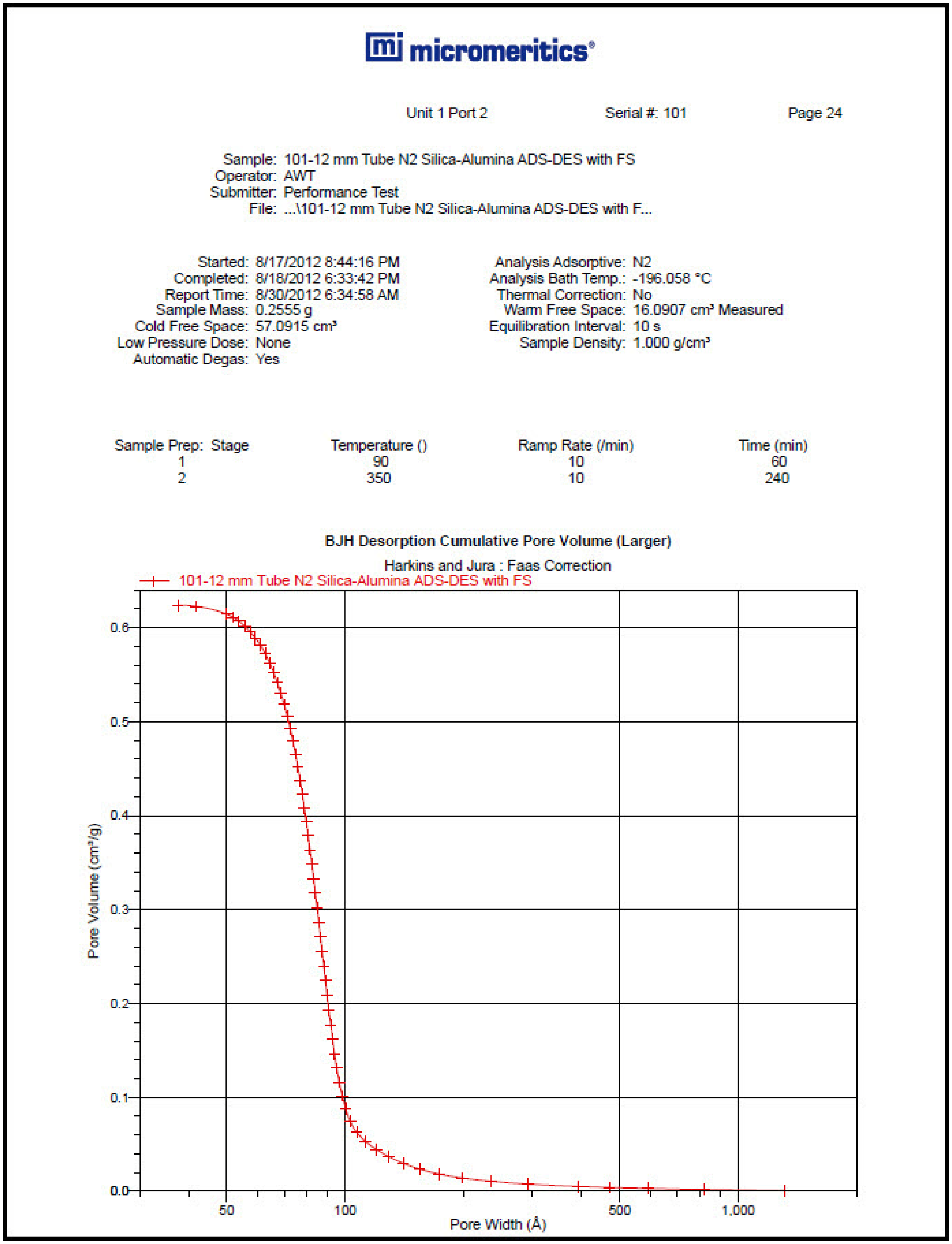
Analysis Adsorptive: N2
Analysis Bath Temp.: -196.058 °C
Thermal Correction: No
Warm Free Space: 16.0907 cm³ Measured
Equilibration Interval: 10 s
Sample Density: 1.000 g/cm³

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

BJH Adsorption Cumulative Pore Volume (Larger)

Harkins and Jura : Faas Correction

BJH Desorption: Cumulative Pore Volume



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Unit 1 Port 2 Serial #: 101 Page 24

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

Started: 8/17/2012 8:44:16 PM
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

BJH Desorption Cumulative Pore Volume (Larger)

Harkins and Jura : Faas Correction

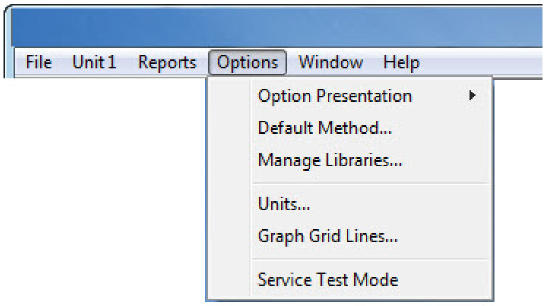
6. OPTIONS MENU

Introduction

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

This chapter contains information on:

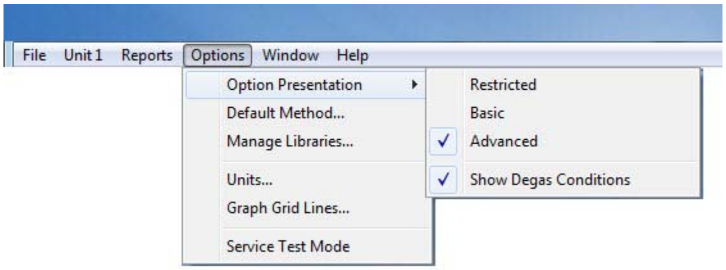
- changing the default presentation format - Restricted, Basic, or Advanced.
- specifying default parameters for sample information files and report option files.
- specifying how units appear on application windows and reports.



Option Presentation

Options** > **Option Presentation

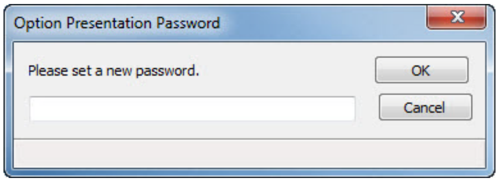
Use to change the default editing format of sample files - Restricted, Basic, or Advanced. Each format type displays sample information and options differently. For descriptions of the presentation types, refer to **Defining Sample Information Files**, page **2-9**. When using **Restricted** format, a password is required to change to **Advanced** format.



- **Show Degas Conditions** - when enabled, displays a Degas Conditions tab when in Advanced presentation format.

Restricted Format

Restricted format presents the sample information file in the same manner as the Basic format except that the operator cannot switch to Advanced format. This format is password protected and is typically used in laboratories where sample parameters must remain constant, for example, in pharmaceutical laboratories.



Enter a password, then click **OK**. In order to switch to either Basic or Advanced when using Restricted format, enter the same password.

Default Method

Options* > *Default Method

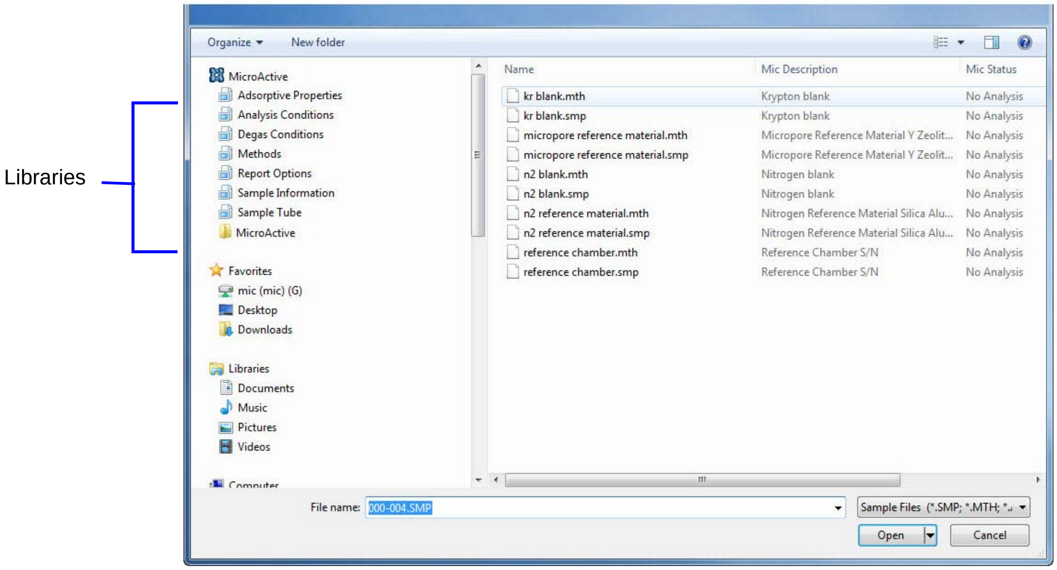
Use to specify default parameters for sample information files. Refer to **Editing the Default Method**, page **2-7** for additional information on using this feature.

Manage Libraries

Options > Manage Libraries

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

Libraries are virtual folders used to organize commonly accessed folders and files. Folders can be added to or removed from a library without affecting the location of the files and folders on disk. Files and folders in a library will appear within the File Selector when that library is selected from the left pane of the File Selector.

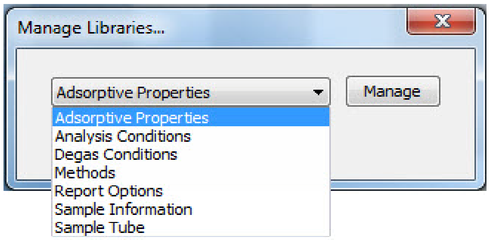


Name	Mic Description	Mic Status
kr blank.mth	Krypton blank	No Analysis
kr blank.smp	Krypton blank	No Analysis
micropore reference material.mth	Micropore Reference Material Y Zeolit...	No Analysis
micropore reference material.smp	Micropore Reference Material Y Zeolit...	No Analysis
n2 blank.mth	Nitrogen blank	No Analysis
n2 blank.smp	Nitrogen blank	No Analysis
n2 reference material.mth	Nitrogen Reference Material Silica Alu...	No Analysis
n2 reference material.smp	Nitrogen Reference Material Silica Alu...	No Analysis
reference chamber.mth	Reference Chamber S/N	No Analysis
reference chamber.smp	Reference Chamber S/N	No Analysis

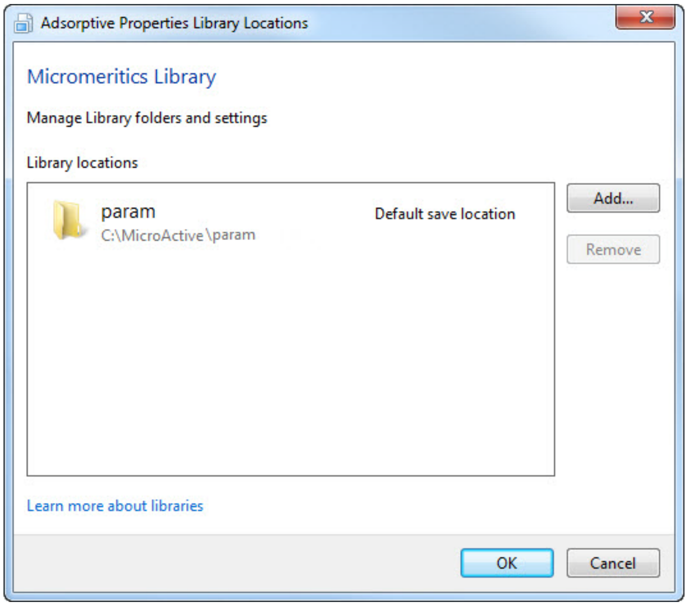
File name: 000-004.SMP

Sample Files (*.SMP; *.MTH; *...

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



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



Adsorptive Properties Library Locations

Micromeritics Library

Manage Library folders and settings

Library locations

param
C:\MicroActive\param
Default save location

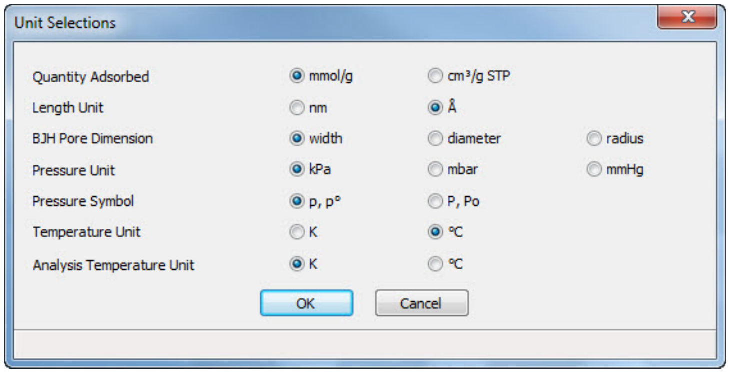
3. To remove a folder, select the folder to be removed from the **Library locations** box and click the **Remove** button.

4. Click **OK** when done.

Units

Options > Units

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



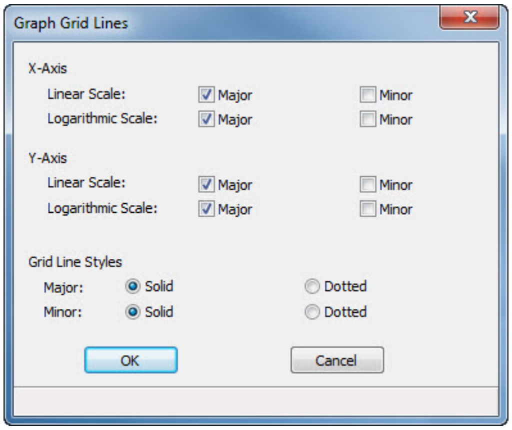
Unit Selections

Quantity Adsorbed	● mmol/g	○ cm³/g STP	
Length Unit	○ nm	● Å	
BJH Pore Dimension	● width	○ diameter	○ radius
Pressure Unit	● kPa	○ mbar	○ mmHg
Pressure Symbol	● p, p°	○ P, Po	
Temperature Unit	○ K	● ºC	
Analysis Temperature Unit	● K	○ ºC	

Graph Grid Lines

Options > Graph Grid Lines

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



Graph Grid Lines

X-Axis
Linear Scale: ☑ Major ☐ Minor
Logarithmic Scale: ☑ Major ☐ Minor

Y-Axis
Linear Scale: ☑ Major ☐ Minor
Logarithmic Scale: ☑ Major ☐ Minor

Grid Line Styles
Major: ● Solid ○ Dotted
Minor: ● Solid ○ Dotted

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

Service Test Mode

Options > ***Service Test Mode***

Service Test Mode is a password protected option used to perform certain service tests with the assistance of a trained Micromeritics service representative. This password is supplied by your service representative. After Service Test Mode has been enabled, the tests are accessible from the Unit menu (***Unit*** > ***Service Test***).

7. TROUBLESHOOTING AND MAINTENANCE

Introduction

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

Troubleshooting

Most operational problems are caused by:

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

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

What Happened	Why	What To Do
Vacuum pump is noisy.	Sample tube connector is loose.	Tighten fitting. Replace O-ring.
	Sample tube O-ring is worn or cracked.	Replace O-ring. Refer to **Replacing the Sample Tube O-ring**, page **7-6**.
	Sample tube is cracked.	Replace with new sample tube.
	No sample tube loaded on a selected port.	Install plug or empty sample tube.
	Gas inlet valve open while vacuum valve open.	With manual control enabled, use the instrument schematic to close gas inlet valve.
Analysis Dewar cannot be raised (or lowered).	Elevator that moves Dewar is stuck.	Check for possible obstruction to elevator movement.
Analysis valves cannot be operated.	Cable from computer to the instrument is loose.	Make sure the cable is connected properly

What Happened	Why	What To Do
Elevator is noisy	The elevator screw may need greasing.	Contact your Micromeritics Service Representative.
Sample is not within specifications.	There may be a leak into or out of the manifold.	Refer to **Inspecting and Changing Vacuum Pump Oil**, page **7-7**.
	Gas may be contaminated.	Perform a blank analysis. If results are good, perform a reference material analysis. Replace tank. Check for line leak which could cause contamination. Flush the lines occasionally to help prevent contamination.
	Incorrect type of gas line.	Ensure the gas line is all metal. It is best to use the one shipped with the instrument. Do not use polymer gas lines or flexible gas lines that may be internally coated with a polymer.

Preventive Maintenance

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

Maintenance Required	Frequency
Check and clean Dewar	Weekly
Replace port filters and O-rings	Every 3 to 6 months
Replace sample tube O-ring	As required or every 3 to 6 months
Inspect and change vacuum pump fluid*	As required or every 3 to 6 months
Replace alumina in oil vapor traps*	As required
Clean the outside of the analyzer	As required or every 6 months
Test analyzer for leaks	As required or every 12 months
Replace vacuum pump exhaust filter	As required or every 12 months
Replace diaphragm in vacuum pump (if an oil-free pump is used)**	Every 12 months

* Oil-sealed vacuum pumps only.

** For oil-free pumps only: After 12 to 18 months of operation, the diaphragm in the pump will wear out and the pump becomes completely inoperable. To prevent instrument downtime due to an inoperable pump, it is recommend that the diaphragm be replaced by a Micromeritics Service Representative every 12 months.

Recovering from a Power Failure

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

Lubricating Elevator Drive Assembly

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

Cleaning the Analyzer

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

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

Cleaning the Analysis Dewar

When handling Dewars, be sure to observe the Dewar precautions outlined in chapter 3.

Ice and suspended frost particles may accumulate in the bottom of an analysis port Dewar. Particles or deposits exceeding 1/4-in. (0.64-cm) in depth may jam between the bottom of the sample tubes and the bottom of the Dewar, causing the Dewar not to raise fully. Accumulations of fine particles impede liquid nitrogen circulation around the bottom of the sample tubes. This causes the sample temperature to be slightly higher which, in turn, can cause pore volume measurement errors in those samples exhibiting high isotherm slope above 0.97 relative pressure.

Accumulated ice is likely to melt and form a pool of water in the Dewar if all liquid nitrogen evaporates. The water must be removed, otherwise it will solidify when liquid nitrogen is added and could press on the bottom of the sample tube causing breakage.

To ensure problems do not develop due to ice accumulation, check the Dewar after each use. Clean the Dewar on a weekly basis.

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

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

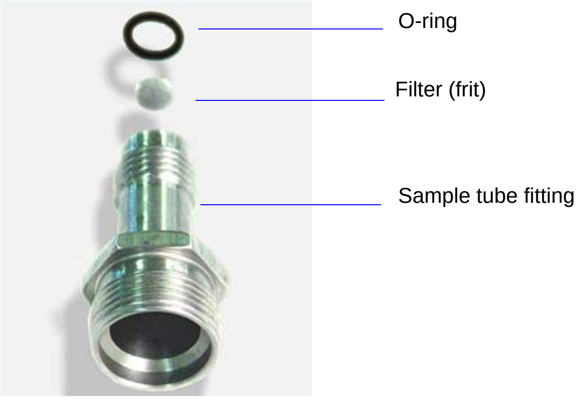
5. Rinse the Dewar with warm water to melt any ice accumulation which may remain in the Dewar, then dry thoroughly.

Replacing Port Filter (Frit) and O-ring

A filter (frit) is located in each analysis port. If the filter is contaminated, the contaminant may adsorb or desorb during analysis, affecting the results. A contaminated filter on the analysis port may be indicated as a leak or a free space reading much lower than normal.

A contaminated filter should be replaced as follows:

1. Select ***Unit [n]*** > ***Show Instrument Schematic***, then ***Unit [n]*** > ***Enable Manual Control***.
2. Right-click on the valve of the appropriate port and select **Close** to close the valve.
3. Use a wrench to remove the sample tube fitting from the port. Pry out the filter and O-ring.



To avoid degassing problems, the filter and O-ring should be clean and should not be touched with bare hands.

4. Replace the filter and O-ring. Carefully reassemble the sample tube fitting and reinstall on the sample port. Tighten by hand, then with a wrench to prevent leaks.

Replacing the Sample Tube O-ring

It is important to maintain a vacuum-tight seal near the top of the sample tube stem. If an O-ring becomes worn or cracked, it does not provide a good seal and will need to be replaced.

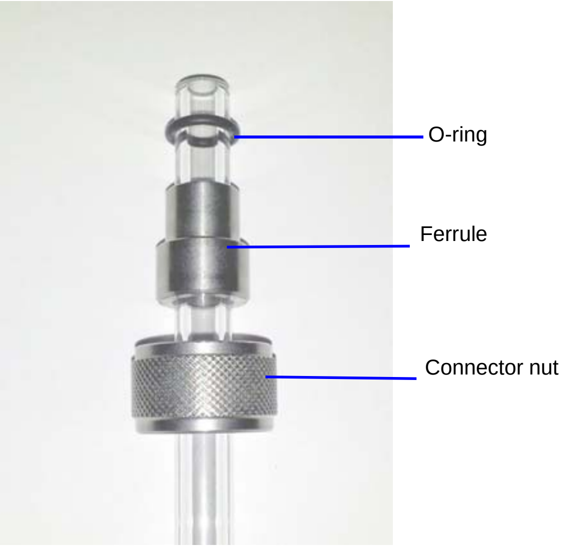
Before removing (or installing) a sample tube, ensure that the port valve is closed. Observe the instrument schematic to verify valve status.

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

Be careful not to let the sample tube connector nut drop onto the bottom of the tube as it may break the tube.

Be sure to use the proper size frit for the sample port. The instrument will not operate properly if the incorrect frit size is used.

2. Carefully pull the sample tube down until it is free from the port. It may be necessary to grasp the sample tube with both hands.



3. Remove the O-ring from the top of the sample tube and replace it with a new one.

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

4. After the new O-ring is in place, insert the sample tube back into the sample port until it is fully seated.

5. Slide the sample tube connector nut up the tube (the ferrule and O-ring will move along with the connector nut). Then, turning clockwise, ***hand-tighten*** the connector nut to the sample connector.

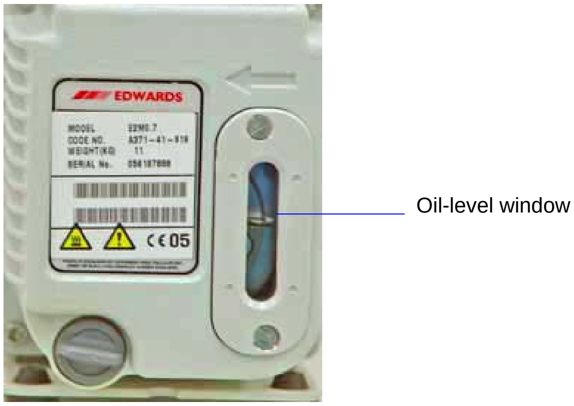
Inspecting and Changing Vacuum Pump Oil

This procedure is also shown in one of the How-To videos located on the Help menu.

The oil in the vacuum pump should be changed every three months when the efficiency of the vacuum pump declines (requiring increased time to reach vacuum levels) or if it becomes discolored. The oil is easily inspected to determine if a change is necessary.

Inspecting the Oil

View the vacuum pump oil through the oil-level window. The oil level should be midway between the indicators on the oil-level window when the pump is running. Oil in good condition is clean, clear or light in color and transparent.



- Change the oil if it has darkened.
- Add oil if it is below the midway level when the pump is running.

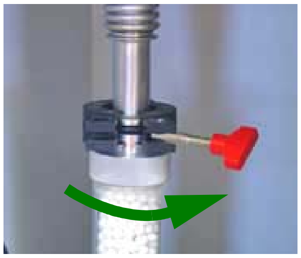
Changing or Adding Oil

Always drain the vacuum pump while the pump is warm and disconnected from the power source.

Use oil supplied by Micromeritics or refer to the vacuum pump manual for other acceptable oils.

1. Unplug the vacuum pump from the power source.

2. Loosen the wing nut on the clamp at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.



3. Grasp the handle on top of the vacuum pump and place it on a work table.

If adding oil, skip to step 5.

4. Drain the used oil:

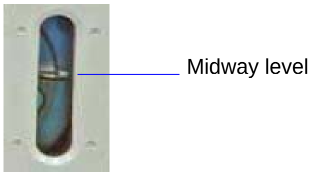
   a.) Place a waste container under the drain spout.

   b.) Remove the plug from the drain spout and allow the oil to drain into the waste container.

   c.) Replace the drain plug.

5. Remove the plug from the oil-fill port.

6. Slowly add oil to the port until the level is midway between the indicator lines in the oil-level window when the pump is running.



Do not allow oil to rise above the midway position when the pump is running. Doing so may cause oil to splash into the oil filter and contaminate it.

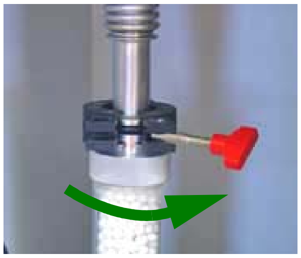
7. Check the washer or O-ring used at the oil-filling port and replace if necessary.
8. Insert the oil-fill plug and turn clockwise to tighten.
9. Check the alumina in the oil vapor trap. If most of the pellets are no longer white, replace the alumina before reattaching the vacuum pump.
10. Reconnect the vacuum pump hose.
11. Reconnect the power cord to the power source.
12. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the fresh fluid and to produce efficient vacuum operations.

Replacing the Alumina in the Oil Vapor Trap

The activated alumina in the oil vapor trap becomes saturated during use. The alumina should be inspected periodically and replaced when most of the alumina pellets are no longer white.

Do not perform the following procedure on used alumina. The resultant oil vapors may cause a fire or an explosion.

1. Disconnect the vacuum pump from the analyzer and place it on a work table.
2. Loosen the wing nut on the clamp at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.



3. Loosen the wing nut on the clamp at the bottom of the oil vapor trap. Swing the clamp open and remove the trap.

4. Remove one end fitting from the trap body and dispose of the used alumina in an appropriate manner.
5. Wash the trap body with a detergent-based soap. Rinse with water, then rinse with isopropyl or ethyl alcohol. Set the trap aside and allow to dry thoroughly.

Exposure of the trap body to oil vapor may cause small cracks on the inside surface of the trap body. Under normal circumstances, these cracks will not cause problems or leaks.

Do not perform the following procedure on used alumina. The resultant oil vapors may cause a fire or an explosion.

6. Prepare fresh alumina as follows:

   a.) Preheat the oven to 300 °C.

   b.) Pour approximately 180 grams of fresh alumina into a glass or metal container (approximately 250 mL if a graduated beaker is used). Place the container in the oven.

   c.) Bake the alumina for two hours at 300 °C.

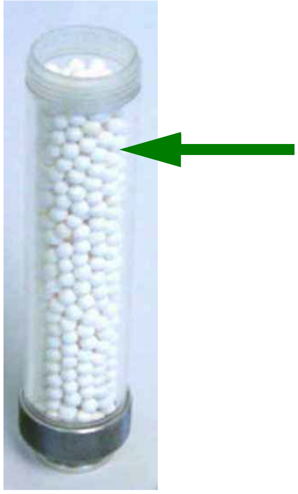
   d.) Remove the baked alumina from the oven and allow it to cool until luke warm. A desiccator may be used to speed the cooling process.

7. Use a small spatula to gently pry the O-ring from the end fittings of each end of the trap body.

8. Inspect the O-rings:

   - If dusty, clean with a lint-free tissue.
   - If damaged, replace with a new O-ring.

9. Screw one of the end fittings onto the trap body.



10. Ensure the trap body is dry and the alumina is lukewarm. Pour the alumina pellets into the trap until level with the top of the trap body.
11. Screw the other end fitting back onto the trap and tighten securely by hand.
12. Lightly tap both ends of the trap body on the work surface to remove remaining dust from the pellets.

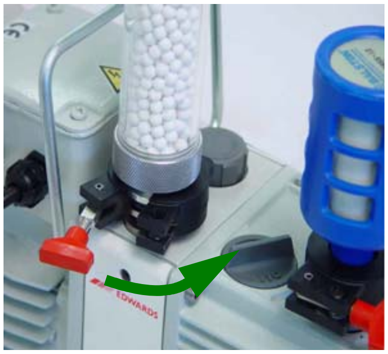
13. Inspect the centering ring before placing it back on the intake port. If it appears to be flattened, replace it. A flattened centering ring can cause vacuum leaks.

    There are two types of centering rings. Use the one with the smaller opening at the intake port.

14. Place the centering ring in place on the intake port.

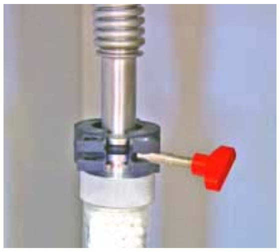
15. Place the trap on the centering ring.

16. Open the clamp and place it around the flange of the intake port and the flange of the trap. Swing the clamp fastening screw toward the intake port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.



17. Reconnect the hose from the analyzer to the oil vapor trap.

    a.) Place the clamp around the vacuum pump hose flange and vapor trap.



b.) Swing the c-clamp fastening screw around until it fits into the slot on the other half of the clamp. Tighten the wing nut securely by hand.

18. Plug the pump power cord into the power source.

19. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the fresh oil and to produce efficient vacuum operations.

Changing the Vacuum Pump Exhaust Filter

The gases used by the analyzer are exhausted by the vacuum pump. An exhaust filter is installed on the exhaust port of oil-filled pumps. The filter minimizes the release of oil vapor and should be replaced when it becomes so saturated with oil that it is ineffective.

Exhaust filters are used to minimize the release of oil vapors. The gases are diluted substantially upon release from the vacuum pump; however, it may be necessary in some locations to provide a fume hood for protection from hazardous gases and vapors released into the work area.

1. Turn the power to the unit off by either removing the plug from the power source or by turning the power switch to the OFF position.

2. Loosen the wing nut of the clamp at the vacuum pump exhaust port. Swing the clamp away from the exhaust port and remove it.

3. Remove and discard the exhaust filter. Do not remove the centering ring.

4. Make sure the centering ring is in place on the exhaust port.

5. Place the new filter on the centering ring.

6. Open the clamp and place it around the exhaust port flange and the exhaust filter flange. Swing the clamp fastening screw toward the exhaust port until it fits into the slot in the other half of the clamp. Tighten the wing nut securely by hand.

7. Turn the power to the unit on by replacing the plug back into the power source or by turning the power switch to the ON position.

Performing a Leak Test

A service representative may request that a leak test be performed to determine if there is a system leak. The service representative may request a copy of the report generated by this test.

The following information is displayed during the test:

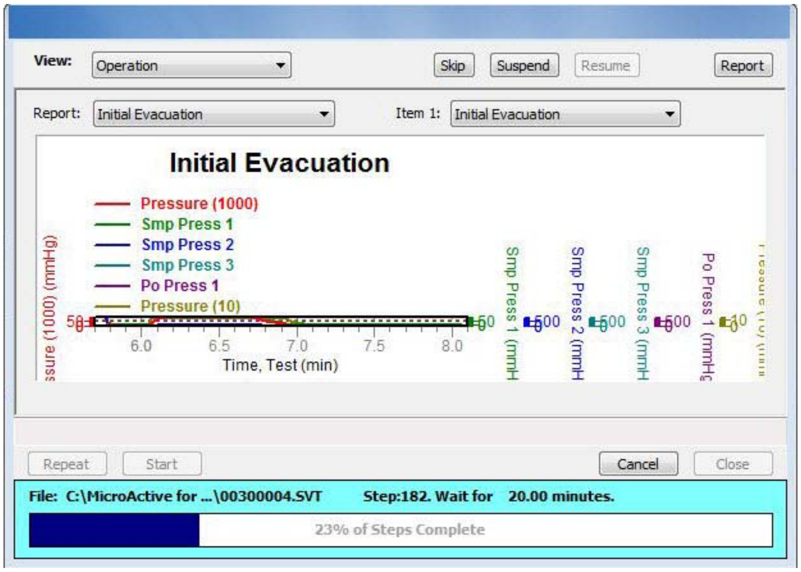
- Prompts on preparing the instrument for the test
- Approximate time period of the test
- Prompts in which an operator response is required

1. To start diagnostic testing, go to ***Unit [n]*** > ***Diagnostics***.

2. Click the down arrow in the **Test** field and select **System Leak Test Rev. [*rev*]**.

3. Select **Report after test** and **Preview** as the destination. Click **OK**.

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



View: Operation

Report: Initial Evacuation

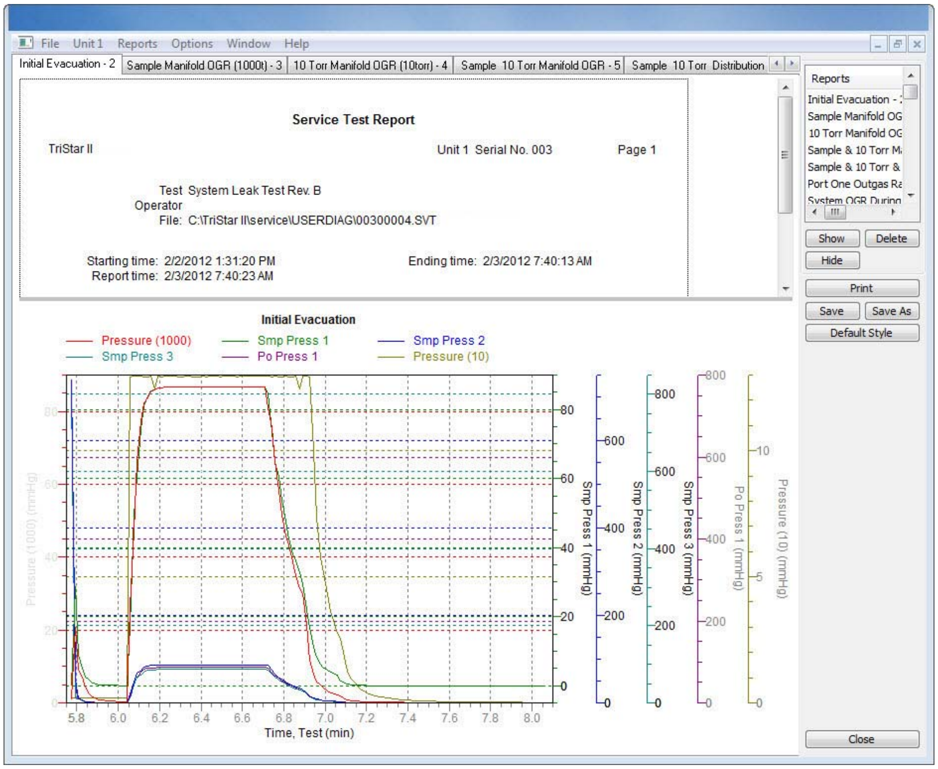
Item 1: Initial Evacuation

File: C:\MicroActive for ...\00300004.SVT Step:182. Wait for 20.00 minutes.

23% of Steps Complete

5. A popup window indicates the test is complete. Click **OK.**

6. Click the **Report** button to display the report.



Service Test Report

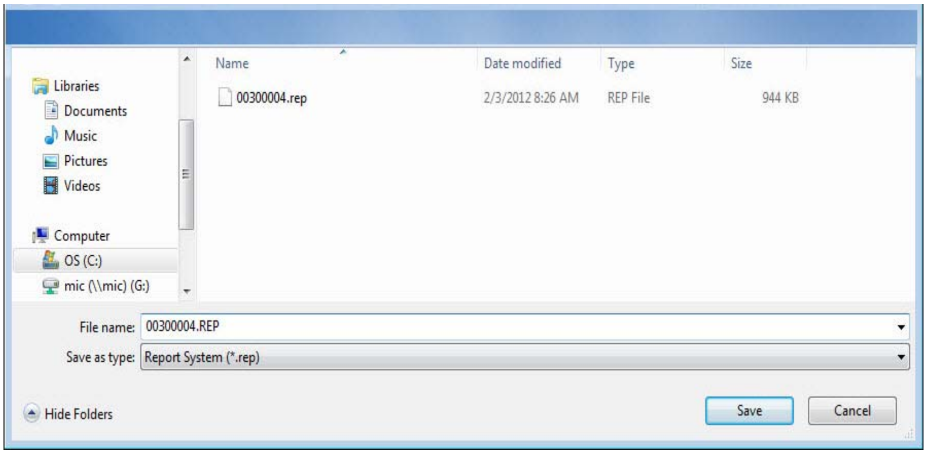
TriStar II Unit 1 Serial No. 003 Page 1

Test System Leak Test Rev. B
Operator
File: C:\TriStar II\service\USERDIAG\00300004.SVT

Starting time: 2/2/2012 1:31:20 PM Ending time: 2/3/2012 7:40:13 AM
Report time: 2/3/2012 7:40:23 AM

7. Verify that all tests have a **Passed** status by clicking the tabs and looking for the **Passed** status for each test run.

8. Click **Save As** and accept the default name displayed the **File name** field.



00300004.rep 2/3/2012 8:26 AM REP File 944 KB

File name: 00300004.REP

Save as type: Report System (*.rep)

9. Click **Save**. Make a note of the directory and filename, then click **Close**.

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

Connecting Gases

This procedure is also shown in one of the video tutorials located on the Help menu.

Guidelines for Connecting Gases to the Analyzer

Guidelines when installing regulators and gas lines:

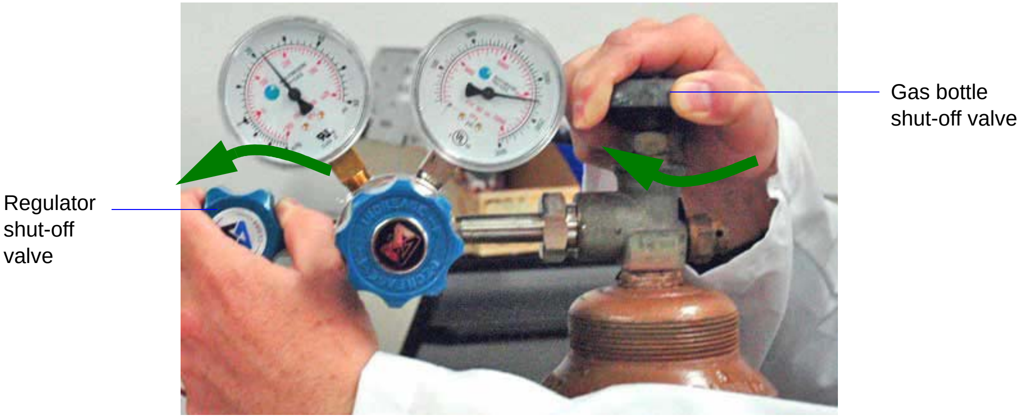
- Place gas bottles close to the analyzer. Using gas line extenders on gas bottles located in remote areas may degrade gas quality and reduce pressure.
- Use a retaining strap (or other appropriate tether) to secure the gas bottle.
- Carefully route the gas lines from the bottle to the analyzer avoiding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

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

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

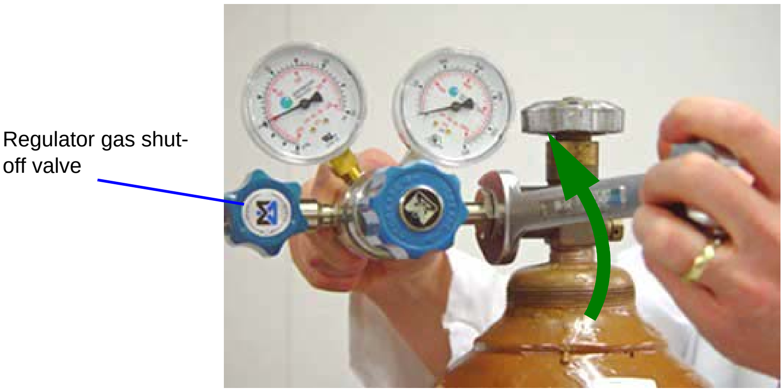
Disconnecting the Depleted Bottle

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



2. Both gauges should read at or near zero. If not, open the regulator gas shut-off valve to release gas. It is not necessary to disconnect the gas line from the regulator or the instrument.

3. Use an appropriate wrench to loosen the nut at the regulator/gas bottle connection then remove the regulator from the bottle.



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

Connecting a Replacement Gas Bottle

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

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

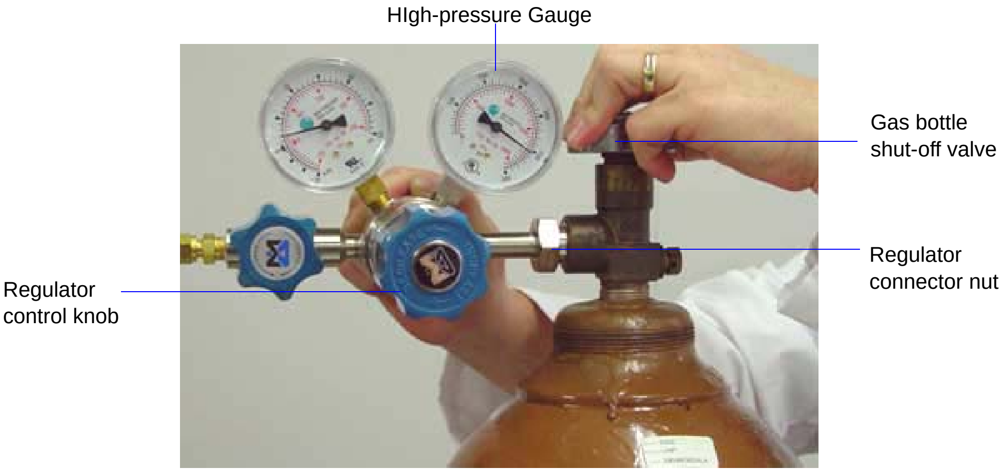
A power failure or loss of cryogen can result in dangerous pressures in the sample tube. The analyzer uses pressure relief valves to vent this pressure into the instrument cabinet and return the instrument to a safe condition. When using toxic or flammable gases, additional venting of the cabinet may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

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

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

Overtightening the fitting may cause a leak.

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



a.) Turn the regulator control knob fully counterclockwise.

b.) Slowly open the gas bottle shut-off valve then close it.

c.) Observe the pressure on the high-pressure gauge.

- If the pressure is stable, proceed with the next step.
- If the pressure decreases, tighten the regulator connector nut until it becomes stable.

4. Purge the air from the lines.

a.) Turn the regulator clockwise until the low pressure gauge shows a few pounds of pressure.

b.) Turn the regulator shut-off valve counterclockwise to open.

c.) Open the gas bottle shut-off valve to flow gas.

d.) Close the regulator shut-off valve to stop flow.

e.) Close the gas bottle valve.

5. Set the instrument pressure.

a.) Turn the regulator control knob clockwise until the low-pressure gauge reads 15 psig (103 kPag).

b.) Open the regulator shut-off valve.

c.) Open the gas bottle shut-off valve and flow gas for 10 to 30 seconds.

d.) Close the gas bottle shut-off valve.

6. If the gas line to the instrument inlet was previously disconnected, reconnect it now.

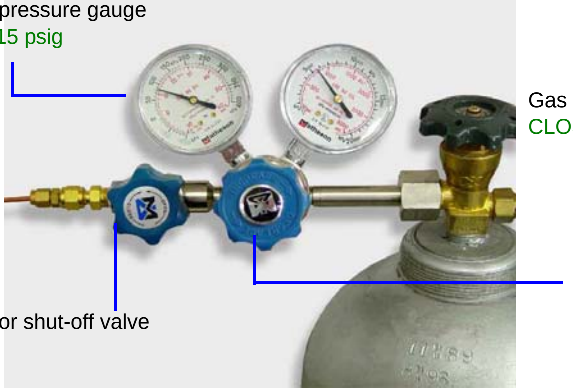
7. Verify the line for the newly connected gas is clean.

8. If the previously disconnected gas has been reconnected, resume operation.
   If a different gas has been connected, the change must be specified in the software. Refer to **Specifying Gas Ports**, page **7-25**.

Cleaning and Verifying the Gas Line

Always clean the gas lines and verify there are no leaks at the connections after a gas bottle is connected. This test examines the gas line from the instrument to the gas bottle, then from the instrument to the regulator shut-off valve. A report is generated at the completion of the test verifying that it has passed or failed. Causes and corrective action for a failure are provided.

Before beginning, confirm that the state for valves and the low-pressure gauge are as follows:



Low-pressure gauge
14 - 15 psig

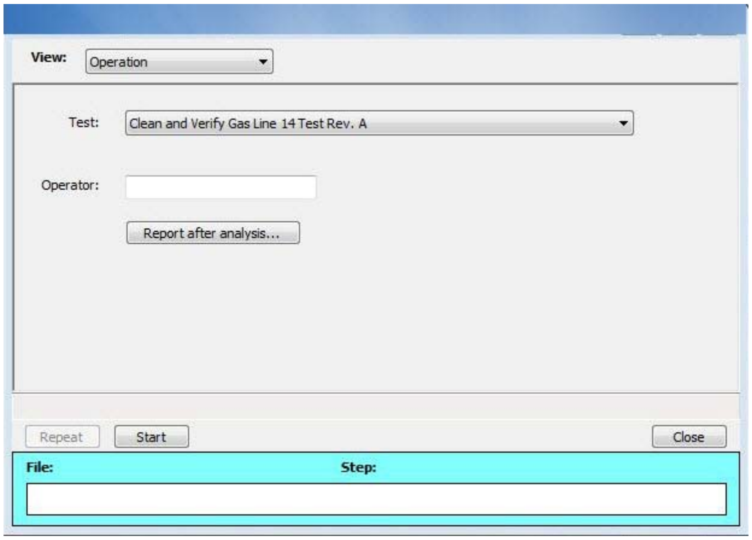
Gas bottle shut-off valve
CLOSED

Regulator control knob
OPEN

Regulator shut-off valve
OPEN

1. To start diagnostic testing, go to ***Unit [n]*** > ***Diagnostics***.

2. Click the down arrow to the right of the **Test** field and select **Clean and Verify Analysis Gas Line Rev [*n*] Test Rev [*n*].**



View: Operation

Test: Clean and Verify Gas Line 14 Test Rev. A

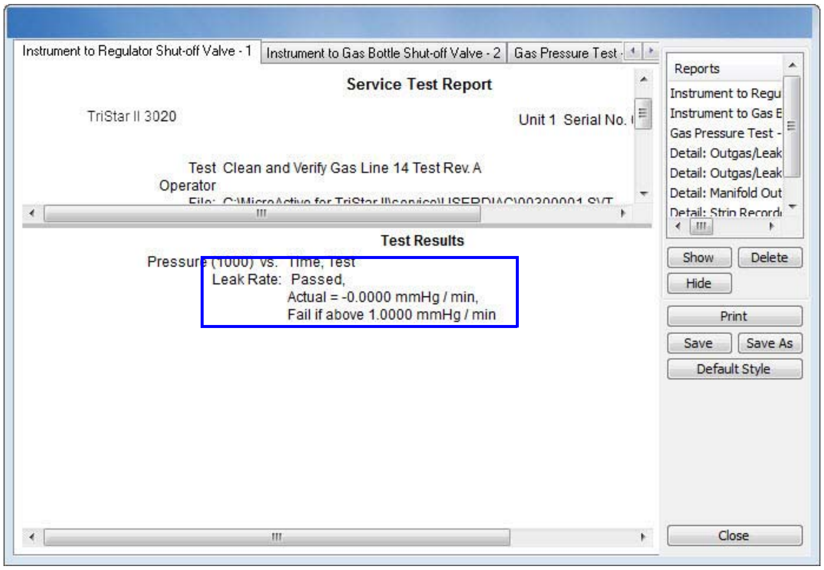
Operator:

3. Click **Report after analysis** and select **Preview** as the destination. Click **OK**.

4. Click **Start**. From the **View** dropdown list, select either **Operation**, **Instrument Log**, or **Instrument Schematic.** Refer to **Sample Analysis**, page **4-3**.

5. A series of prompts display on the screen requiring operator response. The length of time a test will run is also indicated on the screen.

6. A popup window indicates the test is complete. Click **OK**. The report displays on the screen.



Service Test Report

TriStar II 3020 Unit 1 Serial No.

Test Clean and Verify Gas Line 14 Test Rev. A
Operator

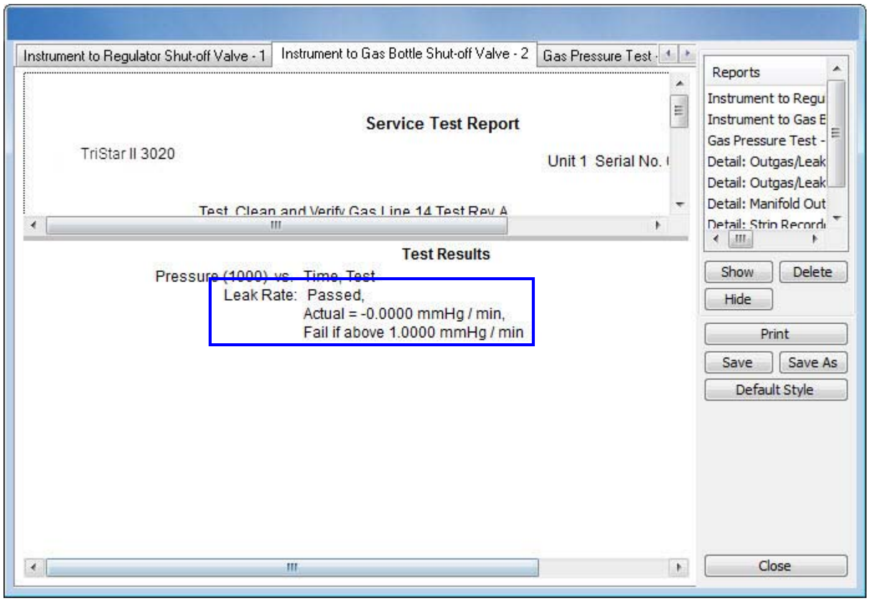
Test Results

Pressure (1000) vs. Time, Test
Leak Rate: Passed,
Actual = -0.0000 mmHg / min,
Fail if above 1.0000 mmHg / min

On the report, the **Leak Rate** field on the **Instrument to Regulator Shut-off Valve -1** report should read **Passed**. A **Passed** reading indicates the gas line from the instrument to the regulator is clean and leak-free.

If the **Leak Rate** field indicates **Failed**, a leak is indicated between this connection. Check the connections from the instrument to the gas regulator valve. Tighten as necessary, then run the test again.

7. Click the **Instrument to Gas Bottle Shut-off Valve - 2** tab to display its report.



Service Test Report

TriStar II 3020

Unit 1 Serial No.

Test Clean and Verify Gas Line 14 Test Rev A

Test Results

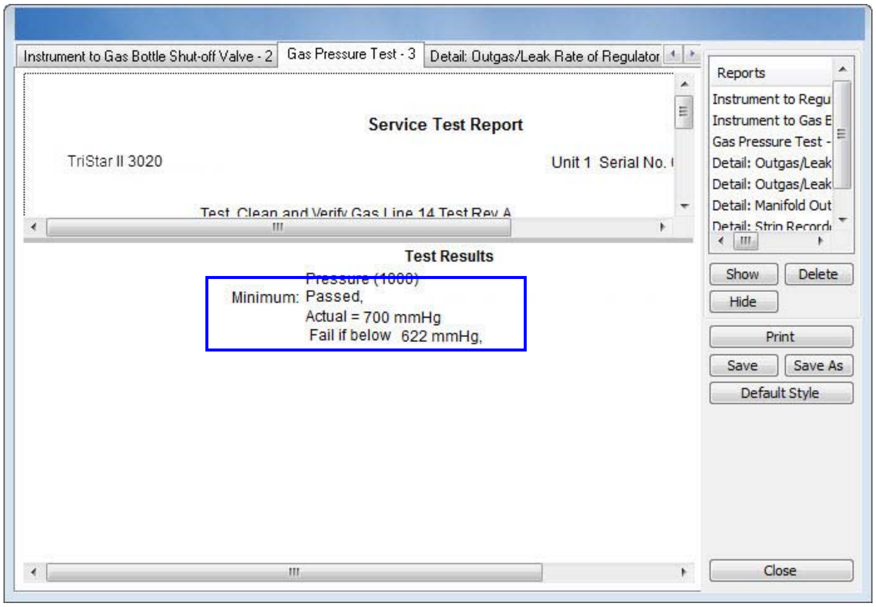
Pressure (1000) vs. Time, Test

Leak Rate: Passed,
Actual = -0.0000 mmHg / min,
Fail if above 1.0000 mmHg / min

On the report, the **Leak Rate** field should read **Passed**. A **Passed** reading indicates the gas line from the instrument to the regulator is clean and leak-free.

If the **Leak Rate** field indicates **Failed**, a leak is indicated between this connection. Check the connections from the regulator shut-off valve to the gas bottle shut-off valve. Tighten as necessary, then run the test again.

8. Click the **Gas Pressure Test - 3** tab to display its report.



Service Test Report

TriStar II 3020 Unit 1 Serial No.

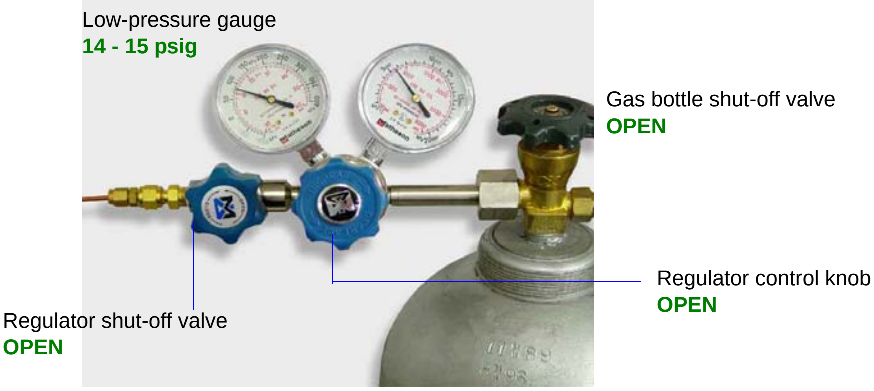
Test Clean and Verify Gas Line 14 Test Rev A

Test Results

Pressure (1000)
Minimum: Passed,
Actual = 700 mmHg
Fail if below 622 mmHg,

On the report, the **Minimum** field should read **Passed**. A **Passed** reading indicates all valves are in the proper state for operation.

If the **Minimum** field indicates **Failed**, one or more valves is not in the proper position. Set the valves as shown below and ensure the appropriate pressure is displayed on the low-pressure gauge.



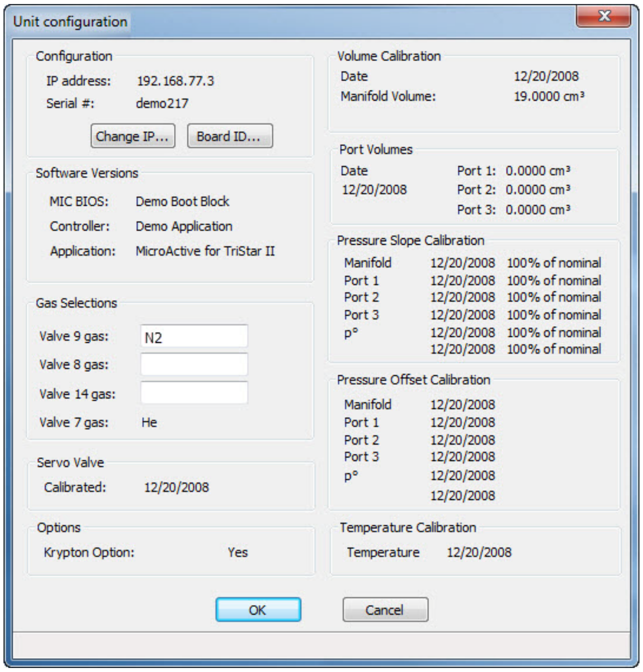
If running the test again, close the gas bottle valve before starting the test.

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

Specifying Gas Ports

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

1. Select ***Unit [n]*** > ***Unit Configuration.***



Configuration
IP address: 192.168.77.3
Serial #: demo217

Software Versions
MIC BIOS: Demo Boot Block
Controller: Demo Application
Application: MicroActive for TriStar II

Gas Selections
Valve 9 gas: N2
Valve 8 gas:
Valve 14 gas:
Valve 7 gas: He

Servo Valve
Calibrated: 12/20/2008

Options
Krypton Option: Yes

Volume Calibration
Date 12/20/2008
Manifold Volume: 19.0000 cm³

Port Volumes

Date		
12/20/2008	Port 1:	0.0000 cm³
	Port 2:	0.0000 cm³
	Port 3:	0.0000 cm³

Pressure Slope Calibration

Manifold	12/20/2008	100% of nominal
Port 1	12/20/2008	100% of nominal
Port 2	12/20/2008	100% of nominal
Port 3	12/20/2008	100% of nominal
p°	12/20/2008	100% of nominal
	12/20/2008	100% of nominal

Pressure Offset Calibration

Manifold	12/20/2008
Port 1	12/20/2008
Port 2	12/20/2008
Port 3	12/20/2008
p°	12/20/2008
	12/20/2008

Temperature Calibration
Temperature 12/20/2008

2. In the **Gas Selections** group box, enter the mnemonic for the gas(es) attached to the gas inlets.

3. Click **OK**, then click **Close**.

Calibrating the System

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

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

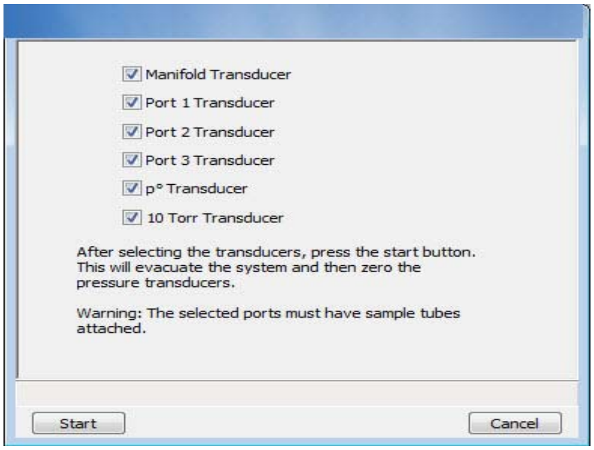
The following calibrations can be performed without the assistance of a service representative:

- Zero pressure
- Match transducers
- Servo valve

Zero Pressure

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

1. Install a blank sample tube on each applicable port.
2. Go to ***Unit [n]*** > ***Calibration*** > ***Zero Pressure.***



☑ Manifold Transducer
☑ Port 1 Transducer
☑ Port 2 Transducer
☑ Port 3 Transducer
☑ p° Transducer
☑ 10 Torr Transducer

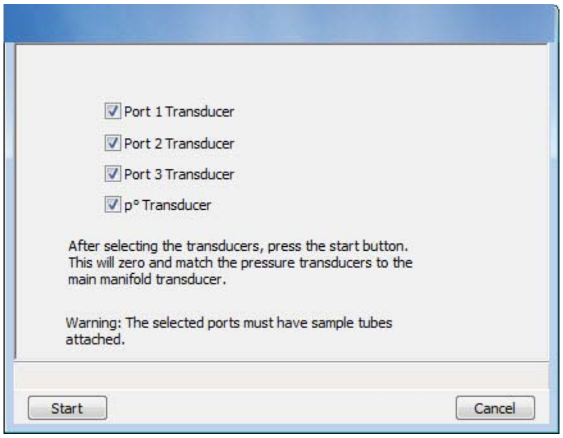
After selecting the transducers, press the start button. This will evacuate the system and then zero the pressure transducers.

Warning: The selected ports must have sample tubes attached.

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

Match Transducers

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



☑ Port 1 Transducer
☑ Port 2 Transducer
☑ Port 3 Transducer
☑ p° Transducer

After selecting the transducers, press the start button. This will zero and match the pressure transducers to the main manifold transducer.

Warning: The selected ports must have sample tubes attached.

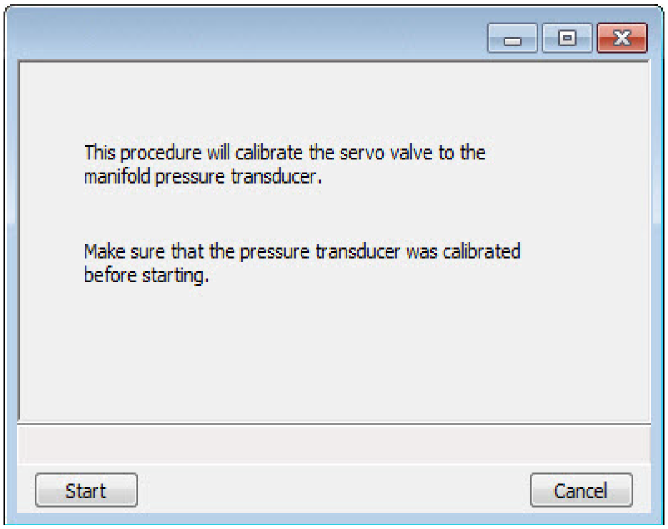
A sample or Po tube must be installed on each selected port before clicking **Start**.

Servo Valve

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

1. Select ***Unit [n]*** > ***Calibration*** > ***Servo Valve.***

2. Click **Start**. The window closes when the calibration is complete.



This procedure will calibrate the servo valve to the manifold pressure transducer.

Make sure that the pressure transducer was calibrated before starting.

8. ORDERING INFORMATION

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

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

Please use the following information to place an order.

Part Number	Item and Description
Analyzer Optional Equipment	
021-00000-00	Model 021 LN_2 transfer system, for easy filling of sample Dewars; includes 47-L dewar, mobile platform, and pump with 100-cm discharge line
060-00030-00	FlowPrep 060, degasses up to six samples at up to 400 ºC with flowing gas. A gas source and regulator is required.
061-00030-00	VacPrep 061, degasses up to six samples at up to 400 °C; uses flowing gas or vacuum. A vacuum pump, gas source, and regulator is required.
065-00000-00	SmartPrep 065, Windows interface provides programmable ramp and soak rate for degassing up to six samples with flowing gas. A gas source and regulator is required.
Cables	
003-63801-01	Cable, Ethernet straight-thru
Dewar and Accessories	
004-34891-00	Stainless steel Dewar, 4.0 liters
240-25895-00	Insulator, Open Dewar
240-25901-00	Dip stick; for checking liquid nitrogen level in Dewar
300-31802-02	Dewar Cover (for glass or stainless steel Dewar), for 1/4 in. and 3/8 in. sample tubes
300-31802-03	Dewar Cover (for glass or stainless steel Dewar), for 1/2 in. sample tube
303-25825-00	Glass Dewar, 3.0 liters

Part Number	Item and Description *(continued)*
Gas Bottle Accessories	
004-25549-00	Reducer, 1/8 in. tube × 1/4 in. tube
004-33601-00	Expansion Kit; adds an additional outlet to the gas regulator
004-33602-00	Pressure Relief Kit; prevents excessive gas pressure in the event of regulator failure (not to be used with toxic gases)
004-62230-32	Gas Regulator, CGA 320, 30 psig (CO_2)
004-62230-58	Gas Regulator, CGA 580 fitting, 30 psig (He, N_2)
290-25846-00	Gas Inlet Line, 1/8 in. × 6 ft. long, copper
290-25846-01	Gas Inlet Line, 1/8 in. × 16 ft. long, copper
Isothermal Jacket	
302-25822-00	Isothermal Jacket, for 1/4 in. sample tube, 5.25 in.
302-25822-01	Isothermal Jacket, for 3/8 in. sample tube, 5.25 in.
302-25822-02	Isothermal Jacket, for 1/2 in. sample tube, 5.25 in.
Operating Supplies	
004-25491-02	Centering ring, NW 40, Buna-N, AL
004-25630-00	Centering ring, NW 16
302-33600-00	Extended Operating Supplies (for 3/8 in. Samples Tubes. Includes additional filler rods, O-rings, sample tubes, stoppers, etc. beyond those provided with the initial instrument supplies.
302-33600-01	Extended Operating Supplies (for 1/2 in. Samples Tubes. Includes additional filler rods, O-rings, sample tubes, stoppers, etc. beyond those provided with the initial instrument supplies.
302-33600-02	Extended Operating Supplies (for 1/2 in. Flat Bottom Samples Tubes. Includes additional filler rods, O-rings, sample tubes, stoppers, etc. beyond those provided with the initial instrument supplies.

Part Number	Item and Description *(continued)*
Reference Material	
004-16816-00	Reference Material, Alumina, ~ 0.25 m^2/g, 15 g; for krypton analysis
004-16818-00	Reference Material, Glass, ~ 5.8 m^2/g, 10 g
004-16821-00	Reference Material, Silica alumina, ~ 215 m^2/g,10 g
004-16833-00	Reference Material, Carbon black, ~ 30.6 m^2/g, 10 g
Sample Tubes and Accessories	
004-25022-00	O-Ring, Buna-N size 012; for 3/8 in. sample tube
004-25466-00	O-Ring, Buna-N size 010; for sample port and 1/4 in. sample tube
004-25469-00	O-Ring, Buna-N size 014; for 1/2 in. sample tube
004-27046-00	Frit, 40 µm, for sample port
004-32004-00	Stopper, rubber, for 3/8 and 1/2 in. sample tubes
004-32604-08	Plastic Cap, .225 × 5/16, Vinyl for 1/4 in. sample tubes
004-54104-00	Brush, for cleaning sample tubes
004-54130-00	Seal Frit extraction tool
004-54618-00	Tool, for removing sample port O-ring
240-14855-00	Rack, sample tube
240-25853-00	Funnel, sample tube
260-25843-00	Ferrule, for 1/2 in. sample tube
260-25873-00	Sample port plug (glass)
260-25890-00	Seal Frit assembly for 1/2 in. sample tube
260-25891-00	Seal Frit opener
300-25825-00	Ferrule, for 1/4 in. sample tube

Part Number	Item and Description *(continued)*
300-25826-00	Ferrule, for 3/8 in. sample tube
300-32800-00	Support, sample weighing, for flat bottom sample tube
302-25821-00	Hanging Filler Rod, for 1/4 in. sample tube
302-25821-01	Hanging Filler Rod, for 3/8 in. sample tube
302-25821-02	Hanging Filler Rod, for 1/2 in. sample tube
302-33601-00	Sample Tube Kit, 1/4 in.; includes six each sample tubes, O-rings, and plastic caps; three each isothermal jackets, filler rods, and ferrules; and a Dewar cover
302 -33602-00	Sample Tube Kit, 3/8 in.; includes six each sample tubes, O-rings, and stoppers; three each isothermal jackets, filler rods, and ferrules; one sample tube brush; and a Dewar cover
302 -33603-00	Sample Tube Kit, 1/2 in.; includes six each sample tubes, O-rings, and stoppers; three each isothermal jackets, filler rods, and ferrules; one sample tube brush; and a Dewar cover
302-33604-00	Sample Tube Kit Flat Bottom; 1/4 in. (6.4 mm) OD stem × 3.5 mm ID
302-33605-00	Sample Tube Kit Flat Bottom; 3/8 in. (9.5 mm) OD stem × 4.7 mm ID
302-33606-00	Sample Tube Kit Flat Bottom; 1/2 in. (12.7 mm) OD stem × 9 mm ID
302-33607-00	Seal Frit Kit; includes three seal frits, nine openers, three filler rods, installation tool and instructions, for 1/2 in. sample tube
302-61001-00	Sample Tube, 1/4 in. stem OD; for analyzing powdered samples where free space management (with a filler rod inserted) is critical
302-61001-01	Sample Tube, 3/8 in. stem OD; for analyzing any type of sample
302-61001-02	Sample Tube, 1/2 in. stem OD; for analyzing larger pieces of granular or pelleted samples
302-61002-00	Sample Tube, 1/4 in. stem OD, flat bottom
302-61002-01	Sample Tube, 3/8 in. stem OD, flat bottom
302-61002-02	Sample Tube, 1/2 in. stem OD, flat bottom

Part Number	Item and Description *(continued)*
302-61005-02	Filler Rod, for 1/2 in. sample tube when using the Seal Frit
Software and Manuals	
202-33027-00	MicroActive; includes software and operator’s manual. This program allows you to generate and manipulate data on a computer other than the one controlling the analyzer or to compare data from another instrument.
202-42827-00	MicroActive Operator’s manual
303-20800-00	TriStar II Plus- current version software
303-42800-00	TriStar II Plus - operator’s manual
Vacuum Pump and Accessories	
004-16003-01	Oil, vacuum pump, 1 Liter
004-16830-00	Activated Alumina, 500 grams; for oil vapor trap
004-25491-02	Centering ring, NW 40, Buna-N, AL
004-25630-00	Centering ring, NW 16
004-25652-00	O-ring, size 217, for oil vapor trap
004-27040-00	Filter, for vacuum pump exhaust
004-62023-01	Service Kit, for vacuum pump P/N 062-00004-00 (for Edwards and VacuuBrand)
062-00000-11	Vacuum Pump with built-in anti-suckback valve, 100/120 VAC, includes hose kit
062-00000-23	Vacuum Pump with built-in anti-suckback valve, 220/240 VAC, includes hose kit
062-00004-00	Hybrid turbo vacuum pump assembly for Krypton units only
062-33002-00	Activated alumina oil vapor trap, for one vacuum pump; for nitrogen units only
200-25879-00	Funnel, for filling vacuum pump with oil

Part Number	Item and Description *(continued)*
Valves	
003-22617-00	Valve, solenoid, 24 VDC 10-32 Mt, 0.025 in. orifice, for standard evacuation (Valves 1 through 10 and 14). Valve has yellow plastic cap.
003-22626-00	Valve, solenoid, 24 VDC 10-32 Mt, 0.060 in. orifice, for fast evacuation (Valves 11, 12, and 13). Valve has green plastic cap.

A. FORMS

This appendix contains the following form:

- Sample Data Worksheet

Copy and use this form as needed.

micromeritics®

Sample Data Worksheet

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

Sample Tube Identification:______________________________

Sample Mass (g)			
NOTE: Record all values in grams.			
	Before Degas	**After Degas**	**After Analysis**
A. Mass for empty sample tube set:	________		
B. Sample tube set plus sample mass:	________	________	________
C. Sample mass (B - A):	________	________	________

Degas Information	
Degas apparatus:	______________________________
Temperature (ºC):	______________________________
Time (hours):	______________________________
Actual time started:	______________________________
Actual time finished:	______________________________

Degas notes: ____

____

____

____

____

____

B. ERROR MESSAGES

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

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

2400 Series

2401- FATAL ERROR: [*error message*]

Cause:	An internal processing and/or hardware error has occurred during communication with the analyzer.
Action:	Contact your Micromeritics service representative.

2430- Error accessing file [*file name*], error code = [*n*].

Cause A:	Media may be damaged.
Action A:	Clean the media drive. If this does not eliminate the problem, attempt operation using a backup copy of the file.
Cause B:	Hard disk may be damaged.
Action B:	Contact your Micromeritics service representative.
Cause C:	A software error occurred when the file was accessed.
Action C:	Contact your Micromeritics service representative.
Cause D:	The file name specified contains one or more invalid characters.
Action D:	Enter a valid file name. Refer to the operating systems manual.

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

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

2432- Invalid response from MMI ‘FILE_READ’ request.

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

2433- New entries have been found in this directory. Refresh the directory information?

Cause:	Several analyzer files (sample information, analysis conditions, adsorptive properties or report options) have been added to this directory by some function other than the analyzer program.
Action:	Select **Yes** to update the directory information with data from each new file. This operation may take a minute. Select **No** to locate the file manually. This option may be feasible if a large number of files have been copied into the directory and the name of the file is known.

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

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

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

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

2437- File/Overlay file [*file name*] does not exist.

Cause:	The entered file specification does not exist.
Action:	Enter an existing file specification or select a file name from the list box.

2439- Could not register file.
2440- Subset not found.
2441- Seek within file failed.
2442- Bad header in subset file.
2443- Subset owner denied access.
2444- Not a valid file format.
2445- Subset wrote the wrong amount of data.
2446- Error reading data.
2447- Error writing data.

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

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

Cause:	A working directory specified in the .INI file is invalid or has been moved or deleted.
Action:	The installation directory will be substituted. The next time a file is opened, use the **Directories** list to move to the correct directory.

2449- This field does not contain a valid file specification.

Cause:	An invalid file name was entered.
Action:	See the description of file naming conventions in a Windows manual and re-enter the name.

2450- Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?

Cause:	An automatic analysis (an analysis in which sample files are created using the defaults) was processing while editing the defaults.
Action:	Finish the edit session of the defaults and close the window then restart the automatic analysis.

2451- Deleted entries have been found in this directory. Refreshing the directory information.

Cause:	Informational message only indicating the system is looking for directory entries that cannot be found.
Action:	Wait a few moments for the system to finish refreshing and retry the operation again.

2456- Insufficient file handles available. Application cannot continue.

Cause:	More than 50 files are open at the same time.
Action:	Refer to an operating system manual and set the limit for open files to 50 or greater.

2458- An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause:	An attempt was made to exit the application while the instrument was performing a critical operation. This operation must be completed before the application can be stopped.
Action:	Wait a short time and attempt to stop the application again.

2459- An instrument is busy. A delay in restarting this application could result in loss of new data. Continue with program Exit? [*Yes, No*]

Cause:	An attempt was made to exit the application while an analysis was in progress. While this is possible, the data collected when the application is inactive will not be permanently recorded until the application is re-started. A power failure to the instrument could cause some data to be lost.
Action:	If not concerned with the potential for loss of data should a power failure occur, click **Yes** to continue; otherwise click **No**.

2460- Fatal Communications error on [*Unit n - S/N: nnnn*]

Cause:	There was a fatal error in communication between the application and the software in the instrument. All displays for that instrument will be closed.
Action:	Ensure that the analyzer is connected to the computer on the communications port configured in the Setup program. Stop and restart the analyzer software. Contact your Micromeritics service representative.

2461- No instruments are in operation. This application will unconditionally terminate.

Cause:	At least one instrument must be active for the application to operate. The initialization of all of the instruments configured with the Setup program has failed. The application stops.
Action A:	Usually this message is preceded by another message giving the reason for the instrument's failure to initialize. Refer to the instructions for that message.
Action B:	Ensure that the instrument is attached to the computer on the communications port configured with the Setup program. Verify that the instrument has the power switch in the ON position and that the light on the front panel is illuminated. If the application continues to fail in its attempts to initialize the instrument, contact your Micromeritics service representative.

2477- Unit [*n*] - S/N: [*n*] did not properly initialize.

Cause:	The software was unable to initialize this instrument. This is usually caused by one of the conditions listed in the error messages above.
Action:	Correct the problem as described above then restart the application.

2478- Error copying sequential data segment.

Cause:	An internal processing and/or hardware error occurred while accessing a portion of a sample file.
Action:	Confirm that the media being accessed does not contain errors, for example, consider using a utility, for example, ScanDisk. Contact your Micromeritics service representative.

2479- Unit [*n*]; Serial [*n*] The instrument is busy performing an operation of which this application is unaware. Do you want to cancel? [*Yes, No*]

Cause:	During initialization of the application the status of the analyzer was found to be in a different state than expected.
Action:	Click **Yes** to cancel the operation in process allowing the analyzer to reset and continue with initialization. Click **No** to cancel the initialization process. If this error message continues, verify that files in the application directory structure are not being changed or removed.

2480- File [*name*] cannot be analyzed. It is currently being edited.

Cause:	An attempt was made to start an analysis using a file that is open for editing.
Action:	Finish editing the file, save and close it then start the analysis.

2481- Error accessing the sample information file [*file name*].

Cause:	An unexplained error prevented access to this file.
Action:	The hard disk drive may be corrupt. Run diagnostics.

2482- File cannot be opened for writing. It is already in use.

Cause:	An attempt was made to open a file currently being used.
Action:	Locate the application using the file (in the Micromeritics application, use the Windows menu item to get a list of all windows, one of which may contain this file).

2483- An analysis cannot be performed on [*file name*]. It is open for editing and contains errors.

Cause:	An attempt was made to use a sample file containing errors that is currently open.
Action:	Go to the window containing the file, correct the errors and save it.

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

Cause:	An attempt was made to start an analysis using a file that contains unsaved changes and is open for editing.
Action:	Select **Yes** to save the changes and proceed with the analysis. Select **No** to cancel the analysis and continue editing the Sample Information file.

2485- The service test file has an invalid status and cannot be used for this analysis.

Cause:	The selected service test file has a status other than *No Analysis*.
Action:	Select a different service test file or create a new one and use **Replace All** to copy parameters from the file originally selected.

2486- Could not construct [*name*] report type. Program will terminate.
.2487- Could not start report generator. Error code [*number*]. Program will terminate.

Cause A:	Full rights to the application's folders and files is required.
Action A:	Contact a system administrator to have full rights granted.
Cause B:	An internal processing and/or hardware error has occurred.
Action B:	Contact your Micromeritics service representative.

2488- File [*file name*] cannot be opened for editing. It is already in use.

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

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

Cause:	The specified file in a **Save As** operation is already open for edit.
Action:	Select a different file for the **Save As** operation.

2490- No '.INI' file present. Application will terminate.

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

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

Cause:	The highlighted fields contain invalid entries. The dialog box cannot be closed until all errors are corrected.
Action:	Check the entries, correct the errors and close the window.

2492- This field's entry is invalid.

Cause:	The highlighted field contains an invalid entry.
Action:	Check the entry and correct the error.

2493- An entry is required for this field.

Cause:	This field requires a valid entry to proceed.
Action:	Enter or select an appropriate value.

2494- Value is out of the valid range.
2495- Value is out of the valid range. Enter a value between [*value*] and [*value*].

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

2496- Invalid number.

Cause:	An invalid number entered in the highlighted field.
Action:	Check the entry and enter or select a valid number.

2497- This field contains an invalid character.

Cause:	An invalid character was entered in the highlighted field.
Action:	Check the entry and enter valid characters.

2498- The requested change to the Sample’s status is invalid at this time.

Cause:	A request to change the file’s status, for example, from *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.

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2500- All sample file names that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause:	No more sample information files can be created using the currently entered file name sequence number.
Action:	Select ***Options*** > ***Default Method*** and enter another sequence number.

2501- System resources have reached a dangerously low level. Please close some windows to avoid the loss of data.

Cause:	A large number of windows are open and consuming the system resources available to all applications.
Action:	Close one or more windows on the screen. Contact your Micromeritics service representative.

2505- Error logger cannot be initialized. Error code [*n*]. Program will exit.

Cause:	An internal processing error has occurred.
Action:	Contact your Micromeritics service representative.

2506- Sample file [*file name*] has a 'No Analysis' status and cannot be used for this operation

Cause:	The selected sample file does not have collected data and cannot be used for operations, for example, reporting.
Action:	Enter the name of a file with a status of Complete, Analyzing or Entered or select a sample file from the list box.

2507- The sample has an invalid status and cannot be used for degassing.

Cause:	A sample file has been selected which does not have a *No Analysis* or *Prepared* status.
Action:	Select a different file with a status of *No Analysis* or *Prepared*.

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

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

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

Cause:	An attempt to save calibration data has failed due to possible media problems.
Action A:	Ensure the destination location has no problems.
Action B:	Choose an alternate media to save the calibration data.

2515- Warning: Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?

Cause:	The process of performing a calibration operation was started.
Action:	Calibration operations should only be done by or under the direction of qualified service personnel.

2516- Warning: Keeping a backup copy of the calibration data is recommended by Micromeritics. Would you like to do so now?

Cause:	A calibration operation was performed and a backup copy is recommended.
Action:	Perform a calibration **Save** operation.

2517- Canceling this dialog will reset the calibration state to what it was when this dialog was first opened. Are you sure you want to cancel?

Cause:	The calibration has not been accepted.
Action:	If the calibration operation was successful, press **Accept**.

2520- No data points available for reporting.

Cause:	The selected sample file does not have collected data and cannot be used for reporting.
Action:	Select a different sample file.

2521- Unable to program controller.

Cause:	A hardware malfunction has occurred.
Action:	Contact your Micromeritics service representative.

2522- Invalid controller application file.

Cause:	The application’s control file has been corrupted or deleted.
Action:	Reinstall the analysis program.

2523- Programming the controller failed.
2524- CRC check failed on programming controller.
2525- Unknown error programming controller.
2526- Controller download was not successful.
2527- Controller CRC error on boot block.
2528- Controller DRAM error.
2529- Controller Com1: error.
2530- Controller Com2: error.
2531- Controller debug port error.

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

2532- The instrument contains a different software version. Do you want to reset it?

Cause:	The application has discovered a different version of software operating in the analyzer.
Action:	If there are no analyzers other than the one connected to the computer, select **Yes** and allow the updated software to load.

2533- Analyzer initialization failed.

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

2548- System status [*n*]

Cause:	There was a problem establishing communication with the analyzer.
Action:	Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2549- Error accessing online manual file [*code #*].

Cause:	The operator’s manual file could not be located.
Action A:	Reinstall the application.
Action B:	Copy the contents of the manual folder from the setup CD to the application directory.

2550- Attempts to acquire the instrument’s status timed out.

Cause:	There was a problem establishing communication with the analyzer.
Action:	Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2551- Unable to establish the TCP connection with the instrument.

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

2552- Configured serial number does not match instrument.

Cause:	An instrument was substituted without properly changing the instrument serial number.
Action:	Use the installation program to add or move devices as necessary.

2553- Dialog ID [*file name*] can not be created!.

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

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

Cause:	The sample file is currently being analyzed and is undergoing a critical operation.
Action:	Open the sample file after the critical operation has completed.

2557- Cannot access web page.

Cause:	The Micromeritics web page for DFT models cannot be accessed. This could be caused by an ISP problem of high internet traffic.
Action:	Try the operation later.

2558- The instrument is busy. The requested operation cannot be executed.

Cause:	The instrument is analyzing and cannot be interrupted.
Action:	Try the operation later.

2576- The instrument is *[sn]* is not calibrated.

Cause:	The analyzer application is in the process of initializing the instrument and is unable to locate the calibration files.
Action A:	Click **OK**. Select ***Unit [n] > Calibration > Load from File*** and select a file containing calibration data.
Action B:	Click **OK**. Close the application and use the Setup program to reinstall calibration files.

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4002- Thermal Transpiration correction had no effect.

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

4003- Error Converting Pressures.
4004- Error Computing Volume Adsorbed.

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

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

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

4011- Analysis gas in sample file does not match any gas in the unit.

Cause:	The analysis gas specified in the sample information file does not match the analysis gas entered in the Unit Configuration.
Action A:	If the wrong adsorptive was selected in the sample information file, change the adsorptive in the file.
Action B:	If necessary, attach the appropriate gas bottle then enter the gas in the Unit Configuration.

4012- Psat gas in sample file does not match any gas in the unit.

Cause:	If using *Measure psat of a gas* in P_o and T options in Analysis Conditions, the selected gas is not one of the selected gases in Unit Configuration.
Action A:	If the incorrect psat was selected, change the psat gas.
Action B:	If the gas was recently connected to the instrument, update the Unit Configuration.

4014- File [*name*] is not a valid file for conversion.

Cause:	The file selected for conversion is not a valid file.
Action:	Select only files that have been created by the proper program.

4015- Error creating export file for sample [*file name*].

Cause:	A file error occurred during creation of an export output file.
Action:	The output file name may be invalid. Ensure that the target directory exists and is not full or write protected. The target disk drive may be damaged or inoperative. Verify that other files may be created on the same drive. Contact your Micromeritics service representative.

4016- Sample [sample file name] has no data for export.

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

4017- Damage to the instrument will result if the sample has not been manually evacuated. Have you evacuated the sample?

Cause:	**Backfill sample at start of analysis** was not selected on the Sample Backfill Options window. The sample tube is normally at atmospheric pressure when an analysis is started and it must be backfilled before the analysis begins to prevent sample material from being drawn into the manifold.
Action:	If the sample tube has been manually evacuated, select **Yes**. If not, select **No** and then either perform a manual evacuation or go to the Sample Backfill Options window and select **Backfill sample at start of analysis**.

4020- Disabling this option may damage the instrument. Are you sure that the sample should not be backfilled?

Cause:	**Backfill sample at start of analysis** was not selected on the Sample Backfill Options window. The sample tube is normally at atmospheric pressure when an analysis is started; it must be backfilled before the analysis begins to prevent sample material from being drawn into the manifold.
Action:	To manually evacuate the sample prior to the start of the analysis, select **Yes**. Otherwise, select **No** and go to the Sample Backfill Options window and select **Backfill sample** at start of analysis.

4021- The entered Po value (Po and Temperature Options of the Analysis Conditions) is outside the range of the pressures listed in the Psat vs Temperature Table (Adsorptive Properties).

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

4022- The entered bath temperature value (Po and Temperature Options of the Analysis Conditions) is outside the range of the temperatures listed in the Psat vs Temperature Table (Adsorptive Properties).

Cause:	The entered bath temperature is outside of the range of temperatures specified in the Adsorptive Properties.
Action A:	Change the entered temperature.
Action B:	Change the adsorptive.
Action C:	Add more temperatures and corresponding pressures to Adsorptive Properties.

4023- The file [*file name*] cannot be prepared for analysis. It is open for editing and contains errors.

Cause:	An attempt was made to start an analysis using a file that contains errors and is open for editing.
Action:	Finish editing this file, save and close it then start the analysis.

4024- Backfill gas in sample file does not match any gas in the unit.

Cause:	The backfill gas specified in the sample information file does not match the analysis gas entered in the Unit Configuration.
Action A:	If the wrong backfill gas was selected in the sample information file, change the backfill gas in the file.
Action B:	If necessary, attach the appropriate gas bottle, then enter the gas in the Unit Configuration.

4026- Cannot calculate Dubinin-Astakhov: bad least squares data.

Cause:	Less than two selected data points are within the fitted pressure range.
Action:	Edit the selection of data points on the Dubinin interactive editor or on the Dubinin pressures window.

4027- Fewer than two sample files have data suitable for heat of adsorption reports.

Cause:	Less than two of the selected sample files for heat of adsorption reports contain appropriate data.
Action:	Edit the Quantity Adsorbed table or select other sample files.

4028- Dubinin calculations cannot be performed because the affinity coefficient of the analysis gas is zero.

Cause:	Dubinin values could not be calculated because the affinity coefficient of the analysis gas is zero.
Action:	Access the Dubinin Report Adsorptive options in the sample file and enter an appropriate value for the analysis gas.

4029- At least two fitted data points are needed for Alpha-S calculations.

Cause:	Fewer than two data points fall within the selected Alpha-s range.
Action:	Edit the calculation assignments or the fitted Alpha-s range or use a different reference curve.

4030- Preparations failed in primary data.

Cause:	Appropriate data were not available to generate the report.
Action:	This message was preceded by a different error message. Refer to the cause/action of the preceding message.

4031- Not enough points with a relative pressure in the range [*n,n*]

Cause:	Fewer than two data points selected for the Dubinin report fall within the selected relative pressure range.
Action:	Edit the calculation assignments or the fitted relative pressure range.

4033- Not enough points to generate Dubinin Tabular Report.

Cause:	There are fewer than two valid data points available for the Dubinin tabular reports.
Action:	At least two micropore pressures must be selected for inclusion in the Dubinin report. Edit the selection of data points on the Dubinin interactive editor or on the Dubinin pressures window.

4034- Fewer than 2 points available for Dubinin calculations.

Cause:	There are fewer than two valid data points available for Dubinin reports in one of the sample files selected for overlaying.
Action:	At least two micropore pressures must be selected for inclusion in the Dubinin report. Edit the selection of data points on the Dubinin interactive editor or on the Dubinin pressures window.

4035- Cannot calculate optimized Astakhov exponent: Not enough points with a relative pressure in the range [(*pressure*), (*pressure*)].

Cause:	There are fewer than two valid data points in the relative pressure range specified. Astakhov reports will not be produced.
Action:	At least two pressures must be selected for inclusion in the Astakhov report. Edit the selection of data points on the Astakhov interactive editor or on the Astakhov pressures window.

4036- Fewer than 2 points available for Horvath-Kawazoe calculations.

Cause:	At least two data points must be selected for inclusion in the Horvath-Kawazoe analysis. No report will be produced.
Action:	Edit the selection of points on the Horvath-Kawazoe interactive editor or on the Horvath-Kawazoe window.

4037- Computations failed while processing the primary data set. No reports will be produced.

Cause:	The preparation of data for reporting could not be successfully completed. No Horvath-Kawazoe reports will be produced. This message will always be preceded with another one containing additional information.
Action:	Refer to the number of the error message which preceded this one for an explanation.

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

Cause:	The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm must include at least two points above 0.02 relative pressure for the Langmuir equation to be applied.
Action:	The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the Horvath-Kawazoe Report Options window to prevent this message from appearing on future reports.

4039- The isotherm does not meet the constraints of the Cheng/Yang assumption. Cheng/Yang correction will not be applied.

Cause:	The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The isotherm does not correlate to the Langmuir assumption with a coefficient of 0.98 or more. The correction is not applicable to this isotherm or to the range of the data points selected.
Action A:	The analysis will be performed without the Cheng/Yang correction. Deselect Apply Cheng/Yang correction on the Horvath-Kawazoe Report Options window to prevent this message from appearing on future reports.
Action B:	Generate the Langmuir report for the same data points selected for the Horvath-Kawazoe report. If the Langmuir correlation coefficient can be brought above 0.98 by removing some points at high relative pressure, remove them and reproduce the Horvath-Kawazoe reports.

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

Cause:	The Cheng/Yang correction to the Horvath-Kawazoe equation requires the value of the monolayer volume as calculated from the Langmuir equation. The computed value is less than the volume adsorbed at the largest relative pressure included in the analysis. The correction is not applicable to this isotherm or to the range of the data points selected.
Action:	The analysis will be performed and the Cheng/Yang correction will be applied to all points with a volume adsorbed less than the value of 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 P/Po.

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 assignments are not being used and more than one of the report pressure table points is above the range of the collected data and more than one is below.
Action:	Change the report pressure table to be more consistent with the collected data.

4045- Points in the report pressure table lie outside the collected data.

Cause:	Calculation assignments are not being used and more than one of the report pressure table points is above the range of the collected data and more than one is below.
Action:	Change the report pressure table to be more consistent with the collected data.

4046- [*file name*] could not be opened for reading.

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

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

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

4048- Warning: An error occurred while restoring the heat of adsorption report editor.

Cause:	The state of the heat of adsorption report editor could not be restored. Default settings will be used.
Action:	No action.

4049- The sample [*file name*] does not have enough data. A minimum of two adsorption points is required.

Cause:	A sample file has been included in the Heat of Adsorption report that does not have enough data.
Action:	Remove the file from the selected file list.

4050- None of the requested quantities adsorbed is within the range of the collected data of more than one sample file.

Cause:	The Heat of Adsorption report failed because the specified quantities are not within the range of the collected data.
Action:	Edit the quantities adsorbed so that they are within the range of the collected data or select other sample files.

4051- The sample [*file name*] does not have any data in the range of the requested quantities adsorbed.

Cause:	The sample's data cannot be interpolated to any of the quantities adsorbed.
Action:	Edit the quantities adsorbed so that they are within the range of the collected data.

4052- Fewer than two points are selected for this report.

Cause:	At least two points are required for the BET calculations.
Action:	Edit the calculation assignments for the BET report.

4053- At least two data points must be selected for t-Plot calculations.

Cause:	At least two points are required for the t-Plot calculations.
Action:	Edit the calculation assignments for the t-Plot report.

4054- Fewer than two data points are inside the fitted thickness range.

Cause:	At least two points must be within the fitted thickness range for the t-Plot calculations.
Action A:	Edit the calculation assignments for the t-Plot report.
Action B:	Edit the fitted thickness range in the t-Plot report editor.

4055- A positive BET surface area was not calculated.

Cause:	Fewer than two points were assigned to the requested surface area calculation in the collected data table.
Action A:	Assign more points to the surface area calculation.
Action B:	Select a different surface area in the t-Plot report editor.

4056- A positive Langmuir surface area was not calculated. Please check your Langmuir report.

Cause:	Fewer than two points were assigned to the requested surface area calculation in the collected data table.
Action A:	Assign more points to the surface area calculation.
Action B:	Select a different surface area in the t-Plot report editor.

4057- At least two data points are needed for Freundlich calculations.

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

4058- At least two data points are needed for Temkin calculations.

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

4059- Fewer than 2 points available for MP-Method calculations.

Cause:	At least two points are required for the MP-Method calculations.
Action:	Edit the calculation assignments for the MP-Method report.

4060- Sample [*file name*] contains no data points.

Cause:	An attempt was made to save a sample without collected data as a t-curve or alpha-S curve.
Action:	Repeat the Save As t-curve or Save As alpha-S operation after opening a sample that has collected data.

4061- The t-curve must contain at least 2 points.

Cause:	At least two points are required in a thickness curve definition.
Action:	Edit the thickness curve.

4062- Error during report preparation.

Cause:	An internal processing error has occurred.
Action:	Contact your Micromeritics service representative.

4063- The data requested on this report are not available. No subreports selected.

Cause:	There is no information in the sample log to report.
Action:	A sample file was selected which no instrument operations has been used. Select a sample file with a status of *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 [*name*].

Cause:	For some reason, the list of available models was corrupted, therefore, the model selected could not be loaded for the deconvolution.
Action:	Exit the program and reinstall the software, then try again.

4071- The selected pressures points do not form a valid set for deconvolution.

Cause:	The data points selected for analysis do not contain enough information to allow a DFT data reduction.
Action:	At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction. Edit the selection of data points on the DFT interactive editor or on the DFT pressures window.

4072- The range of pressures selected is too small to deconvolute using this model.

Cause:	A null result was found using the selected model.
Action:	At least two points with strictly increasing pressures and volumes adsorbed are required for a DFT Plus data reduction. Edit the selection of data points on the DFT interactive editor or on the DFT pressures window.

4073- The analysis gas [*name*] does not match the model gas [*name*].

Cause:	The model assumes a specific gas and the sample file uses a different one.
Action:	Select a model that assumes the same gas.

4074- The analysis temperature [*nn*] does not match the model temperature [*nn*].

Cause:	The temperature for the selected model did not match the analysis temperature.
Action:	Select a different model.

4075- The models cannot be located in the models folder. Reinstall the software.

Cause:	The models could not be located. They may have been inadvertently deleted or moved.
Action:	Reinstall the software.

4077- Cannot get surface area for: [*file name*]

Cause:	The Isotherm report for the named overlay file has **Per gram** selected for the **Volume Adsorbed** and the Isotherm report for the primary file has a surface area option selected for the **Volume Adsorbed**.
Action A:	Edit the Isotherm report for the named overlay file and select a surface area option for **Volume Adsorbed**.
Action B:	Click **Overlays** on the Report options window of the primary file and remove the named overlay file from the list.

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

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

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

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

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

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

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

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

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

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

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

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

6000 Series

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

Cause:	An attempt to load a previously saved calibration file was unsuccessful.
Action:	Ensure the file exists and the file name is entered correctly. Then try again.

6004- Unable to write the calibration file [*number*].

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

6012- Cannot read the analysis conditions parameter file.
6013- Cannot read the adsorptive properties parameter file.
6014- Cannot read the report options parameter file.

Cause:	The parameter file is either corrupt or has been deleted.
Action A:	If this is a file created in your lab, recreate the file.
Action B:	If this is a default file created during application installation, re-install the software.

6015- Cannot read the sample tube properties parameter file.

Cause:	The selected sample tube file on the QuickStart screen cannot be read.
Action:	Select a different file.

6016- Dosing manifold from valve [*number*] failed.

Cause A:	The maximum time was exceeded before the target pressure point was reached. The nitrogen regulator may be set too low or turned off.
Action A:	Set the analysis gas regulator to at least 10 psig (0.7 bar), then resume the analysis.
Cause B:	The analysis gas bottle is empty.
Action B:	Connect a new analysis gas bottle then resume the analysis.

6017- Leak test failed on port [*number*].

Cause:	With the sample port valve closed, the sample pressure increased by 0.15 mmHg before the leak test duration was completed.
Action:	Check sample tube fitting and ensure that it is securely attached to the port then restart the analysis.

6018- Volume dosed exceeded 1000 cm^3 STP. Analysis is canceled.

Cause:	There is a problem with the analyzer's calibration.
Action:	Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6019- Elevator failed to reach upper/lower limit switch.

Cause A:	There is an obstruction in the elevator path.
Action A:	Clear all obstructions and restart the analysis.
Cause B:	Ice is present in the bottom or the neck of the Dewar preventing the elevator from rising completely.
Action B:	Check the Dewar, remove ice and restart the analysis.
Action B:	If results for Actions A and B failed, contact a Micromeritics service representative.

6020- Warning, servo valve performance is out of specification.

Cause:	The servo valve tried to dose to a pressure but was unable to reach it within specification. The analysis will continue.
Action:	At the next appropriate time, calibrate the servo valve to bring it back within specification. Refer to **Servo Valve**, page **4-32**.

6021- Servo calibration failed.

Cause A:	The maximum time was exceeded before the target pressure point was reached. The nitrogen regulator may be set too low or turned off.
Action A:	Set the analysis gas regulator to at least 10 psig (0.7 bar), then resume the analysis.
Cause B:	The analysis gas bottle is empty.
Action B:	Connect a new analysis gas bottle, then resume the analysis.

6024- Evacuation failed.

Cause:	While attempting to zero the pressure transducers, the instrument was unable to evacuate to a pressure of less than 1 mmHg. This may be due to a leak or a bad calibration.
Action A:	Check the sample tube fitting and ensure that it is securely attached to the port.
Action B:	Use the Setup program to reinstall the calibration files.

6025- Target pressure [*PRn*] [*PR-U*] exceeded maximum manifold pressure of [*PRn*] [*PR-U*]. Analysis is canceled.

Cause:	An absolute pressure greater than (pressure) units was attained that exceeded the specified maximum manifold pressure.
Action:	The analysis was canceled. All previously collected data were stored. Change the maximum manifold pressure value in the Adsorptive Properties file.

6026- Psat gas is not condensing.

Cause A:	The working Dewar does not contain enough bath liquid.
Action A:	Refill the Dewar and try the operation again.
Cause B:	The Psat gas is contaminated.
Action B:	Replace the Psat gas supply.
Cause C:	The Psat tubing from the regulator to the instrument is contaminated.
Action C:	Pump out the tubing.

6027- There is no nitrogen attached to the unit.

Cause:	A calibration requiring nitrogen was attempted but the software does not recognize that nitrogen is attached.
Action:	Ensure that a nitrogen gas bottle is installed at one of the analysis ports, then Select ***Unit*** **>** ***Unit configuration*** and enter **N2** for the appropriate valve.

6029- The Po in the sample file does not match any gas in the unit.

Cause:	An attempt was made to start an analysis with a gas that is not connected to the instrument or has not been designated in the software.
Action A:	Ensure the gas is connected to the instrument. Then select ***Unit [n]* > *Unit configuration*** to tell the application that the gas is connected.

6030- Dosing method choice is invalid. The Krypton analysis requires that Adsorptive Properties “Dosing Method” is set to “From Psat tube.”

Cause:	A file was selected for a krypton analysis that has **Normal** selected for the **Dosing Method**. **Normal** is for standard analyses only.
Action:	Open the sample file and change the **Dosing Method** to **From Psat tube** or select a different file for the analysis.

6031- Dosing method choice is invalid. The analysis requires that Adsorptive Properties “Dosing Method” is set to “Normal.”

Cause:	A file was selected for a standard analysis that has **From Psat tube** selected for the **Dosing Method**. **From Psat tube** is for krypton analyses only.
Action:	Open the sample file and change the **Dosing Method** to **Normal** or select a different file for the analysis.

6032- Template file [*file name*] for the selected analysis type does not exist. Select another analysis type.

Cause:	A program piece required to run the PCP analysis is missing. Applies when in Service Test Mode.
Action:	Re-install the software.

6033- Krypton gas is not condensing in the Psat tube ([*PR1*] [*PR-U*]).

Cause A:	The krypton gas may be contaminated.
Action A:	Evacuate the krypton gas inlet line.
Action A:	Manually verify the saturation pressure of the krypton gas: 1. Evacuate the psat tube. 2. Backfill with krypton gas to 20 mmHg. 3. Raise the Dewar. These steps should condense the krypton gas to a pressure below 3 mmHg.
Cause B:	The Dewar does not contain enough cryogen.
Action B:	Refill the Dewar.

6034- Zeroing of a transducer failed. Analysis Canceled.

Cause:	The transducer did not respond correctly.
Action:	Contact your Micromeritics service representative.

6035- Purification of krypton in the Psat tube failed.

Cause A:	Krypton pressures in the psat tube did not stabilize after the purification steps.
Action A:	Evacuate the krypton gas inlet line.
Action A:	Manually verify the saturation pressure of the krypton gas: 1. Evacuate the psat tube. 2. Backfill with krypton gas to 20 mmHg. 3. Raise the Dewar. These steps should condense the krypton gas to a pressure below 3 mmHg.
Cause: B:	The Dewar does not contain enough cryogen.
Action B:	Refill the Dewar.

6040- Failed to reach pressure dosing through servo. Calibration canceled.

Cause A:	There was insufficient gas pressure to calibrate for matching transducers. The gas is not connected or the tank is almost empty.
Action A:	Ensure the gas is connected properly and assigned correctly in the Unit Configuration window. Replace the tank if necessary.
Cause B:	The gas valve is not working properly.
Action B:	Contact your Micromeritics service representative.

6041- Servo Calibration failed.
6042- Master pressure transducer calibration failed. Offset is out of range.
6043- Port 1 pressure transducer calibration failed. Offset is out of range.
6044- Port 2 pressure transducer calibration failed. Offset is out of range.
6045- Port 3 pressure transducer calibration failed. Offset is out of range.
6046- Po pressure transducer calibration failed. Offset is out of range.
6047- 10 torr pressure transducer calibration failed. Offset is out of range.
6048- Master pressure transducer calibration failed. Scale is out of range.
6049- Port 1 pressure transducer calibration failed. Scale is out of range.
6050- Port 2 pressure transducer calibration failed. Scale is out of range.
6051- Port 3 pressure transducer calibration failed. Scale is out of range.
6052- Po pressure transducer calibration failed. Scale is out of range.

6053- 10 torr pressure transducer failed. Scale is out of range.

Cause:	There is a problem with the analyzer's calibration.
Action:	Use the Setup program to reinstall the calibration files. Contact your Micromeritics service representative.

6054- When dosing from the Psat tube, Po must be entered or calculated..

Cause:	The adsorptive properties for the adsorptive used in a sample specify **From Psat tube** as the dosing method. This is dosing method is only compatible with an entered or calculated Po.
Action:	If the dosing method is incorrect, change the dosing method for the sample in the Adsorptive Properties to **Normal**. If the dosing method is correct, change the Po option in the Po and Temperature Options to **Enter Po** or **Calculate Po**.

6055- Krypton analysis cannot be performed because the instrument is not a Krypton-enabled system.

Cause:	Krypton analysis was selected but the required 10 torr transducer was not detected.
Action:	Contact your Micromeritics service representative.

6056- The Psat gas must be the same as the analysis gas if Po is calculated from Psat and the analysis gas is dosed from the Psat tube.

Cause:	Dosing from the Psat tube is selected in the adsorptive properties and the analysis conditions Po and temperature options specify that Po should be calculated from the Psat of a gas, but the Psat gas differs from the analysis gas.
Action:	Select a gas for the Psat measurement that has the same mnemonic as the analysis gas. Typically the analysis and Psat gas will be krypton but the Psat gas will include the solid Psat vs. T table.

6557- File *[file name]* already selected for the analysis

Cause:	The same sample file is already assigned to a different port for this analysis.
Action:	Select a different sample file.

6558- Gas [*n*] in sample file [*file name*] does not match any gas in the unit.

Cause:	The analysis gas specified in the sample information file does not match the analysis gas entered in the Unit Configuration.
Action A:	If the wrong adsorptive was selected in the sample information file, change the adsorptive in the file.

10000 Series

10050- Pirani offset calibration is invalid

Cause:	There is a problem with the Pirani offset calibration.
Action:	Contact your Micromeritics service representative.

10070- Cold cathode offset calibration is invalid.

Cause:	There is a problem with the cold cathode offset calibration.
Action:	Contact your Micromeritics service representative.

10080- Cold cathode scale calibration is invalid.

Cause:	There is a problem with the cold cathode scale calibration.
Action:	Contact your Micromeritics service representative.

10100- Vacuum gauge (Pirani) error detected.

Cause:	There is a problem with the vacuum gauge (Pirani).
Action:	Contact your Micromeritics service representative.

10110- Vacuum gauge (cold cathode) error detected.

Cause:	There is a problem with the vacuum gauge (cold cathode).
Action:	Contact your Micromeritics service representative.

10120- Vacuum gauge communications error.

Cause:	There is problem with communication between the instrument and the vacuum gauge.
Action:	Contact your Micromeritics service representative.

10180- *[XDCR]* Transducer offset calibration rejected (current: *[PR4] [PR-U]*, new: *[PR4]*, nominal: *[PR4]*, max: *[PR4]*).

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

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

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

10200- Transducer underrange detected.

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

10210- Transducer overrange detected.

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

10240- Temperature offset calibration rejected.

Cause:	There is a problem with the temperature offset calibration.
Action:	Contact your Micromeritics service representative.

10250- Temperature scale calibration rejected.

Cause:	There is a problem with the temperature scale calibration.
Action:	Contact your Micromeritics service representative.

10270- Invalid servo calibration error.

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

10280- Servo DAC timeout detected.

Cause:	There is a problem with the servo DAC timing out.
Action:	Contact your Micromeritics service representative.

10300- Turbo pump failure detected.

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

10310- Turbo pump temperature error detected.

Cause:	There is a problem with the turbo pump temperature.
Action:	Contact your Micromeritics service representative.

10320- Turbo pump communications error detected.

Cause:	There is a problem with the turbo pump communication.
Action:	Contact your Micromeritics service representative.

10360- Time limit exceeded while waiting for the elevator to rise into position (elapsed: *[0]* s, max allowed: *[0]* s, status: *[HEX]*, alarm code: *[HEX]*, inputs: *[HEX]*, position: *[0])*.

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

10370- Time limit exceeded while waiting for the elevator to lower into position (elapsed: *[0]* s, max allowed: *[0]* s, status: *[HEX]*, alarm code: *[HEX]*, inputs: *[HEX]*, position: *[0]*).

Cause A:	There is a problem with the elevator.
Action A:	Check the Dewar and remove ice if necessary. Then restart the analysis. Contact your Micromeritics service representative if necessary.
Cause B:	The Psat tube is interfering with elevator movement.
Action B:	Ensure the Psat tube is close to the sample tube and the Dewar lid is over both the sample and Psat tubes. Then restart the analysis. Contact your Micromeritics service representative if necessary.

10380- Elevator error detected (code: *[HEX]*).

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

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

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

10410- Elevator communications error detected.

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

10420- *[PORT]* over-pressure detected (pressure: [*PR4] [PR-U]*, max allowed: *[PR4]*).

Cause A:	A pressure greater than 1000 mmHg was detected in the instrument. The instrument has automatically canceled any operations in progress and taken action to relieve the pressure.
Action A:	If the instrument was being operated manually, review recent activity to identify the cause of the overpressure and avoid a repetition. If problem repeats, contact your Micromeritics service representative.
Cause B:	If the error occurred when the Dewar was lowered, excessive adsorption of condensation of gas may have occurred during analysis and returned to gas phase too rapidly when the Dewar was lowered.
Action B:	Revise the analysis conditions or sample quantity to prevent recurrence. If problem repeats, contact your Micromeritics service representative.
Cause C:	If the error occurred during dosing from a gas inlet, the gas inlet pressure might be excessive.
Action C:	Adjust the gas inlet pressure to recommended range. If problem repeats, contact your Micromeritics service representative.

10480- Operation cancelled by operator.

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

10490- Operation cancelled by instrument.

Cause:	The instrument canceled the operation.
Action:	An accompanying message will display explaining why the operation was canceled. Correct the indicated problem and restart the operation.

10500- Operation skipped by operator.

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

10510- Operation suspended by operator.

Cause:	The operator suspended the operation.
Action:	None. If you are on the analysis window, click the **Play** button to resume the operation.

10520- Operation suspended by instrument.

Cause:	The instrument suspended the operation.
Action:	An accompanying message will display explaining why the operation was suspended. Correct the indicated problem. If you are on the analysis window, click the **Play** button to resume the operation.

10530- Operation resumed by operator.

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

10560- Instrument communications error detected.

Cause:	There was a fatal error in communication between the application and the software in the instrument. All displays for that instrument will be closed.
Action:	Ensure that the analyzer is connected to the computer on the communications port configured in the Setup program. Stop and restart the analyzer software. Contact your Micromeritics service representative.

10710- Manifold temperature error detected (manifold: *[DegC1] [DegC-U]*, port: *[DegC1] [DegC-U]*, heater: *[DegC1] [DegC-U]*, heater target: *[DegC1] [DegC-U]*, power: *[2]*).

Cause:	An error was detected during manifold temperature control.
Action:	Ensure the cover to the upper cabinet of the instrument is securely fastened. If the source of the problem has been identified and corrected, close the application program, cycle power to the instrument off for a few seconds and then turn the power ON. Restart the application program. If the problem repeats or is not resolved, contact your Micromeritics service representative or local fire department.

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

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

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

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

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

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

10750- Time limit exceeded during evacuation (target: *[PR4] [PR-U],* pressure: *[PR4] [PR-U]*, elapsed: *[0]* s).

Cause:	The maximum time for evacuating the sample was exceeded. Possible causes are a leak in the sample tube fitting, a crack in the sample tube, or a poorly degassed sample.
Action:	Check the sample tube and the sample tube fitting; ensure that the tube is securely attached to the port. Verify that the sample is properly degassed; then restart the analysis.

10760- Time limit exceeded while dosing (gas: [*GAS]*, valve: *[0]*, target: *[PR4] [PR-U],* pressure: *[PR4] [PR-U]*, elapsed: *[0]* s).

Cause A:	The specified pressure was not attained. The gas regulator may be set too low or turned off.
Action A:	Set the gas regulator to 10 psig (0.7 bar). Then restart the analysis.
Cause B:	The gas bottle is empty.
Action B:	Connect a new gas bottle. Then restart the analysis.

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

Cause:	There was a problem dosing the sample to target pressure. The instrument was unable to dose the required quantity of gas after several attempts.
Action:	Check that the outlet stage of the gas regulator is within specification. Review the analysis parameters.

10780- Leak test failed (sample port: *[0]*, interval: *[0]* s, leak rate: *[PR4] [PR-U]*/min, max allowed: *[PR4] [PR-U]*/min).

Cause:	With the sample port valve closed, the sample pressure increased by 0.15 mmHg before the leak test duration was completed.
Action:	Check sample tube fitting and ensure that it is securely attached to the port. Then restart the analysis.

10790- Quantity dosed on sample port *[0]* (*[Q2] [Q-U]*) has exceeded the maximum of *[Q2] [Q-U]* (qty dosed this point: *[Q2] [Q-U]*, pressure: *[PR4] [PR-U]*, *[P0Sym]*: *[PR4] [PR-U]*, gas: *[GAS]*).

Cause:	An excessive quantity of gas has been dosed into the sample port due to an excessive quantity of a sample with high pore volume, condensation of gas due to a lower than expected saturation pressure, or a leak.
Action:	Review the analysis conditions, the sample quantity, and the sample tube connection to identify and correct the problem before repeating the analysis.

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

Cause:	Attempts to condense the adsorptive gas in the sample tube have failed due to an excessive quantity of a sample with high pore volume, adsorptive gas contamination, a higher than expected saturation pressure, or a leak.
Action:	Review the analysis conditions, the sample quantity, the gas supply, and the sample tube connection to identify and correct the problem before repeating the analysis.

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

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

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

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

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

Cause:	A target pressure was requested that exceeds the maximum allowed. The maximum pressure may be based on saturation pressure of the gas at the temperature of the gas source, the manifold, the sample, or ambient temperature.
Action:	Review the maximum allowable pressures in the error message and the analysis conditions to identify and correct the problem before repeating the analysis.

10860- Psat gas *[GAS]* is not condensing. (pressure: *[PR4] [PR-U]* maximum manifold pressure: *[PR4] [PR-U]*).

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

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

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

10880- Zeroing of the transducers failed.

Cause:	The transducers failed to zero out.
Action:	Contact your Micromeritics service representative.

10890- Purification of the adsorptive *[GAS]* failed at *[PR4] [PR-U]* (charge pressure: *[PR4] [PR-U]*, minimum allowed: *[PR4] [PR-U]*).

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

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

Cause:	A target pressure for desorption was requested that is below the minimum desorption pressure allowed.
Action:	Review the analysis conditions to remove the disallowed pressure.

10950- Power supply voltage error detected (type: *[0]*, voltage: *[2]* volts, nominal: *[1]* volts).

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

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

Cause:	An error was detected with the manifold heater temperature.
Action:	Check the top cover of the instrument. Ensure it is installed and sealed properly.

C. CALCULATIONS

This appendix contains the calculations used in the analysis program.

Saturation Pressure

Saturation pressure (Po) is selected on the Po and Temperature Options window. It may be entered or measured in the Po tube.

- If entered, Po = user-entered value.
- If measured, Po = equilibrated pressure reading after saturating Po tube or the sample tube. Po is measured for each data point.

Relative Pressure Calculations

If Po was entered, use the entered Po:

$$Po_i = Po_E$$

Calculate relative pressure for the ith data point:

$$P_{rel_1} = \frac{P_i}{Poi}$$

where

Po_I = saturation pressure for the ith data point
Po_1 = most previous measured saturation pressure before ith data point
Po_2 = first measured saturation pressure after the ith data point
Po_C = calculated Po
Po_E = entered saturation pressure
t_I = time when the ith data point was taken (minutes)
to_1 = time when Po_1 was measured (minutes)
to_2 = time when Po_2 was measured (minutes)
P_{rel_I} = relative pressure for the ith data point
P_I = absolute pressure for the ith data point, taken at equilibrium

Quantity Adsorbed Calculations

Free Space

Measured

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

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

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

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

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: used in non-ideality correction (cm^3 at standard temperature)
V_{fc} = volume of free space, cold (cm^3 at standard temperature)
V_{fw} = volume of free space, warm (cm^3 at standard temperature)
V_{man} = manifold volume (cm^3)

Calculated

The calculated free space is determined by subtracting the gas capacity of the volume occupied by the sample from the measured free space of the empty tube.

$$V_{fw} = V_{wb} + \Delta V - V_s \times \frac{T_{STD}}{T_{amb}}$$

$$V_{fc} = V_{cb} + \Delta V - V_s \times \frac{T_{STD}}{T_{bath}}$$

where

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

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 equations below are those used to compute weighted average and first derivative, respectively, for the 6th point of an 11-point window.

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

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

$$P_{\text{pcp, i}} = 100\% \frac{P_{\text{chg}}}{P_{\text{avg}}}$$

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_{i} = i^{th} pressure reading taken at equilibrium intervals

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.

Thermal Transpiration Correction

During data reduction, thermal transpiration correction is applied to the data if the user selected **Thermal transpiration correction** from the Report Options dialog. Starting with the first collected pressure, the following calculations are performed until the pressure ratio (PC/P) is greater than or equal to 0.99.

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

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

$$F = \frac{1}{\alpha Y^2 + \beta Y + \mu}$$

$$P = \left(1 - F\left(1 - \sqrt{\frac{T_{\text{bath}}}{T_{\text{amb}}}}\right)\right)$$

where:

α = Weber's coefficient, 0.033
β = Weber's coefficient, 0.245
F, Y, μ = intermediate values for subsequent calculations
G = Weber's coefficient, 2.5
H = Weber's coefficient, 2
MD = thermal transpiration hard sphere diameter of gas (Å), from the **Adsorptive Properties** window
P = equilibrated collected pressure measured by gauge at temp T_{amb}
SD = inside diameter of sample tube (mm), from the **Report Options** window
T = average temperature $\frac{T_{\text{bath}} + T}{2}$
T_{amb} = room temperature (298 K)
T_{bath} = analysis bath temperature (K), from the ***Po* and Temperature Options** window

BET Surface Area

For each point designated for surface area calculations, the BET[2] transformation is calculated as:

$$\frac{1}{N_{ads}\left(\frac{P_o}{P} - 1\right)}$$

A least-squares fit is performed on the (P_{rel}, B) designated pairs where P_{rel} is the independent variable and B is the dependent variable. The following are calculated:

a.) Slope (S g/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

BET Surface Area (m^2/g):

$$SA_{\mathrm{bet}} = \frac{CSA \times N_A}{(22414\ \mathrm{cm}^3\ STP)\left(\frac{10^{18}\ \mathrm{nm}^2}{\mathrm{m}^2}\right)(S + Y_{\mathrm{int}})}$$

where

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

N_A = Avogadro constant ($6.02214129 \times 10^{23}$ mol^{-1})

BET C value:

$$C = \frac{S + Y_{\mathrm{int}}}{Y_{\mathrm{int}}}$$

Quality of the Monolayer (cm^3/g STP):

$$Q_{\mathrm{m}} = \frac{1}{CY_{\mathrm{int}}} = \frac{1}{S + Y_{\mathrm{int}}}$$

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

$$BET_{\text{err}} = \frac{SA_{\text{bet}}(S_{\text{err}}{}^2 + YI_{\text{err}}{}^2)^{0.5}}{Y_{\text{int}} + S}$$

Langmuir Surface Area

For each point designated for surface area calculations, the Langmuir[3] transformation is calculated as:

$$L = \frac{P_{\text{rel}}}{N_{\text{ads}}}$$

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):

$$SA_{\text{Lan}} = \frac{CSA \times N_A}{(22414\ \text{cm}^3\ STP)\left(\frac{10^{18}\ \text{nm}^2}{\text{m}^2}\right)S}$$

where

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

N_A = Avogadro constant (6.02214129 × 10^{23} mol^{-1})

Quantity of the Monolayer (cm^3/g STP):

$$Q_{\mathrm{m}} = \frac{1}{S}$$

Langmuir b Value:

$$b = Y_{\mathrm{int}}\ V_{\mathrm{m}}$$

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

$$LAN_{\mathrm{err}} = \frac{SA_{\mathrm{Lan}}S_{\mathrm{err}}}{S}$$

Freundlich Isotherm

The Freundlich isotherm has the form

$$\frac{Q}{Q_{\mathrm{m}}} = CP^{\frac{1}{\mathrm{m}}}$$

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,

$$Q = Q_{\mathrm{m}}CP^{\frac{1}{\mathrm{m}}}$$

Taking the log of both sides yields

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

Temkin Isotherm

The Temkin isotherm has the form

$$\frac{Q}{Q_{\mathrm{m}}} = \frac{RT}{q_0 \alpha} \ln(A_0 P)$$

where

A = $\mathrm{a}_0 \exp\left\{\frac{-q_0}{RT}\right\}$, 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 $8.31441 \times 10^{-3} \frac{kJ}{\mathrm{mol}K}$

T = bath temperature (K)

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 $\frac{RTQ_{\mathrm{m}}}{q_0}$ and intercept $\ln A \frac{RTQ_{\mathrm{m}}}{q_0 a}$.

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):

$$\frac{SV_{mol}}{F \times 22414\text{cm}^3 STP} \times 10^4$$

where

10^4 = unit conversions
F = surface area correction factor, user-entered on the **t-Plot Report Options** window
V_{mol} = liquid molar volume, from the fluid property information

Micropore Surface Area (m^2/g):

$$SA_{\mu p} = SA_{total} + SA_{ext}$$

where SA_{total} is the BET surface area if the user enabled the BET report exclusively, or Langmuir surface area if the user enabled the Langmuir report exclusively. If neither report has been selected, SA_{total} is the BET surface area value calculated using a set of default parameters.

Micropore Volume (cm^3 liquid/g):

$$\frac{Y_{int}V_{mol}}{22414\text{cm}^3 STP}$$

Alpha-S Method

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

$$a_i = \frac{Q_i}{Q_{0.4}}$$

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

A least-squares analysis fit is performed on the (α_i,$Q_{ads,i}$) data pairs where α_i is the independent variable and $Q_{ads,i}$ is the dependent variable. The following are calculated:

a.) Slope (S cm³/g STP)

b.) Y-intercept (Q_0 cm³/g STP)

c.) Error of the slope (cm³/g STP)

d.) Error of the Y-intercept (cm³/g STP)

e.) Correlation coefficient

Surface area is calculated as:

$$A = \frac{A_{ref} S}{Q_{0.4}}$$

Pore size is calculated as:

$$\frac{Q_0 V_{mol}}{22414 \mathrm{cm}^3 \mathrm{STP}}$$

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

f-Ratio Method

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

$$f_{\mathrm{i}} = \frac{Q_{\mathrm{i}}}{Q_{\mathrm{ref}}\,P_{\mathrm{i}}}$$

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

BJH Pore Volume and Area Distribution

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

The data used in these calculations must be in order of strictly decreasing numerical value. Points which do not meet this criterion are omitted. The remaining data set is composed of relative pressure (P), quantity adsorbed (Q) pairs from (P_1, Q_1) to (P, 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[7] 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[8] developed the method (known as the BJH method) which incorporates these ideas. The algorithm used is an implementation of the BJH method.

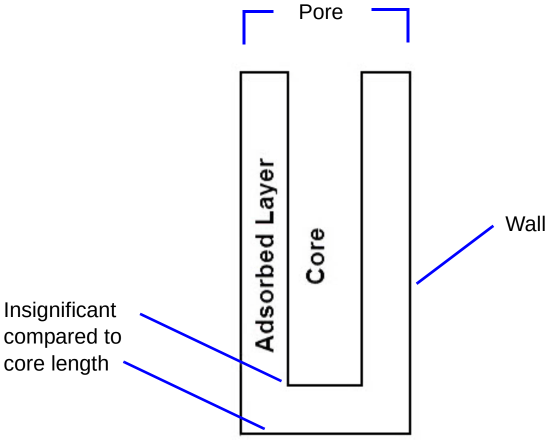
Explanation of Terms

A pore filled with condensed liquid nitrogen has three zones:

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

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

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



Calculations

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

$$Vl_{\mathrm{i}} = \frac{Q_i V_{\mathrm{mol}}}{22414\mathrm{cm}^3\ \mathrm{STP}}$$

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:

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

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 Calculations**, page **C-42**.

For the last pressure interval from the lowest Pr_i to zero relative pressure, reference the calculations from the equations in **Thickness Curve Calculations**, page **C-42**.

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

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

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

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

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

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

$$Vd_i = \sum_j (LP_j)(CSAa_j) \qquad \text{for all previously opened pores}$$

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

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

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

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

where

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

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

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

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 $(Vl_i - Vl_{1+1})$, 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:

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

$D_{\mathrm{avg,k}}$ 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 $Rc_{\mathrm{k+1}}$, if its length was the mean of the lengths at those radii.

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

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

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

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

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

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

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

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

The length of the newly opened pores is calculated as:

$$LP_{\mathrm{k}} = \frac{Vc_{\mathrm{i}}}{CSAc_{\mathrm{k}}}$$

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

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

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

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

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

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

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

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:

$$Dp_{\text{j}} = 2(Rc_{\text{j}})$$

for all Rc_j including Rc_{k+1}.

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

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

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

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

$$VP_{\text{cum, i}} = \sum_{j} Vp_{\text{j}} \text{ for } (J \le 1)$$

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

$$SA_{\mathrm{i}} = \pi(LP_{\mathrm{i}})\left(\frac{10^{-2}\mathrm{m}}{\mathrm{cm}}\right)(D_{\mathrm{avg,\,i}})\left(\frac{10^{-10}\mathrm{m}}{\mathrm{Å}}\right)$$

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

$$SA_{\mathrm{cum,\,10}} = \sum SA_{\mathrm{j}}\ for\ J \leq 1$$

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

$$\frac{dV}{dD_{\mathrm{i}}} = \frac{VP_{\mathrm{i}}}{Dp_{\mathrm{i}} - Dp_{\mathrm{i+1}}}$$

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

$$\frac{dDv}{d\log D_{\mathrm{i}}} = \frac{VP_{\mathrm{i}}}{\log\left(\frac{Dp_{\mathrm{i}}}{Dp_{\mathrm{i+1}}}\right)}$$

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

$$\frac{dA}{dD_{\mathrm{i}}} = \frac{SA_{\mathrm{i}}}{Dp_{\mathrm{i}} - Dp_{\mathrm{i+1}}}$$

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

$$\frac{dA}{d\log D_{\mathrm{i}}} = \frac{SA_{\mathrm{i}}}{\log\left(\frac{Dp_{\mathrm{i}}}{Dp_{\mathrm{i+1}}}\right)}$$

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

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

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

calculated for all intervals in the fixed pore size table.

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

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

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

where $INTERP$(x) is the value interpolated from the function $X = Dp_{\text{j+i}}$ and $Y = VP_{\text{cum,i}}$, using an AKIMA semi-spline interpolation.

(3.) Incremental Pore Volume (VpF_{i}, cm^3/g):

$$VpF_{\text{i}} = VpF_{\text{cum, i}} - VpF_{\text{cum}_{\text{i - 1}}}$$

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

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

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

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

(5.) Incremental Surface Area (SAF_{i}, m^2/g):

$$SAF_{\text{i}} = SAF_{\text{cum, i}} - SAF_{\text{cum}_{\text{i - 1}}}$$

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

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

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

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

(7.) *dV/dlog(D)* pore volume [$dV/dlog(DpF_{\mathrm{i}})$, cm^3/g]:

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

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

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

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

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

(9.) *dA/dlog(D)* pore area [$dA/dlog(DpF_{\mathrm{i}})$, m^2/g]:

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

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

Compendium of Variables

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

Dollimore-Heal Adsorption

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

Pore Diameter

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

$$D_{\mathrm{i}} = 2r_k(P_{\mathrm{i}}) + t(P_{\mathrm{i}})$$

The average pore diameter is the arithmetic mean of the diameters that bound the interval.

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

Pore Length

$$l_{\mathrm{i}} = \frac{A_{\mathrm{p,\,i}} + 10^8}{\pi \overline{D}_{\mathrm{i}}}$$

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

$$\Delta V_{\mathrm{p}} = C_\upsilon (D(Q_{\mathrm{i}-1} - Q_{\mathrm{i}}) - \Delta t \times 10^8 (A_{\mathrm{p,\,cum}} - 2\pi \bar{t}\, l_{\mathrm{i,\,cum}})$$

$$C_\upsilon = \left(\frac{\overline{D}_i}{2(\bar{r}_{\mathrm{k}} + t(P_{\mathrm{i}}) - t(P_{\mathrm{i}+1}))}\right)^2$$

$$\bar{t} = \frac{\overline{D}_i}{2 - \bar{r}_{\mathrm{k}}}$$

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

where

ΔV_p = Change in pore volume
$A_{p,i}$ = Pore surface area
$A_{p,i,cum}$, $l_{i,cum}$ = Summations over the lengths and areas calculated so far
C_v = Volume correction factor
D = Density conversion factor
$\overline{r}_k$ = Average Kelvin radius
$\overline{t}$ = Average thickness

Horvath-Kawazoe

A relative pressure lower limit is determined such that L-d_0 never equals zero. All pressure points less than this limit are discarded. For each collected relative pressure point, values of L are chosen in an iterative manner, and the relative pressure (P/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 HK)

When you use the original Horvath-Kawazoe[9] 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.

$$\ln\frac{P}{Po} = \frac{N_A}{RT}\,\frac{IP \times 10^{32}}{\sigma^4 L - 2d_0}\,\frac{\sigma^4}{3L - d_0^3} - \frac{\sigma^{10}}{9L - d_0^9} - \frac{\sigma^4}{3d_0^3} + \frac{\sigma^{10}}{9d_0^9}$$

where

10^{32} = the number of cm^4 that are equal to Å^4
σ = gas solid nuclear separation at zero interaction energy (Å), $\dfrac{Z_S + Z_A}{2}$
d_0 = $\dfrac{D_A + D_s}{2}$

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

N_A = Avogadro constant (6.02214129 × 10^{23} mol^{-1})

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 you use the Saito-Foley[10] 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.

$$\ln\left(\frac{P}{Po}\right) = \frac{3\pi N_A}{4\,RT} \times \frac{IP \times 10^{32}}{{d_0}^4} \times \sum_{k=0}^{\infty}\left[\frac{1}{k+1}\left(1-\frac{d_0}{r_p}\right)^{2k} \times \left\{\frac{21}{32}\alpha_k\left(\frac{d_0}{r_p}\right)^{10} - \beta_k\left(\frac{d_0}{r_p}\right)^{4}\right\}\right]$$

where

10^{32} = the number of cm^4 that are equal to Å^4

β_k = $\left(\frac{-1.5-k}{k}\right)^2 \beta_{k-1}, \beta_0 = 1.0$

α_k = $\left(\frac{-4.5-k}{k}\right)^2 \alpha_{k-1}, \alpha_0 = 1.0$

d_0 = $\frac{D_A + D_S}{2}$

where:

D_A = molecular diameter (Å) from the **Horvath-Kawazoe Physical Properties** window

D_S = diameter of sample atom (Å) from the **Horvath-Kawazoe Physical Properties** window

IP = interaction parameter (10^{-43} erg-cm^4) from the **Horvath-Kawazoe Report Options** window

N_A = Avogadro constant (6.02214129 × 10^{23} mol^{-1})

L = pore width (nucleus to nucleus) (Å)

P = equilibrium pressure
Po = saturation pressure
R = gas constant (8.31441×10^7 erg/mol K)
r_p = radius of the cylindrical pore, $\frac{L}{2}$
T = analysis bath temperature (K), from an entered or calculated value on the **Po and Temperature Options** window

Sphere Pore Geometry (Cheng/Yang)

When you use the Cheng/Yang[11] 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.

$$\ln\left(\frac{P}{Po}\right) = \frac{6N_1\varepsilon^*_{12} + N_2\varepsilon^*_{22}\ L^3 \times 10^{32}}{RTL - d_0^3}\left[-\left(\frac{d_0}{L}\right)^6\left(\frac{T_1}{12}+\frac{T_2}{8}\right)+\left(\frac{d_0}{L}\right)^{12}\left(\frac{T_3}{90}+\frac{T_4}{80}\right)\right]$$

where

10^{32} = the number of cm^4 that are equal to Å^4

ε^*_{12} = $\frac{Å_S}{4d_S{}^6}$, where $Å_S = \frac{6(mc^2)\alpha_S\alpha_A}{\frac{\alpha_S}{\chi_S}+\frac{\alpha_A}{\chi_A}}$

ε^*_{22} = $\frac{A_A}{4D_A{}^6}$, where $Å_A = \frac{3(mc^2)(\alpha_A)(\chi_A)}{2}$

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:

$$\ln\left(\frac{P}{Po}\right) = G(L) - \left[1 - \frac{1}{\theta}\ln\left(\frac{1}{1-\theta}\right)\right]$$

where

$G(L)$ = one of the Horvath-Kawazoe equations given above

θ = degree of void filling; θ is estimated by first computing the monolayer capacity (Q_m) with the Langmuir equation over the range of data points from relative pressure 0.02 to 0.2 or the maximum relative pressure included in the Horvath-Kawazoe analysis. θ is computed as the quantity adsorbed over Q_m.

Interaction Parameter

The interaction parameter (IP) results from the following calculations:

The Kirkwood-Muller dispersion coefficients

$$A_{\mathrm{S}} = \frac{6mc^2 a_{\mathrm{S}} a_{\mathrm{A}}}{\frac{\alpha_{\mathrm{S}}}{\chi_{\mathrm{S}}} + \frac{\alpha_{\mathrm{A}}}{\chi_{\mathrm{A}}}}$$

$$A_{\mathrm{A}} = \frac{3mc^2 \alpha_{\mathrm{A}} \chi_{\mathrm{A}}}{2}$$

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)

χ_{A} = diamagnetic susceptibility of gas molecule (cm^3)

$$IP = (N_M A_{\mathrm{A}}) + (N_{\mathrm{S}} A_{\mathrm{S}})$$

where:

N_M = 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

χ_{S} = diamagnetic susceptibility of sample atom (cm^3)

Refer to **Interaction Parameter Components**, page **C-30** for recommended values.

Additional Calculations

Based on the previous calculations, the following can be calculated:

Adjusted Pore Width (Å):
(Shell to Shell)

$$AL_\mathrm{i} = L_\mathrm{i} - Ds$$

Cumulative Pore Volume (cm^3/g):

$$V_{\mathrm{cum,\,i}} = \frac{Q_\mathrm{i} V_\mathrm{mol}}{22414\mathrm{cm}^3\mathrm{STP}}$$

where

V_mol = liquid molar volume from the fluid property information

dV/dD Pore Volume (cm^3/g-Å):

$$\frac{dV}{dD_\mathrm{i}} = \frac{V_{\mathrm{cum,\,i}} - V_{\mathrm{cum,\,i-1}}}{AL_\mathrm{i} - AL_{\mathrm{i-1}}}$$

Median Pore Width (Å):

$$V_\mathrm{half} = \frac{V_{\mathrm{cum,\,n}}}{2}$$

$$D_\mathrm{med} = 10\left[\log(D_\mathrm{l}) + [\log(V_\mathrm{half}) - \log(V_\mathrm{l})] \times \frac{\log(D_\mathrm{g}) - \log(D_\mathrm{l})}{\log(V_\mathrm{g}) - \log(V_\mathrm{l})}\right]$$

where

D_l = pore width (L_i) that corresponds to V_l
D_g = pore width (L_i) that corresponds to V_g
$V_\mathrm{cum,n}$ = total cumulative pore volume ($V_\mathrm{cum,i}$) for points designated for Horvath-Kawazoe calculations
V_g = cumulative pore volume ($V_\mathrm{cum,i}$) for first point greater than V_half
V_half = 50% of total cumulative pore volume
V_l = cumulative pore volume ($V_\mathrm{cum,i}$) for first point less than V_half

Interaction Parameter Components

Table C-1. Interaction Parameters

Gas	Bath Temperature (K)	Sample Type	Interaction Parameter Calculated Value*
Argon	87.3	Carbon (Ross/Olivier value)	2.61
		Carbon (Horvath/Kawazoe value)	5.89
		Zeolite	3.19
Carbon Dioxide	298.15	Carbon (Ross/Olivier value)	4.20
		Carbon (Horvath/Kawazoe value)	9.20
		Zeolite	5.08
	273.15	Carbon (Ross/Olivier value)	4.34
		Carbon (Horvath/Kawazoe value)	9.35
		Zeolite	5.22
	194.65	Carbon (Ross/Olivier value)	4.72
		Carbon (Horvath/Kawazoe value)	9.72
		Zeolite	5.60
Nitrogen	77.15	Carbon (Ross/Olivier value)	2.84
		Carbon (Horvath/Kawazoe value)	6.53
		Zeolite	3.49

* The interaction parameter is entered in the **Horvath-Kawazoe Report Options** window in the following field:

Interaction parameter: (calculated value) x 10^{-43} ergs-cm^{4}

The following values were used to calculate the values in Table C-1.

Carbon-Graphite

D_S = 3.40

N_S = 3.845×10^{15}

χ_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}

χ_S = 1.94×10^{-29}

α_S = 0.85×10^{-24}

Nitrogen

D_A = 3.00

N_A = 6.71×10^{14}

χ_A = 3.6×10^{-29}

α_A = 1.76×10^{-24}

Argon

D_A = 2.95

N_A = 7.608×10^{14}

χ_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)

χ_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.
χ and α values are derived from data found in Ross and Olivier[12].

The physical parameters referenced in Saito/Foley are:

Aluminophosphate

D_S = 2.60

N_S = 1.48×10^{15}

χ_S = 1.3×10^{-29}

α_S = 2.5×10^{-24}

Aluminosilicate

D_S = 2.76

N_s = 1.31×10^{15}

χ_S = 1.3×10^{-29}

α_S = 2.5×10^{-24}

DFT (Density Functional Theory)

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

This energetic heterogeneity greatly affects the shape of the adsorption isotherm with the result that simple theories such as the Langmuir and BET formulas can, at best, give only approximate estimates of surface area. Porous solids virtually are never characterized by a single pore dimension, but instead exhibit a more or less wide distribution of sizes. The observed adsorption isotherm for a typical material is therefore the convolution of an adsorption process with the distribution of one or more properties which affect that process. This was first stated mathematically by Ross and Olivier[12] for the case of surface energy distribution and has become known as the integral equation of adsorption.

The Integral Equation of Adsorption

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

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

where

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

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

Application to Surface Energy Distribution

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

$$Q(p) = \int d\varepsilon \; q(p, \varepsilon) \; f(\varepsilon) \tag{2}$$

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

$$Q(p) = \int dH \; q(p, H) \; f(H) \tag{3}$$

where

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

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

Performing the Deconvolution

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

$$Q(p) = \sum_{\mathrm{i}} q(p, Z_{\mathrm{i}})\, f(Z_{\mathrm{i}}) \qquad (4)$$

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

Regularization

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

Dubinin-Radushkevich

The Dubinin-Radushkevich[13] equation is:

$$\log(Q) = \log(Q_0) - \frac{BT^2}{\beta} \times \left[\log\frac{Po}{P}\right]^2$$

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)

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):

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

Micropore surface area (m^2/g):

$$SDP = \frac{\sigma Q_0(6.023 \times 10^{23})}{22414\ \mathrm{cm}^3\left(\frac{10^{18}\ \mathrm{nm}^2}{\mathrm{m}^2}\right)}$$

where

σ = molecular cross sectional area of gas (nm^2) from the **Adsorptive Properties** window

Dubinin-Astakhov

The Dubinin-Astakhov equation is:

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

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:

$$LV = \log(Q)$$

$$LP = \left[\log\frac{Po}{P}\right]^N$$

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):

$$V_0 = \frac{Q_{\mathrm{o}} V_{\mathrm{mol}}}{22414\mathrm{cm}^3\mathrm{STP}}$$

where

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

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

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

Characteristic Energy (KJ/mol):

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

Modal Equivalent Pore Diameter (Å):

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

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*[12].

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

Mean Equivalent Pore Width (Å):

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

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 $0 \le \times \le 1$:

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

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):

$$D_{\mathrm{i}} = 2\left[\frac{-\left(\dfrac{10^3\mathrm{nm}^3/\text{Å}^3}{\beta \cdot E_0}\right)^N}{\ln(W_{\mathrm{i}}) - \ln(W_0)}\right]^{1/3\mathrm{N}}$$

dV/dD Pore Volume (cm^3/g-Å):

$$\frac{dV}{dD_{\mathrm{i}}} = 0.5 \times W_0 \times 3N\left(\frac{10^3\mathrm{nm}^3/\text{Å}^{3N}}{\beta \cdot E_0}\right)\left(\frac{D_{\mathrm{i}}}{2}\right)^{-(3N+1)} \times \exp\left[-\left(\frac{10^3\mathrm{nm}^3/\text{Å}^3}{\beta \cdot E_0}\right)^N\left(\frac{D_{\mathrm{i}}}{2}\right)^{-3N}\right]$$

MP-Method

With the (t_i, Q_i) data pairs, the Akima semi-spline interpolation method is used to interpolate quantity adsorbed values based on thickness values that are evenly spaced 0.2 angstrom apart starting at the first outlier point. Outliers are defined as those points that have the maximum instantaneous slope within an iteratively shrinking subset of all points. The remaining pore surface area calculation result is the slope of the line defined by two consecutive interpolated points. The slopes of each pair of consecutive points from the origin to the last point must be monotonically decreasing and non-negative. With the interpolated points set the following can be calculated:

Average pore hydraulic radius (Å):

$$R_{\mathrm{i}} = \frac{t_{\mathrm{i}} + t_{\mathrm{i}-1}}{2}$$

Remaining pore surface area for the i$^{\mathrm{th}}$ **point (m^2/g):**

$$S_{\mathrm{i}} = \frac{Q_{\mathrm{i}} - Q_{\mathrm{i}-1}}{t_{\mathrm{i}} - t_{\mathrm{i}-1}} \frac{V_{\mathrm{mol}}}{22414\mathrm{cm}^3\mathrm{STP}} \times 10^4$$

where

10^4 = unit conversions
V_{mol} = liquid molar volume from the fluid property information

Incremental pore surface area occluded for the i$^{\mathrm{th}}$ **point (m^2/g):**

$$S_{\mathrm{inc,\,i}} = S_{\mathrm{i}-1} - S_{\mathrm{i}}$$

Cumulative pore surface area occluded for the i$^{\mathrm{th}}$ **point (m^2/g):**

$$S_{\mathrm{cum}_{\mathrm{i}}} = S_{\mathrm{inc,\,i}} + S_{\mathrm{inc,\,i}-1} + \ldots + S_{\mathrm{inc,\,i}}$$

dA/dR pore surface area for the ith **point (m²/g-Å):**

$$\frac{dA}{dR_{\mathrm{i}}} = \frac{S_{\mathrm{inc}_{\mathrm{i}}}}{t_{\mathrm{i}} - t_{\mathrm{i}-1}}$$

Incremental pore volume occluded for the i^{th} **point (cm^3/g):**

$$V_{\mathrm{inc,\,i}} = S_{\mathrm{inc,\,i}} R_{\mathrm{i}} \times 10^{-4}$$

Cumulative pore volume occluded for the i^{th} **point (cm^3/g):**

$$V_{\mathrm{cum,\,i}} = V_{\mathrm{inc,\,i}} + V_{\mathrm{inc,\,i}-1} + \dots + V_{\mathrm{inc,\,i}}$$

dV/dR pore volume for the i^{th} **point (cm^3/g-Å):**

$$\frac{dV}{dR_{\mathrm{i}}} = \frac{V_{\mathrm{inc,\,i}}}{t_{\mathrm{i}} - t_{\mathrm{i}-1}}$$

Thickness Curve Calculations

For each point designated, the following parameters are used in thickness curve calculations:

C_1 = parameter #1
C_2 = parameter #2
C_3 = parameter #3
$P_{\mathrm{rel,i}}$ = relative pressure for the i^{th} point
t_{i} = thickness for i^{th} point

Kruk-Jaroniec-Sayari

$$t = \left(\frac{C_1}{C_2 - \log(P_{\mathrm{rel,\,i}})}\right)^{C_3}$$

Halsey

$$t_{\mathrm{i}} = C_1\left[\frac{C_2}{\ln(P_{\mathrm{rel,\,i}})}\right]^{C_3}$$

(Halsey[5])

Harkins and Jura

$$t_{\mathrm{i}} = \left[\frac{C_1}{C_2 - \log(P_{\mathrm{rel,\,i}})}\right]^{C_3}$$

(Harkins and Jura[6])

Broekoff-de Boer

$$\log(P_{\mathrm{rel,\,i}}) = \frac{C_1}{t^2, i} + C_2 \exp(c_3 t_i)$$

Carbon Black STSA

$$t_{\mathrm{i}} = C_1(P_{\mathrm{rel,\,i}})^2 + C_2(P_{\mathrm{rel,\,i}}) + C_3$$

SPC Report Variables

Regression Chart Variables

The line of best fit for the Regression Chart is calculated by the usual Least Squares method. (Refer to *BASIC Scientific Subroutines Vol II*, by F.R. Ruckdeschel, Copyright 1981 BYTE Publications/ McGraw Hill, p. 16.) If there is only a single point or all N points have the same x-value, there can be no line of best fit in the standard form.

$$\bar{X} = \frac{\Sigma\ \mathrm{x_i}}{N}$$

$$\bar{Y} = \frac{\Sigma\ \mathrm{y_i}}{N}$$

$$\text{Slope} = \frac{\Sigma\ (x_\mathrm{i} - \bar{X})(y_\mathrm{i} - \bar{Y})}{\Sigma\ (x_\mathrm{i} - \bar{X})^2}$$

The coefficient of Correlation for this line is also calculated in the usual way. (Refer to *Mathematical Handbook for Scientists and Engineers*, by Granino A. Korn and Theresa M. Korn, Copyright 1961, 1968 McGraw Hill, Sec. 18.4.)

$$\sigma_x = \sqrt{\frac{\Sigma\ (x_\mathrm{i} - \bar{X})^2}{N}}$$

$$\sigma_y = \sqrt{\frac{\Sigma\ (y_\mathrm{i} - \bar{Y})^2}{N}}$$

$$Cov(x, y) = \frac{\Sigma\ (x_\mathrm{i} - \bar{X})(y_\mathrm{i} - \bar{Y})}{N}$$

$$\text{Correlation Coefficient} = \frac{Cov(x, y)}{\sigma_x \sigma_y}$$

Control Chart Variables

$$\text{Mean} = \frac{\Sigma\, y_\text{i}}{N}$$

$$\text{StdDev} = \sqrt{\frac{\Sigma\,(y - \text{Mean})^2}{N - 1}}$$

$$\text{CoefVar} = \frac{\text{StdDev}}{\text{Mean}}$$

$$\text{PlusNSig} = \text{Mean} + n \cdot \text{StdDev}$$

$$\text{MinusNSig} = \text{Mean} - n \cdot \text{StdDev}$$

Summary Report

The following calculations and the results of previous calculations (as noted) are used to generate the summary report:

a.) Single-point Surface Area (m^2/g)

$$S_{1\text{PT}} = \frac{[Q(1-P)] \times CSA(6.023 \times 10^{23})}{22414\text{cm}^3\ \text{STP}\left(\frac{10^{18}\ \text{nm}^2}{\text{m}^2}\right)}$$

where

P = pressure closest to 0.3 of the relative pressure points designated for surface area calculations.

Q = quantity adsorbed corresponding to P

b.) Multi-point Surface Area. Refer to **BET Surface Area**, page **C-6**.

c.) Langmuir Surface Area. Refer to **Langmuir Surface Area**, page **C-7**.

d.) t-Plot Micropore Surface Area. Refer to **t-Plot**, page **C-10**.

e.) t-Plot External Surface Area. Refer to **t-Plot**, page **C-10**.

f.) BJH Cumulative Adsorption

g.) BJH Cumulative Desorption

h.) Adsorption Total Pore Volume

i.) Desorption Total Pore Volume

j.) t-Plot Micropore Pore Volume. Refer to **t-Plot**, page **C-10**.

k.) Freundlich. Refer to **Freundlich Isotherm**, page **C-8**.

l.) Temkin. Refer to **Temkin Isotherm**, page **C-9**

m.) Alpha-S. Refer to **Alpha-S Method**, page **C-11**.

n.) DFT Pore Size and DFT Surface Energy. Refer to **DFT (Density Functional Theory)**, page **C-32**.

o.) Nanoparticle Size

$$d = \frac{6 \times 10^4}{A\rho}$$

where

ρ = sample density

A = BET surface area

d = side length (for cubic particles or diameter (for spherical particles)

p.) Dubinin-Astakhov Micropore Surface Area. Refer to **Dubinin-Astakhov**, page **C-36**.

q.) Dubinin-Astakhov Micropore Volume. Refer to **Dubinin-Astakhov**, page **C-36**.

r.) Dubinin-Radushkevich Micropore Surface Area. Refer to **Dubinin-Radushkevich**, page **C-35**.

s.) Dubinin-Radushkevich Monolayer Capacity. Refer to **Dubinin-Radushkevich**, page **C-35**.

t.) MP-Method Cumulative Surface Area of Pores

$S_{total} = S_{cum,i}$, (refer to **MP-Method**, page **C-40**) for the last collected data point used in the MP-method Calculations, and the range of hydraulic pore radii over which the cumulative surface area was computed.

u.) MP-Method Cumulative Pore Volume of Pores

$V_{\text{total}} = V_{\text{cum,i}}$, (refer to **MP-Method**, page **C-40**) for the last collected data point used in the MP-method calculations, and the range of hydraulic pore radii over which the cumulative pore volume was computed.

v.) Average Pore Hydraulic Radius (Å)

$$\bar{r} = \frac{V_{\text{total}}}{S_{\text{total}}} \times 10^4$$

w.) Horvath-Kawazoe. Refer to **Horvath-Kawazoe**, page **C-24**.

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D. FREE SPACE

Free space is that volume of the sample tube which is unoccupied by the sample. The quantity of gas dosed into the sample tube is calculated from the difference in pressures in the manifold before and after the dose is delivered. The quantity of gas adsorbed by the sample is calculated by subtracting the quantity of gas remaining in the free space of the sample tube after equilibrium is established from the quantity of gas originally dosed into the sample tube. Free space must be determined accurately to obtain a precise value for quantity adsorbed.

Static-volumetric systems consist basically of a gas manifold joined to a sample tube by an isolation valve. The manifold section has connections for an absolute pressure transducer, a temperature gauge, and a vacuum system. It also has inlets for the adsorptive gas and helium. A Dewar flask containing a cryogenic liquid (usually LN_2 at approximately 77 K) is situated so that it can be raised to immerse most of the sample tube. Two temperature zones exist within the sample tube when immersed in the cryogenic bath: a warm zone (the volume above the liquid level and near ambient temperature) and a cold zone (the volume below the liquid level at cryogenic temperature). Not only must the total free space volume be determined, but it also is necessary to determine the quantity of gas residing within the “cold” zone since a nonideality correction must be applied to only that quantity of gas.

The total quantity of gas in the partly immersed sample holder cannot simply be determined using $n = PV/RT$ because temperature is not constant over the total volume, but instead is distributed as two temperature zones with a steep temperature gradient between them. A convenient method for resolving this problem is to derive two factors which, for the existing temperature profile, can be multiplied by the prevailing pressure to reveal the molar volume of gas contained in the cold zone and the total quantity residing in the free volume of the immersed sample holder (the cold free space).

The system provides the following methods for free space determination:

- Measure
- Calculate
- Enter

Measure

Generally, this method, although requiring a little more time (approximately 10 minutes), is the most preferred one for determining free space. It is simple, automatic, requires very little information, and essentially is error-proof. With this method, the instrument first evacuates the manifold and sample tube (containing sample), then isolates the sample tube by closing the valve. Then the manifold is charged with helium, the pressure measured, and the valve opened allowing the helium to expand into the sample tube at ambient temperature. Again the pressure is measured.

The dewar is raised and the sample tube is cooled to cryogenic temperature. Again pressure drops; when pressure has equilibrated, the value is recorded. Warm and cold free spaces are calculated from (1) system volume, (2) system, ambient, and bath temperatures, and (3) measured pressures. From these, the value of the portion of cold free space at cryogenic temperature which requires correction for nonideality can be determined.

This method may be undesirable if:

- Helium is unavailable; free space determination requires the use of helium
- Analysis speed is a major factor; a helium free space measurement of 10 to 15 minutes is required
- Your sample tends to absorb and retain helium for a prolonged period of time or if it adsorbs helium

Calculate

This method is the most rapid and efficient way of compensating for free space. You must ensure the following is accomplished:

- Perform a blank analysis on the sample tube
- Load the blank analysis file data into the sample tube file
- Enter the analysis bath temperature (found on the Po and Temperature window)
- Enter the sample mass and density (found on the Sample Information window)

Enter

This method allows you to enter predetermined values for the warm and cold free spaces. The values to enter may be obtained in one of two ways:

- A pre-analysis free space calibration of the sample tube containing sample

- The total free space of an empty sample tube is measured and the displacement of the sample calculated from its mass and density and subtracted from the total free space

In either procedure, ensure that the level (or, in cases where the Isothermal Jacket is used, the effective level) of the cryogen bath on the sample tube is the same when the analysis is performed as it was when gathering data for free space calculations.

E. MAINTAINING HIGH PURITY GASES

The system was designed to accurately measure the surface area of all types of materials. It is important that the gases (especially krypton) used for these measurements be of highest purity, especially when analyzing low surface area samples. Three ways to ensure high purity gases are to always maintain:

- thoroughly purged gas pressure regulators
- non-permeable gas lines
- leak-free connections

Impure gas is strongly indicated, for example, if a series of measurements on a low surface area material yields decreasing specific surface areas with decreasing quantities of sample. The analyzer uses very small amounts of helium; therefore any residual air in the regulator can distort results of subsequent analyses for quite some time.

Micromeritics offers the following suggestions to assist you in maintaining high purity gases (particularly helium).

- Use metal gas lines only
- Remove trapped air from the regulator and gas lines

Using Metal Gas Lines

You should always use metal gas lines which have been carefully cleaned of any oils and greases used in the manufacturing process. *Do not use plastic or rubber gas lines*. When these types of permeable, nonmetallic gas lines are used with helium, contaminants accumulate at a much faster rate. This causes errors in analysis results and can also contaminate a clean sample.

Removing Trapped Air

When connecting the regulator to the gas bottle, air is unavoidably trapped on the high- and low-pressure sides of the regulator, as well as in the gas lines. You should remove as much of this air as is possible ***before*** opening the gas bottle valve. If this air is allowed to remain in the regulator, it will mix with the helium and cause inaccurate results in subsequent analyses. Or if the valve is open for any length of time, the air trapped on the high pressure side may diffuse back into the gas bottle and contaminate its entire contents.

There are two methods for removing trapped air from the regulator lines: the Purge Method and the Evacuation Method.

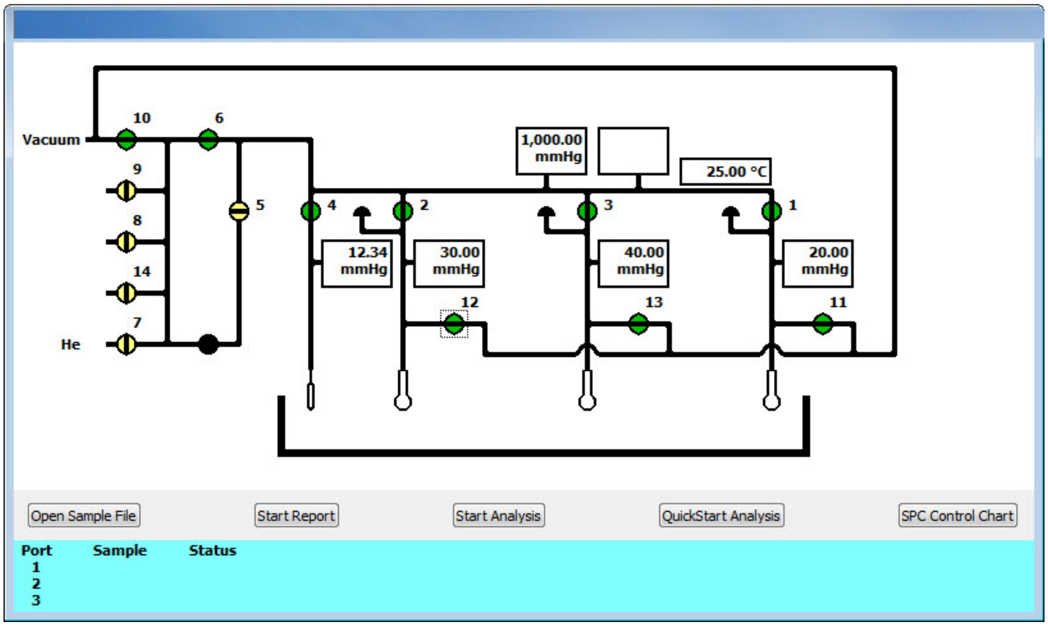
Purge Method

This is the preferred method for removing trapped air.

1. Go to ***Unit*** > ***Enable Manual Control*** (if the instrument schematic is not displayed, select ***Show Instrument Schematic***).

If multiple instruments are installed, make sure to choose the correct Unit menu.

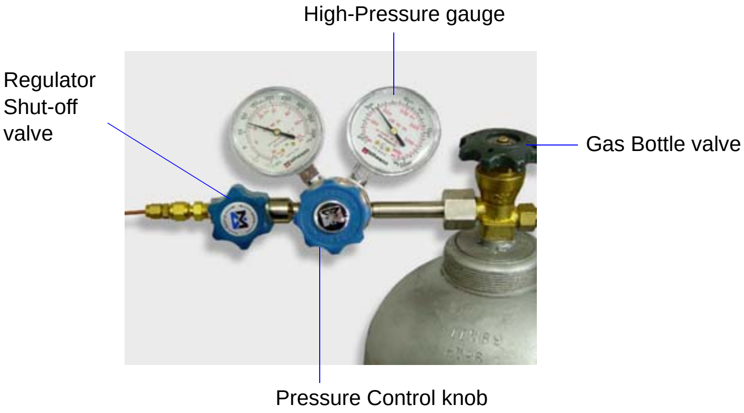
2. Close all valves by right clicking on each valve and selecting **Close**.



Port	Sample	Status
1		
2		
3		

3. Open the Regulator Shut-off valve.

4. Open the gas bottle valve ***briefly*** and allow the regulator to be charged with gas until the high-pressure gauge reads just over half the tank pressure, then ***quickly*** close the valve.



5. Using the Pressure Control knob, set the output pressure (gas bottle pressure gauge) to 15 psig.
6. Loosen the fitting at the helium inlet (on the rear panel of the instrument) until the low pressure side drops to approximately 3 psig (0.02 MPa), then tighten the fitting.
7. Repeat steps 4, 5, and 6 three times.
8. Briefly open the gas bottle valve; then, using the Pressure Control knob, reset the regulator output pressure to 15 psig.
9. After the pressure has stabilized (indicating there are no leaks), open the gas bottle valve.

Evacuation Method

To use this method, the gas tank must be within 10 feet of the instrument.

1. Do one of the following:

If...	Then...
The regulator has not been filled with gas and the gas line is attached to the instrument:	Close the gas bottle valve.
	Open the regulator Shut-off valve.
The regulator is filled with gas:	Close the gas bottle valve.
	Open the regulator Shut-off valve.
	Loosen the helium inlet fitting (or nut) on the rear panel of the instrument.
	Allow all of the gas in the regulator to expel from the line (pressure reading will be zero).
	Retighten the helium inlet fitting (or nut).

2. Go to ***Unit*** > ***Enable manual control*** (if the instrument schematic is not displayed, select ***Show instrument schematic***).

If multiple instruments are installed, make sure to choose the correct Unit menu.

3. Close all valves, then open valves 6, 7, and 10.
4. Allow evacuation to continue for 20 minutes. This pulls a vacuum on the helium line to the gas bottle. The manifold pressure transducer should fall close to zero.

Be sure to allow evacuation for a full 20 minutes. If evacuation time is too short, trapped air may remain in the lines.

5. Close valves 6, 7, and 10.

F. DFT MODELS

Theories are developed by scientists in an attempt to explain a class of observed behavior. In the experimental physical sciences, theories are often expressed in terms of a model that can be visualized and described mathematically. Early models of physical adsorption were quite simple, both conceptually and mathematically, for very practical reasons — hand computations were required. Today we can explore complex models that describe adsorption systems on the atomic scale of size and sub-picosecond time frame. This is not because scientists are smarter, but because of available tools. The DFT models are created by classical approaches to adsorption as well as models based on modern statistical thermodynamics.

Models Based on Statistical Thermodynamics

Included in this group are methods that model the adsorption system in terms of forces acting between individual molecules.

Theoretical Background

Traditional adsorption theories attempt to describe experimental adsorption isotherms with an isotherm equation containing a small number of parameters. At a minimum, these parameters include the extent of the surface, such as the monolayer capacity (Q_m), and the molar intensity of the gas-surface interaction, such as the Langmuir “K” constant or the BET “C” constant. In some equations, additional parameters take into account the lateral interaction of adsorbed molecules with each other. Other theories, such as the Dubinin-Astakhov approach, also include parameters for the effect of adsorbent porosity.

Instead of this classical kinetic or phenomenological approach, we can use a molecular-based statistical thermodynamic theory that allows us to relate the adsorption isotherm to the microscopic properties of the system: the fluid-fluid and fluid-solid interaction energy parameters, the pore size, the pore geometry, and the temperature.

The following example is given so that you may understand how such a theory is constructed.

A clean sample of a solid material containing slit-shaped pores of a single width is placed in an evacuated space. It is kept at a fixed temperature as a known quantity of pure argon gas is admitted into the space surrounding the sample. The pressure within the space is recorded over time. In this situation, the pressure falls rapidly from its initial value and gradually approaches a steady reading, called the equilibrium pressure. The amount adsorbed corresponds to the quantity of gas effectively removed from the gas phase by the solid surface. A graph that plots amount adsorbed versus equilibrium pressure is called an adsorption isotherm.

Under such conditions, the argon atoms that randomly enter the pore space feel the presence of the solid surface as the action of an external attractive force (the dispersion forces or Van der Waal’s forces) and spend more time near the surface. As a result, the space near the surface acquires a greater average density of argon atoms than regions farther removed.

If the equilibrium distribution of the gas atoms near the surface could be described as a function of pressure and the molecular properties of the components of the system, then a model could be constructed for the adsorption isotherm for the system. Modern physical chemistry provides several ways to calculate this distribution. All these methods are based on the fundamental thermodynamic law that such a system adopts a configuration of minimum free energy at equilibrium. Also needed is a description of the pairwise interaction energy between atoms, U(s), commonly given by a Lennard-Jones potential:

$$U(s) = 4\varepsilon\left[\left(\frac{\sigma}{s}\right)^{12} - \left(\frac{\sigma}{s}\right)^{6}\right]$$

where

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* is determined by the pressure of the system using well-known equations. The fluid near the walls is not of constant density; its chemical potential is composed of several position-dependent contributions that must total at every point to the same value as the chemical potential of the bulk fluid.

As noted previously, at equilibrium, the whole system has a minimum (Helmholtz) free energy, known thermodynamically as the grand potential energy (GPE). Density functional theory describes the thermodynamic grand potential as a functional of the single-particle density distribution; therefore, calculating the density profile that minimizes the GPE yields the equilibrium density profile. The calculation method requires the solution of a system of complex integral equations that are implicit functions of the density vector. Since analytic solutions are not possible, the problem must be solved using iterative numerical methods. Although calculation using these methods still requires supercomputing speed, the calculation of many isotherm pressure points for a wide range of pore sizes is a feasible task. The complete details of the theory and the mathematics can be found in the papers listed under **References** at the end of this appendix.

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

Figure D-1 shows the density profile for argon at a carbon surface as calculated by density functional theory for a temperature of 87.3 K and a relative pressure of about 0.5.

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*.Chemical potential may be thought of as the energy change felt by a probe particle when it is inserted into the system from a reference point outside the system. It can also be defined as the partial derivative of the grand potential energy with respect to density (or concentration).

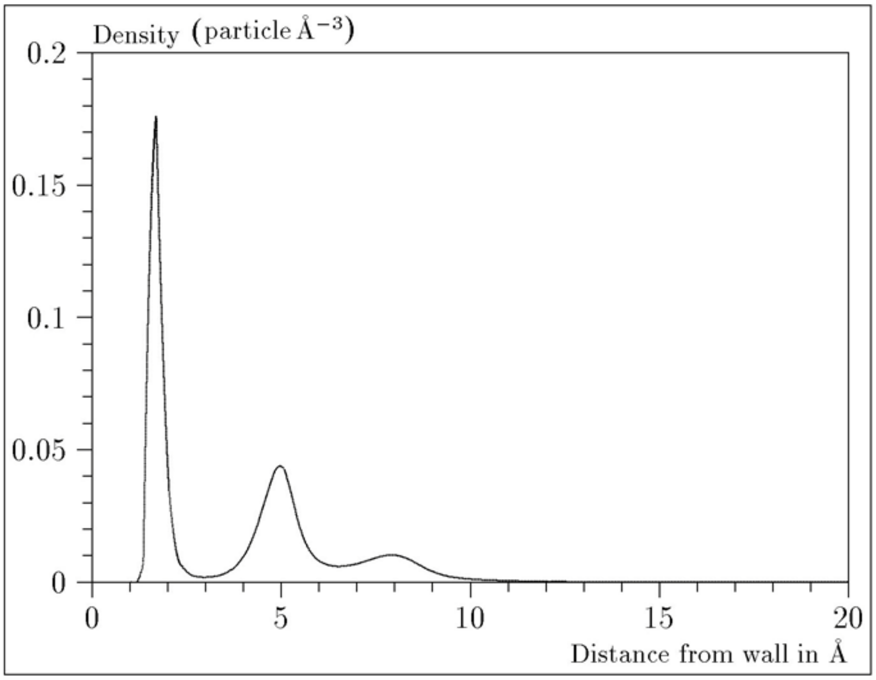

Figure D-1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

This figure represents a cross-section of the region near the surface. Note the layerwise distribution of adsorbate; the first monolayer is sharply defined and a third layer can be distinguished. The area under the profile curve represents the amount adsorbed per unit area at this pressure. The positions of the maxima are separated by a distance determined by the size of the adsorptive atom.

Given the density profile, the amount adsorbed at the stated pressure can be easily calculated as the integral over the profile. Repeating this calculation over a range of pressures yields the adsorption isotherm for the model. If the value of H is very large, the isotherm obtained corresponds to that of an external, or *free*, surface. If H is smaller, a range of pressures is reached where two minima exist for the grand potential, showing the presence of two metastable phases having different density distributions but the same chemical potential. The phase with the lower GPE is the stable one. As the pressure is increased, a point is reached where the other phase becomes the stable one. This phase transition reflects condensation of adsorbate in the pore; the pressure at which it occurs is called the *critical pore-filling pressure*. This pressure is analogous to the condensation pressure predicted by the Kelvin equation in the classical model of pore filling.

Figure D-2 shows how the profiles change with pressure for a model pore with H = 40 Å. The insets show the density profiles for the corresponding points of the isotherm.

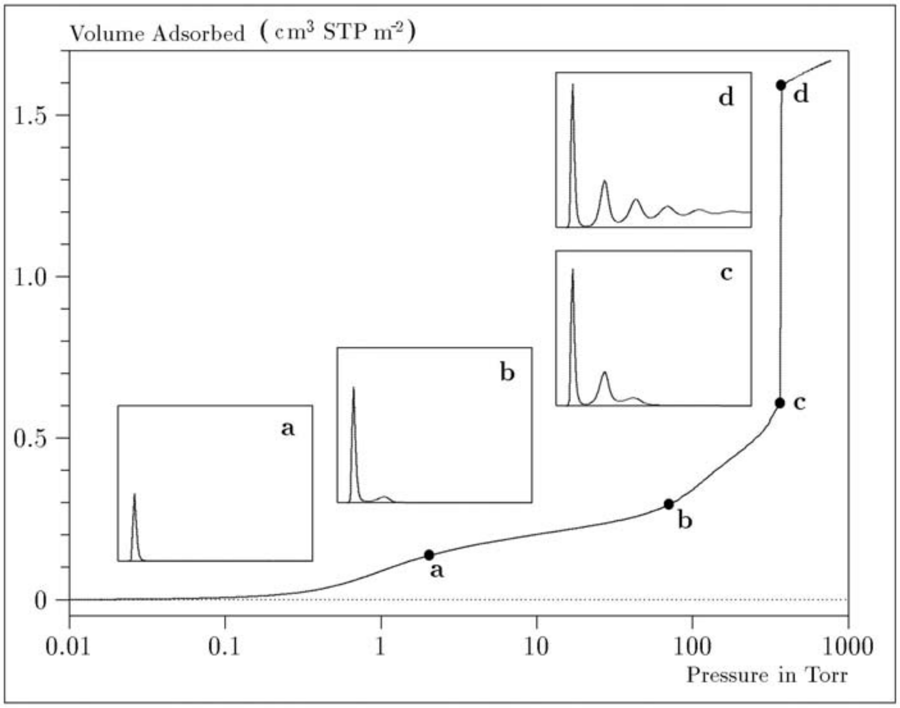

Figure D-2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

The profiles show the density distribution from one wall to the center of the slit; the other half of the distribution is a mirror image of the profile shown.

As the pressure is first increased from zero, almost all the adsorbed atoms occupy a position close to the surface.

- Inset **a** shows the profile corresponding to point a on the isotherm where the surface is about half covered.
- At point **b**, the first layer is so full that it is more favorable for atoms to start a new layer.
- At point **c**, a third layer is forming. Point c, for this size slit, is the critical pore-filling pressure. In inset c, the profile shows the density decreasing to near zero (actually the bulk gas density) at 4 or 5 molecular diameters from the surface.
- Inset **d** shows the profile converging on a density similar to that of bulk liquid argon in the center of the pore, indicating a phase transition.

Note that the adsorption isotherms for pores larger than the one shown in *Figure D-2* is identical up to point **c**. The lower branch of the isotherm simply continues to a higher pressure for larger pores. This trend is illustrated in *Figure D-3*, where isotherms for some larger size pores are shown. It is clear that pore size is uniquely characterized by a corresponding critical pore-filling pressure. At large pore sizes, density functional theory produces results for the critical filling pressures that are in good agreement with those produced by the Kelvin equation.

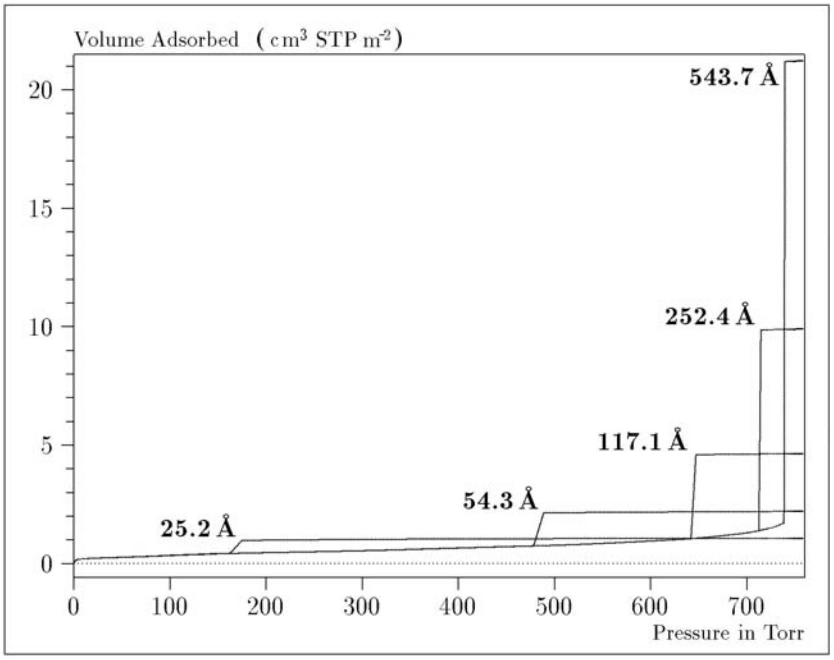

Figure D-3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

Figure D-4 shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.

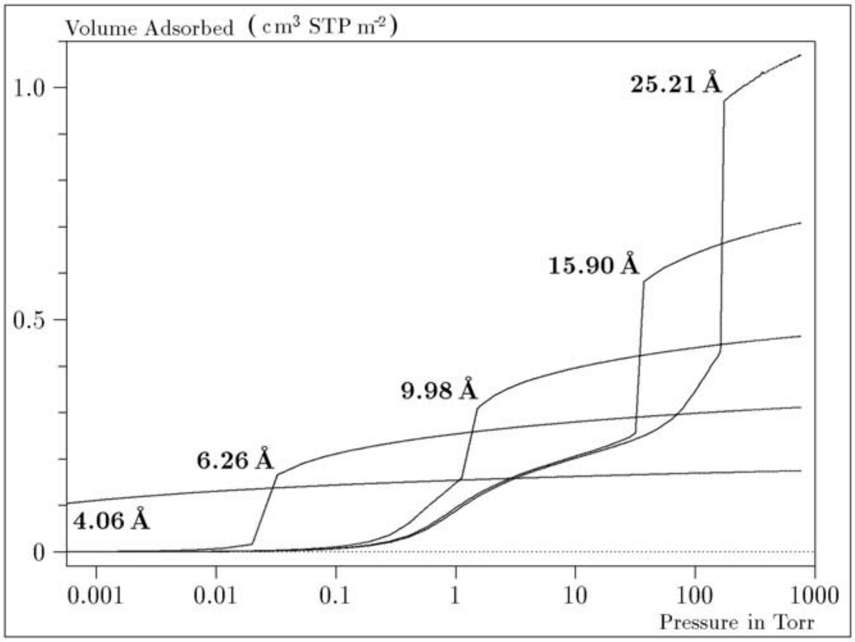

Figure D-4. Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

Pores of 4 Å width, barely larger than the argon atom (3.38 Å), fill at pressures below 1 millitorr. Pores below 15 Å fill before a monolayer is completed on the surface of the larger pores. In the micropore size range, the pore volume fills more gradually with pressure and the total shape of the isotherm is important in characterizing the pore size.

Models Included

Non-Local Density Functional Theory with Density-Independent Weights

N2 - DFT Model
AR - DFT Model

Geometry:	Slit
Substrate:	Carbon (graphite)
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Using the methods of non-local density functional theory, two sets of isotherms have been calculated to serve as kernel functions for the characterization of porous solids from adsorption data. The model isotherms are stored in binary format files. These models assume a *slit-like pore geometry*. The pore size range from 4.0 to 4000 Å is covered in 91 classes in a geometric progression. The class intervals are rounded to the nearest 0.02 molecular diameters. A model for the free or external surface is included to account for unfilled pores. Each of the 92 model isotherms has been calculated at 181 pressure points from near $1x10^{-6}$ to near 1.00 relative pressure.

These models are identical to those supplied with the original DOS version of DFT software. Some slight difference from the DOS results may be noted when they are applied to the same data due to improvements in the deconvolution algorithm and better regularization of the current software.

Non-Local Density Functional Theory with Density-Dependent Weights

N2 - Modified Density Functional

Geometry:	Free surface
Substrate:	Surface energy
Method:	Nitrogen at 77 K

Using the modified Tarazona prescription described by Olivier (refer to **References**, numbers 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’s constant. The units are therefore Kelvin.

N2 - Cylindrical Pores - Oxide Surface
AR - Cylindrical Pores - Oxide Surface

Geometry	Cylinder
Substrate	Oxide
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using a combination of statistical mechanical calculations and experimental observations for macroporous silicas and MCM-41 mesoporous silicas as well as zeolites. The pore-filling pressures were determined as a function of the pore size from adsorption isotherms on MCM-41 materials characterized by X-ray and other techniques. The variation of the pore fluid density with pressure and pore size has been accounted for by density functional theory calculations. The N2 model reports pore sizes ranging from 3.8 to 387 Å and the AR model from 3.8 to over 500 Å.

Reference: M. Jaroniec, M. Kruk, J.P. Olivier, and S. Koch, “A New Method for the Accurate Pore Size Analysis of MCM-41 and Other Silica-Based Mesoporous Materials,” Proceedings of COPS-V, Heidelberg, Germany (1999).

N2 – Cylindrical Pores – Pillared Clay Surface (Montmorillionite)

Geometry	Cylinder
Substrate	Crystalline Silicate
Category:	Porosity
Method:	Nitrogen at 77 K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFT) calculations and experimental isotherms for reference samples of 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 Å.

References:	Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V* , Unger, et al, Eds, Elsevier, Amsterdam, 2000.
	James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of a Pillared Interlayered Clay (PILC) from a Hybrid Density Functional Theory (DFT) Method," *The Journal of Physical Chemistry B*; 2001, 105(3), 623-629.
	M. L. Occelli, J. P. Olivier, J. A. Perdigon-Melon, and A. Auroux, "Surface Area, Pore Volume Distribution, and Acidity in Mesoporous Expanded Clay Catalysts from Hybrid Density Functional Theory (DFT) and Adsorption Microcalorimetry Methods," *Langmuir* 2002, 18, 9816-9823.9b.
	James P. Olivier, "The Importance of Surface Heterogeneity in Developing Characterization Methods." *6th International Symposium on the Characterization of Porous Solids*, Studies in Surface Science and Catalysis 144, Elsevier, 2002.
	James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of Pillared Rectorite Catalysts from a Hybrid Density Functional Theory Method," *Microporous and Mesoporous Materials* 2003, 57, 291-296.

C02 - DFT Model

Geometry	Slit
Substrate	Carbon
Category:	Porosity
Method:	Carbon dioxide at 273 K

Model isotherms were calculated using the non-local prescription of Tarazona, employing molecular parameters derived from the known bulk properties of carbon dioxide.

AR - Modified Density Functional Model

Geometry	Free Surface
Substrate	Any
Category:	Surface energy
Method:	Argon at 87 K

This model was produced in the same manner as the N2 Modified Density Functional model listed earlier, except applicable to argon adsorbed at 87.3 K.

N2 - Tarazona NLDFT, Esf = 30.0K

Geometry	Cylinder

Substrate	Oxide
Category:	Porosity
Method:	Nitrogen at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a cylindrical pore geometry. The wall potential used is k = 30 K, typical for a silica or alumina surface.

This model file is particularly useful for sizing zeolites or zeolite containing materials that have substantial micropore volume. The reported pore size range is 3.8 to 387 Å.

Reference: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Slit Pores by NLDFT
Ar - Carbon Slit Pores by NLDFT

Geometry	Slit
Substrate	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a slit-like pore geometry. These models are slightly different from N2-DFT and Ar-DFT models that were calculated using NLDFT with density independent weighting functions.

The reported pore size range is from 3.5 to 1000 Å.

Reference: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Finite Pores, As=6, 2D-NLDFT
Ar - Carbon Finite Pores, As=6, 2D-NLDFT

Geometry	Finite Slit
Substrate	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions assuming 2D model of finite slit pores having a diameter-to-width aspect ratio of 6.

This model is particularly useful for microporous carbon materials. The reported pore size range is from 3.5 to 250 Å.

Reference:	Jacek Jagiello and James P. Olivier. “A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.,” *The Journal of Physical Chemistry C*, 113(45):19382-19385, 2009.

N2 - Carbon Finite Pores, As=12, 2D-NLDFT
Ar - Carbon Finite Pores, As=12, 2D-NLDFT

Geometry	Finite Slit
Substrate	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the same methods and assumptions that were used in the model above except in this model, the aspect ratio is equal to 12.

These two finite pore models may be used as a research tool in conjunction with independent analytical techniques such as high-resolution transmission electron microscopy (HRTEM) and/or X-ray diffraction (XRD) to obtain comprehensive information about the structure of studied carbon material.

Reference:	See above reference.

N2 - Carbon Cylinder, single-wall nanotube by NLDFT
Ar - Argon Cylinder, single-wall nanotube by NLDFT

Geometry	Cylinder
Substrate	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the graphitic surface of an infinitely long cylinder.

This model is particularly useful for characterizing carbon single-wall nanotubes. The reported pore size range is from 3.5 to 1000 Å.

Reference:	P. Tarazona, Phys. Rev. A 31: 2672 (1985). Idem, Phys. Rev. A 32: 3148 (1985). P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Cylinder, multi-wall nanotube by NLDFT
Ar - Argon Cylinder, multi-wall nanotube by NLDFT

Geometry	Cylinder
Substrate	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and multiple concentric graphitic surfaces of infinitely long cylinders.

This model is particularly useful for characterizing carbon multi-wall nanotubes. The reported pore size range is from 3.5 to 1000 Å.

Reference: See above reference.

Ar - Zeolites H-Form by NLDFT

Geometry	Cylinder
Substrate	Zeolite
Category:	Porosity
Method:	Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is particularly useful for characterizing oxides and H^+ and $(NH_4)^+$ exchanged zeolites. The reported pore size range is from 3.5 to 300 Å.

Ar - Zeolites Me-Form by NLDFT

Geometry	Cylinder
Substrate	Zeolite
Category:	Porosity
Method:	Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is similar to the model above, but it more appropriate is for characterizing alkali metal exchanged zeolites. The reported pore size range is from 3.5 to 300 Å.

Models Based on Classical Theories

Both surface energy distribution and pore size distribution may be evaluated using classical approaches to model kernel functions for use with equation (1) of the DFT Theory in the calculations appendix. Be aware that the deconvolution method only provides a fitting mechanism; it does not overcome any inherent shortcomings in the underlying theory.

Surface Energy

The use of classical theories to extract adsorptive potential distribution is mostly of historical interest. At a minimum, the equation must contain a parameter dependent on adsorption energy and another dependent on monolayer capacity or surface area. This is sufficient to permit the calculation of the set of model isotherms that is used to create a library model. The Langmuir equation has been used in the past, as have the Hill-de Boer equation and the Fowler-Guggenheim equation. All of these suffer from the fact that they only describe monolayer adsorption, whereas the data may include contributions from multilayer formation.

Pore Size

It is well established that the pore space of a mesoporous solid fills with condensed adsorbate at pressures somewhat below the prevailing saturated vapor pressure of the adsorptive. When combined with a correlating function that relates pore size with a critical condensation pressure, this knowledge can be used to characterize the mesopore size distribution of the adsorbent. The correlating function most commonly used is the Kelvin equation. Refinements make allowance for the reduction of the physical pore size by the thickness of the adsorbed film existing at the critical condensation pressure. Still further refinements adjust the film thickness for the curvature of the pore wall.

The commonly used practical methods of extracting mesopore distribution from isotherm data using Kelvin-based theories, such as the BJH method, were for the most part developed decades ago and were designed for hand computation using relatively few experimental points. In general, these methods visualize the incremental decomposition of an experimental isotherm, starting at the highest relative pressure or pore size. At each step, the quantity of adsorptive involved is divided between pore emptying and film thinning processes and exactly is accounted for. This computational algorithm 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 at 77 K

The kernel function is calculated using the Halsey equation with standard parameters:

$$t = 3.54\left(\frac{-5.00}{\ln(P/\mathrm{Po})}\right)^{1/3}$$

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 at 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

Reference: G. Halsey, J. Chem. Phys 16, 931 (1948).

Kelvin Equation with Harkins and Jura Thickness Curve

N2 - Harkins and Jura Thickness Curve

Geometry	Slit
Substrate	Average
Category:	Porosity
Method:	Nitrogen at 77 K

The kernel function is calculated using the Harkins and Jura equation with standard parameters:

$$t = \left(\frac{13.99}{0.034 - \log(P/Po)}\right)^{1/2}$$

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 at 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

Reference: W. D. Harkins and G. Jura, J.A.C.S. 66, 1366 (1944).
J. H. DeBoer et al., J. Colloid and Interface Sci. 21, 405 (1966).

Kelvin Equation with Broekhoff-de Boer Thickness Curve

N2 - Broekhoff-de Boer Model

Geometry	Slit
Substrate	Average
Category:	Porosity
Method:	Nitrogen at 77 K

The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

$$\log(p/p^{0}) = \frac{-16.11}{t^{2}} + 0.1682^{-0.1137^{t}}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}

Molar density = 0.02887 g cm^{-3}

N2 - Broekhoff-de Boer Model

Geometry	Cylinder
Substrate	Average
Category:	Porosity
Method:	Nitrogen at 77 K

The calculation is similar to the above except that cylindrical geometry is assumed, and the film thickness depends on pore size (see reference).

Reference: Specifically, equations 20 and 21 in: J.C.P. Broekhoff and J.H. de Boer, “The Surface Area in Intermediate Pores,” Proceedings of the International Symposium on Surface Area Determination, D.H. Everett, R.H. Ottwill, eds., U.K. (1969).

References

The papers listed below provide additional information on DFT models:

1. “Determination of Pore Size Distribution from Density Functional Theoretic Models of Adsorption and Condensation within Porous Solids,” J.P. Olivier and W.B. Conklin, Micromeritics Instrument Corp; presented at the International Symposium on the Effects of Surface Heterogeneity in Adsorption and Catalysts on Solids, Kazimierz Dolny, Poland (July 1992).

2. “Classification of Adsorption Behavior: Simple Fluids in Pores of Slit-shaped Geometry,” Perla B. Balbuena and Keith E. Gubbins, *Fluid Phase Equilibria*, 76, 21-35, Elsevier Science Publishers, B.V., Amsterdam (1992).

3. “Modeling Physical Adsorption on Porous and Nonporous solids Using Density Functional Theory,” J.P. Olivier, *Journal of Porous Materials*, 3, 9-17 (1995).

4. “The Determination of Surface Energetic Heterogeneity Using Model Isotherms Calculated by Density Functional Theory,” J.P. Olivier; presented at the Fifth International Conference on the Fundamentals of Adsorption, Pacific Grove, CA (1995).

G. USER-DEFINED REPORTS, PYTHON MODULE

The mic Python module allows you to access primary and overlay isotherm data and create graphical, tabular, and summary reports. Graphical reports consist of a single graph with one or more curves on one or two y-axes. Tabular reports consist of one or more tables consisting of one or more labeled columns of data. Summary reports consist of summary sections, each containing a two-column table of label and value pairs.

TABLE 1: Example Python Script for User-defined Reports

1. import mic	Import the mic (required) and numpy (optional) packages.
2. import numpy as np 3. # Isotherms as list of components	
4. # Isotherms as list of components 5. iso_rel = mic.isotherm('rel')	Get the isotherm data.
6. iso_abs = mic.isotherm('abs') 7. # or as components. 8. prel, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('rel') 9. pabs, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('abs') 10. # Overlays work the same way but are not guaranteed to be valid.	
11. overlays = []	Get the overlay isotherm data.

```
12. for i in range( 8 ) :
13.     ov = mic.overlay(i, 'rel')
14.     overlays.append( ov if ov[0].any() else None )
15. # A graphical report with 3 curves.
```

16. mic.graph('Graphical Report 1', 'Rel. Press', 'Qads')	Add a graphical report.
17. mic.graph.add('Sample isotherm', prel, qads)	Add some curves to the report.
18. mic.graph.add('Linear transform', prel, qads * 4.0 + 1.0) 19. mic.graph.addyy('Another transform', prel, np.log(qads * qads)) 20. # Another graphical report, this time with 1 curve	
21. mic.graph('Graphical Report 2')	Add another graphical report.
22. mic.graph.add('Sample isotherm', pabs, qads) 23. # A tabular report with 2 tables.	
24. mic.table()	Add a tabular report.
25. mic.table.addtable("Table 1")	Add a table to the report.

TABLE 1: Example Python Script for User-defined Reports *(continued)*

26. mic.table.addcolumn("Column 1",	Add columns to the table. Note that the column values are strings; this allows arbitrary formatting.
27. ["1", "2.5", "%8.3f" % np.exp(5.0)]) 28. mic.table.addcolumn("Column 2", 29. ["Val A", "Val B", "Val C" % np.exp(5.0)]) 30. mic.table.addtable("Table Two") 31. mic.table.addcolumn("Column A", ["Small", "Smaller"]) 32. mic.table.addcolumn("Column B", ["Big", "Bigger"]) 33. # Another tabular report	
34. mic.table("Tabular Report Two")	Add another table.
35. mic.table.addtable("One") 36. mic.table.addcolumn("Column A", ["Small", "Smaller"]) 37. mic.table.addcolumn("Column B", ["Big", "Bigger"]) 38. mic.table.addcolumn("Column C", ["Embiggen", "Cromulent"]) 39. # A summary report	
40. mic.summary("A summary report")	Add a summary report.
41. mic.summary.add("Quantity Adsorbed",	Add sections to the summary report. Note that summary sections are just specialized reports with two columns and no column headers.
42. ["First", "Last"], 43. ["%8.3f" % qads[0], "%8.3f" % qads[-1]) 44. mic.summary.add("Rel Pressure", 45. ["First", "Last"], 46. ["%8.3f" % prel[0], "%8.3f" % prel[-1])	

Note that the mic module is automatically imported when the user script is run. For test purposes, a mic.pyc pre-compiled module will be provided.

TABLE 2: Function Reference for mic Python Module (User-defined Reports)

mic.isotherm(press_type='rel')

Get the primary isotherm data.

Keyword arguments:
press_type --- the pressure basis; use 'rel' for relative pressure, 'abs' for absolute (default = 'rel')

Usage:
p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.isotherm('rel')

p --- array of pressure (relative or absolute)
qads --- array of cumulative quantity adsorbed
num_ads --- number of points in the adsorption curve
warm_fs --- warm free-space
cold_fs --- cold free-space
mass --- sample mass
desc --- sample description

mic.sample_information(item, sample_number = 0) :

Get Sample Information item

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:

item = sample_information('sample mass')
item = sample_information('sample mass',0)

TABLE 2: Function Reference for mic Python Module (User-defined Reports) *(continued)*

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

    Get report results for the indicated report and sample

    Keyword arguments:

      sample_number --- Identifier for the sample data to retrieve (default = 0).
                          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'  )

    Report Name and corresponding named report result

      bet
        surface area --- Surface arrea ( m^2/g )
        bet constant  --- BET constant ( dimensionless )
        monolayer capacity  --- Monolayer capacity ( cm^3/g )

      tplot
        external surface area --- External surface area (m^2/g)
        micropore volume  --- Micropore volume (cm^3/g)

      bjhads
        incremental distribution

      bjhdes
        incremental distribution

      dhads
        incremental distribution

      dhdes
        incremental distribution

      hk
        incremental distribution

      dft
        incremental distribution

      nldft
        incremental distribution
```

TABLE 2: Function Reference for mic Python Module (User-defined Reports) ***(continued)***

```
 where the pore distribution result is structured as follows

      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.

mic.overlay(overlay_number=1, press_type='rel')

    Get overlay isotherm data.

    Keyword arguments:
    overlay_number      --- the overlay number (1 through 8)
    press_type          --- the pressure basis; use 'rel' for relative pressure, 'abs' for
                               absolute (default = 'rel')

    Usage:
    p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.overlay(1, 'rel')

    p      --- array of pressure (relative or absolute); empty-array if overlay is unavailable.
    qads    --- array of cumulative quantity adsorbed; empty-array if overlay is unavailable.
    num_ads  --- number of points in the adsorption curve; 0 if overlay is unavailable.
    warm_fs --- warm free-space; 0.0 if overlay is unavailable.
    cold_fs --- cold free-space; 0.0 if overlay is unavailable.
    mass    --- sample mass; 0.0 if overlay is unavailable.
    desc    --- sample description; empty-string if overlay is unavailable.
```

TABLE 2: Function Reference for mic Python Module (User-defined Reports) *(continued)*

mic.imported_pore_data(import_number = 1)

Get imported pore data.

Keyword arguments:
import_number --- the import number (1 through 8)

Usage:

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

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

mic.adsorptive_data(sample_number = 0)

Get adsorptive data for each sample

Keyword arguments:
sample_number --- Identifier for the adsorptive data to retrieve (default = 0).
0 : the current sample file
1 through 8 : the corresponding overlay sample file

Usage:

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

csa --- cross sectional area (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')

TABLE 2: Function Reference for mic Python Module (User-defined Reports) *(continued)*

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

Create a new graphical report.

Keyword arguments:
title --- the graphical report title (default = 'User Graph')
xlabel --- x-axis label (default = 'X axis')
ylabel --- y-axis label (default = 'Y axis')
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)

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

Add a curve to the last created graphical report.

Keyword arguments:
name --- the curve name
x --- list of x values; must be a list of floats (or convertible) and the same length as y
y --- list of y values; must be a list of floats (or convertible) and the same length as x
yyaxis --- place this curve on the yy-axis if True otherwise place on the y-axis (default = False)
color --- RGB color as an HTML hex string (e.g., '#4169e1') or a three- element list or tuple (e.g., [65,105,225]); if None, color is automatically selected (default = None)
linestyle --- line style; (default = '-')
  '-' : solid
  '--' : dash
  '.' : dot
  '-.' : dash dot
  '-..' : dash dot dot
marker --- marker shape; (default = 'a')
  '' or None : no marker
  '+' : plus
  'o' or '0' : circle
  'x' : cross
  '^' : up triangle
  'v' : down triangle
  's' : square
  'd' : diamond
  '8' : hourglass
  '~' : horizontal hourglass
  'a' : automatically selected

TABLE 2: Function Reference for mic Python Module (User-defined Reports) *(continued)*

graphtype --- graph type; (default = 'both')
'curve' or 'c' : curve
'points' or 'p' : points
'both' or 'b' : curve-and-points
'hist' or 'h' : histogram

mic.table(title='User Table')

Create a new tabular report.

Keyword arguments:
title --- the tabular report title (default = 'User Table')

mic.table.addtable(name)

Add a table to the last created tabular report.

Keyword arguments:
name --- the table name

mic.table.addcolumn(header, values)

Add a column to the last created table.

Keyword arguments:
header --- column header; must be a string (or convertible)
values --- column values; must be a list of strings (or convertible)

mic.summary(title='User Summary')

Create a new summary report.

Keyword arguments:
title --- the summary title

mic.summary.add(self, name, labels, values)

Add a summary section to the last created summary report.

Keyword arguments:
name --- summary section name
labels --- column of labels; must be a list of strings (or convertible) and the same length as values
values --- column of values; must be a list of strings (or convertible) and the same length as values

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