MICROACTIVE ASAP® 2460

ACCELERATED SURFACE AREA AND POROSIMETRY SYSTEM

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Effective Solutions for
Material Characterization

OPERATOR MANUAL

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ABOUT MICROMERITICS

Micromeritics Instruments Corporation is a leading global provider of solutions for material characterization with best-in-class instrumentation and application expertise in five core areas: density; surface area and porosity; particle size and shape; powder characterization; and catalyst characterization and process development. Founded in 1962, the company is headquartered in Norcross, Georgia, USA and has more than 300 employees worldwide. With a fully integrated operation that extends from a world class scientific knowledge base through to in-house manufacture, Micrometrics delivers an extensive range of high-performance products for academic research and industrial problem-solving. The implementation of tactical partnerships to incubate and deliver valuable new technologies exemplifies the company's holistic, customer-centric approach which extends to a cost-efficient contract testing laboratory – the Particle Testing Authority (PTA). The strategic acquisitions of Freeman Technology Ltd and Process Integral Development S.L. (PID Eng & Tech) reflect an ongoing commitment to optimized, integrated solutions in the industrially vital areas of powders and catalysis.

Freeman Technology (Tewkesbury, UK) brings market-leading powder characterization technology to Micromeritics' existing portfolio of particle characterization techniques. The result is a suite of products that directly supports efforts to understand and engineer particle properties to meet powder performance targets. With over 15 years of experience in powder testing, Freeman Technology specializes in systems for measuring the flow properties of powders. In combination with detailed application know-how these systems deliver unrivalled insight into powder behavior supporting development, formulation, scale-up, processing and manufacture across a wide range of industrial sectors.

PID Eng & Tech (Madrid, Spain) complements Micromeritics' renowned offering for catalyst characterization with technology for the measurement and optimization of catalytic activity, with a product range that extends to both standard and bespoke pilot scale equipment. Launched in 2003, PID Eng & Tech is a leading provider of automated, modular microreactor systems for the detailed investigation of reaction kinetics and yield. These products are supported by a highly skilled multidisciplinary team of engineers with in-depth expertise in the design, construction and operation of laboratory units and process scale-up.

The Particle Testing Authority (PTA) provides material characterization services for fine powders and solid materials using Micromeritics' instrumentation alongside complementary solutions from other vendors. With the certification and expertise to operate across a wide range of industries the PTA offering runs from single sample analysis to complex method development, method validation, new product assessment, and the analytical support required for large scale manufacturing projects. An experienced, highly trained team of scientists, engineers, and lab technicians works closely with every client to ensure that all analytical requirements are rapidly and responsively addressed.

Micromeritics has a strong global network with offices across the Americas, Asia, and Europe complemented by a dedicated team of distributors in additional locations. This ensures that local, knowledgeable support is available for every customer, in academia or industry. Micromeritics works across a truly diverse range of industries from oil processing, petrochemicals and catalysts, to food and pharmaceuticals, and at the forefront of characterization technology for next generation materials such as graphene, metal-organic-frameworks, nanocatalysts, and zeolites. Engineering solutions that work optimally for every user is a defining characteristic of the company.

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ABOUT THIS MANUAL

Log in to your customer portal to access the following:

- Calculations
- Error Messages
- MicroActive ASAP 2460 Operator Manual in PDF format
- MicroActive Report Tutorials
- Smart VacPrep Operator Manual in PDF format

Parts and accessories can be found online at www.Micromeritics.com.

 This manual contains instructions for both standard installations and installations in 21CFR11 environments.

The following icons may be found in this manual:

 NOTE — Notes contain important information applicable to the topic.

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

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

 NOTE — Notes that apply to 21CFR11 environments only (Confirm applications).

Table of Contents

8 Selected Reports ⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯ *8 - 1*

9 Diagnostics ⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯ *9 - 1*

1 ANALYZER COMPONENTS

FRONT COMPONENTS

Front Components

P_0 port connector	For P_0 (saturation pressure) tube installation.
Power indicator	Blinks when power is applied to the analyzer; illuminates when the analysis program is initiated and ready for operation.
Sample port connector	For sample tube installation.

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SIDE PANEL COMPONENTS - MASTER MODULE
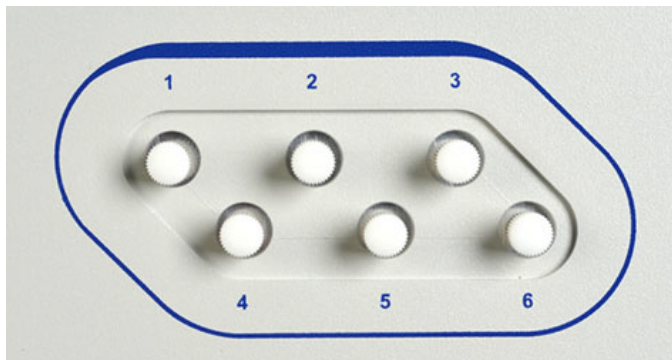
Side Panel Components

Component	Description
Gas inlet ports 1 - 6	Use to connect up to six analysis gas supplies to the analyzer. When measuring free space, any one of the ports may be used for helium.

BACK PANEL COMPONENTS - MASTER UNIT
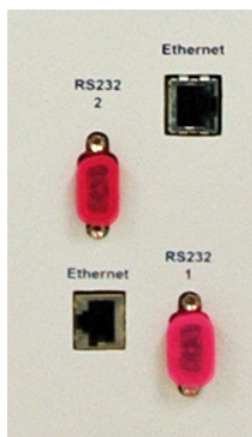
**Ethernet Ports
and RS232 Ports**
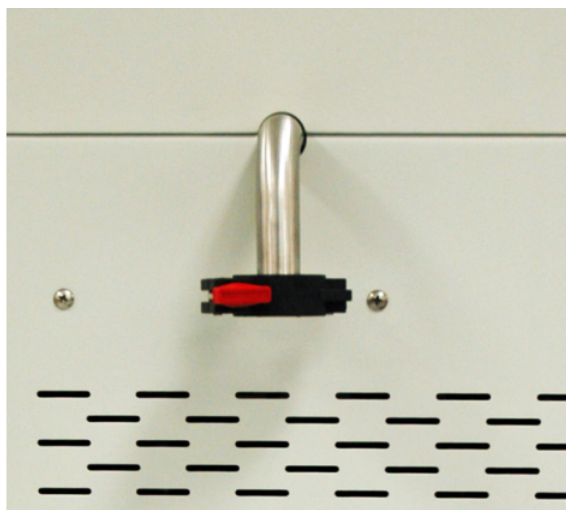
Vacuum Pump Port

Back Panel Components

Component	Description
Ethernet port	Use to connect the analyzer to the computer.
Power connector	(Not shown.) Located at the bottom of the back panel. Use to connection the power supply.
Power supply switch	(Not shown.) Located at the bottom of the back panel. Use to Power ON or OFF the analyzer. The switch also serves as the main breaker for the analyzer. This switch automatically powers OFF in the event of an electrical fault.
RS232 port	Use to connect a Smart VacPrep or SmartPrep degassing unit.
Vacuum pump port	Use to connect the vacuum pump.

EQUIPMENT OPTIONS AND UPGRADES

Parts and accessories can be found online at www.Micromeritics.com.

Equipment Option	Description
Chiller Dewar	A closed loop recirculating system that utilizes a high surface area copper coil to provide excellent heat transfer between the dewar and the recirculating liquids. Log in to your customer portal to access the Chiller Dewar Quick Start Guide (part number 025-42801-00).
Krypton Option	A low surface area model includes the addition of a 10 mmHg trans-ducer and permits accurate measurement of very low surface area on materials such as API (active pharmaceutical ingredient), powdered metals, etc.
Micropore Option	The micropore model includes the addition of a micropore transducer which extends the low pressure measurement capabilities and allows enhanced performance for characterizing microporous materials using nitrogen, argon, carbon dioxide, hydrogen, and other fixed gases. The micropore transducer also increases pressure resolution in the range necessary for micropore analysis.

DEGASSER OPTIONS

Parts and accessories can be found online at www.Micromeritics.com.

Degasser Option	Description
FlowPrep	The FlowPrep applies both heat and a stream of inert gas to the sample to remove adsorbed contaminants from the surface and pores in preparation for analysis for up to six samples. Choose the temperature, gas, and flow rate best suited for your sample material. The FlowPrep is an independent unit and not controlled by the analyzer.
Smart VacPrep	The Smart VacPrep prepares samples by heating and evacuation. It contains six sample ports in which up to five temperatures, ramp rates, and soak times per sample are individually controlled by the analyzer program so that all degas information is integrated into the sample data file for future reference. Samples can also be prepared, started, and completed independently. There is no need to wait for samples on other ports to finish. Front panel buttons allow a QuickStart operation with preprogrammed conditions.

Up to three additional Smart VacPrep degassers can be connected to one computer permitting 24 preparation ports to be used. The Smart VacPrep is the recommended degassing unit. |
| VacPrep | The VacPrep offers two methods for removing contaminants. In addition to flowing gas, it provides vacuum to prepare samples by heating and evacuation of up to six samples. This combination provides preparation method options best suited to your material or application. Needle valves are also provided for introducing the vacuum slowly to prevent fluidization of samples. The VacPrep is an independent unit and not controlled by the analyzer. |

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GAS REQUIREMENTS

Compressed gases are required for analyses. Gas cylinders or an outlet from a central source should be located near the analyzer. Up to five different non-reactive adsorptives — for example, N_2, Ar, CO_2, and Kr, plus helium for free space — can be attached to the analyzer simultaneously.

Appropriate two-stage regulators which have been leak-checked and specially cleaned are required. Pressure relief valves should be set to no more than 30 psig (200 kPag). All gases should be of a purity of 99.999% or better. Gas regulators can be ordered from Micromeritics. Parts and accessories can be found online at www.Micromeritics.com.

(CGA 580) N_2	99.999%
(CGA 580) He	99.999%
(CGA 580) Kr	99.995% (Krypton needed for krypton units only)

CRYOGEN REQUIREMENTS

Liquid nitrogen is used as the cryogen to cool the sample during analysis. A liquid nitrogen transfer system eliminates the need to pressurize storage dewars. The Model 021 liquid nitrogen transfer system is available from Micromeritics. Log in to your customer portal to access parts and accessories.

SPECIFICATIONS FOR *ASAP 2460*

Specification	Description
Electrical	
Frequency	50 or 60Hz
Power	800 VA, exclusive of vacuum pumps, which are powered separately
Voltage	100/115/230 VAC (±10%)
Environment	
Humidity	Up to 90% (non-condensing) for instrument
Temperature	10 to 30 °C, operating -10 to 55 °C, storing or shipping
Capacity	
Analysis System	2, 4, or 6 sample ports (-1 for krypton), each with a constantly monitored saturation pressure port
Analysis System	
Manifold Temperature Transducer	**Type**. Platinum resistance device (RTD) **Accuracy.** ±0.10 ºC by keyboard entry **Stability.** ±0.10 ºC per month
Manifold Pressure Transducer(s)	**Range**: • 0 to 950 mmHg operating: 1000 mmHg maximum • 0 to 10 mmHg added for krypton option **Resolution**: • 1000 mmHg Transducer: 0.001 mmHg • 10 mmHg Transducer *: 0.00001 mmHg • Micropore Transducer**: 0.000001 mmHg × full scale **Accuracy**: • 1000 mmHg Transducer: within 0.15% of reading • 10 mmHg Transducer *: within 0.15% • Micropore Transducer**: within 0.12% of reading Includes nonlinearity, hysteresis, and non-repeatability * Active only when running krypton samples ** Presented only in the enhanced micropore option

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Specification	Description
Sample Port Transducers and P$_0$ Port Transducers	**Range.** 0 to 950 mmHg **Resolution.** 0.001 mmHg **Accuracy.** ±0.1% full scale
Vacuum Transducer	**Type.** Thermocouple **Range.** 0.001 to mmHg
Vacuum System	
Pumps	**Nitrogen.** Oil-sealed pump **Krypton.** MP high-vacuum pump **Oil-free and high vacuum pump.** 3.8×10^{-9} ultimate vacuum * * Ultimate vacuum measured by pump manufacturer according to Pneurop Standard 5608
Physical	
Master Module	Height 94 cm (37 in.) Width 38 cm (15 in.) Depth 59 cm (23 in.) Weight 54 kg (119 lbs)
Auxiliary Module	Height 94 cm (37 in.) Width 38 cm (15 in.) Depth 39.5 cm (15.5 in.) Weight 29 kg (64 lb)
Computer Requirements	

- **Operating System.** Windows 7 Professional or higher operating system is recommended for the best user experience. For 21CFR11 environments, Windows 10 Professional or Windows 10 Enterprise or higher is required.
- **Desktop Installation Required.** The application should not be installed on a network drive with shared access. Multiple users cannot operate the application at the same time.
- **10 Base T or 100 Base T Ethernet Port.** If the computer is to be connected to a network, two Ethernet ports are required. If more than one Ethernet based unit is connected to the same computer (or if a Smart VacPrep is purchased), an Ethernet switch will also be required.
- **Read / Write Permissions.** All users of the application will need Read/Write permission to all directories and subdirectories where the application is installed. For 21CFR11 environments, permission may be limited to the installation directory.
- **Drives.** CD-ROM drive and USB port.

In keeping with a policy of ongoing product improvement, specifications are subject to change without notice.

2 ABOUT THE SOFTWARE

> **See also:**
>
> *About the Software in 21CFR11 Environments on page 2 - 30* for CFR11 environments.

The analyzer allows other computer programs to run while an automatic operation is in progress. The *Help* menu provides access to the online operator manual.

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

The MicroActive feature offers a Windows interface with an easy way to collect, organize, archive, reduce raw data, and store sample files for later use. Scalable and editable graphs, and copy and paste graphics, are easily generated. Customized reports can be viewed on a computer monitor, printed, or exported for use in other programs.

In addition to customizable standard reports, user defined calculations and reports can be created through the Advanced reports feature (using Python).

Data can be manipulated and displayed interactively using MicroActive reports.

SOFTWARE SETUP

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

The *Setup* program is located on the installation media and is used to reinstall the software and make analyzer changes — such as adding or removing a unit, etc.

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

ANALYSIS MODES

The analysis program supports four analysis modes:

- **Standard.** See *Perform a Sample Analysis on page 6 - 13*.

  In *Standard* mode, the system performs nitrogen, or similar gas, analyses at 77 K. All analyses must use the same gas. Samples can be removed and added to any of the ports without disturbing the analyses being performed on other ports.

- **High Throughput.** See *Perform a High Throughput Analysis on page 6 - 16*.

  In *High Throughput* mode, up to six nitrogen, or similar gas, analyses are started together. All analyses must use the same analysis gas and the same Psat gas, which may be different from the analysis gas. Once a set is started, no other samples can be started until the set is complete. The data collection is done in parallel. This mode also supports measured freespace.

- **Krypton.** See *Perform a Krypton Analysis on page 6 - 17*.

  In *Krypton* mode, the analyses are started together. Port 2, 4, or 6 (depending on system configuration) is used to store Krypton for dosing. The data collection is done sequentially — one analysis starts and completes before the next is started.

- **Micropore.** See *Perform a Micropore Analysis on page 6 - 20*.

  In *Micropore* mode, up to six analyses are started together. Once a set is started, no other samples can be started until the set is complete.

Each analysis port is equipped with an elevator that raises and lowers the analysis bath fluid dewar automatically. A removable shield to enclose the dewar should be used for safety purposes.

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

The use of isothermal jackets maintains a stable thermal profile along the full length of the sample and Psat tubes.

MENU STRUCTURE

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

Main Menu Bar Options

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

COMMON FIELDS AND BUTTONS

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

Common Fields and Buttons

Field or Button	Description
Add	Add an item to the list.
Add Log Entry	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Append	Use to insert one row at the end of a table.
Autoscale	When enabled on report parameters windows, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y-ranges will be set so that all the data is shown. If *Autoscale* is not selected, the entered range is used.
Axis Range	On report parameters windows, the *From* / *To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and/or y-axes.
Bar Code *(default field label name)*	Go to *Options > Default Method* to rename this field to something suitable for your environment. A bar code reader may be used to enter text into many of the fields on the *Sample Description* window. The bar code reader must be connected to the computer's USB port. To use a bar code reader, use a mouse to click in the field first where information is to be entered then scan the bar code with the bar code reader. If a bar code reader is not used, this alphanumeric field can be used to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Browse	Searches for a file.
Cancel	Discards any changes or cancels the current process.
Clear	Use to clear the table entries and display only one default value.
Close	Closes the active window.
Close All	Closes all active windows. If changes were made and not yet saved, a prompt displays for each changed file providing the option to save the file.

Common Fields and Buttons (continued)

Field or Button	Description
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Copies	Select the number of copies to print. This field is only enabled when *Print* is selected.
Delete	When working with pressure tables and pressures, **Delete** removes the selected information.
Destination	Select the report destination.
Edit	When working with report parameters, highlight the item in the *Selected Reports* list box and click **Edit** to modify the report details.
Exit	Exits the application. If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the application without saving the changes.
Export	Exports data in a sample file as a .REP, .TXT, or .XLS file. When saved to a file, the data can be imported into other applications.
File	Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
File name	Select a file name from the list shown or enter a file name. If the required file type is not shown, select the type of file from the list.
From / To	When working with report parameters windows, enter the *From* and *To* range for x- and/or y-axes.
Insert	Use to insert one row above the selected row in the table.
List	Creates a list of sample or other type files. The list will contain file name, date / time the file was created or last edited, file identification, and file status.
Name	The *Name* column is a list of files in the selected directory or library.
Next	Click to move to the next window or next step.
OK	Saves and closes the active window.
Open	Opens the selected file. Alternatively, double click the file name in the *Name* column to open the file.
Prev	Click to move to the previous window.
Preview	Previews predefined reports. Click the tabs at the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled.

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Common Fields and Buttons (continued)

Field or Button	Description
Print	Sends the report to the selected destination (screen, printer, or file).
Remove	Remove the selected file or files from the list.
Replace	Click to select another file where the values will replace the current file's values.
Replace All	Click to select another .SMP file where the values will replace all values for the active sample file. The original file will remain unchanged.
Report	Click to display a window to specify report output options.
Save	Saves changes to the active window.
Save As	Saves a file in the active window under a different file name.
Start	Starts the report, test, analysis, or operation.
Start Date	Displays a calendar to select the start date for the report.
View	**Operation**. Use to display the data from the current analysis. **Instrument Log**. Use to display recent analyses, calibrations, errors, or messages. **Instrument Schematic**. Use to display a schematic of the analyzer system.

FILE STATUS, DESCRIPTION, EXTENSION, AND LOCATION

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

File Status

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

File Type, Extension, and Location

File Type	File Name Extension	Default Location
Alpha-s Curve *	.ALS	Param directory
Adsorptive Properties	.ADP	Param directory
Analysis Conditions	.ANC	Param directory
Degas Conditions	.DEG	Param directory
Heat of Adsorption Report	.HOA	Param directory
Methods	.MTH	Param directory
Report Options	.RPO	Param directory
Sample Information	.SMP	Data directory
Sample Tube Properties	.STB	Param directory
Thickness Curve **	.THK	Param directory

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* **Alpha-S Curve file.** Saves the relative pressures and resulting quantities adsorbed as an ASCII text file. These data are derived by dividing the isotherm by the quantity adsorbed at 0.4 relative pressure.

** **Thickness Curve file.** Saves the relative pressures and corresponding thicknesses as an ASCII text file. These data are derived by dividing the condensed volume of adsorptive by the selected surface area. The density conversion factor in the adsorptive properties file is used to convert quantity adsorbed to volume of condensed adsorptive.

File types available when printing or exporting reports:	
Portable document format	.PDF
Report	.REP
Spreadsheet	.XLS
Unicode	.TXT
Extensible markup language	.XML

KEYBOARD SHORTCUTS

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

Certain menus or functions can also be accessed using the **Alt** key plus the underlined letter in the menu command. For example:

- to access the *File* menu, press **Alt + F**, then press the underlined letter on the submenu
- **Alt + F** opens the *File* menu, then press **O** to access the *File Selector*

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

Keyboard Shortcuts

Field or Button	Description
Alt +[Unit n]	Opens the *Unit [n]* menu.
Alt + F	Opens the *File* menu.
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Alt + H	Opens the *Help* menu.
Alt + I	Opens the *Options* menu.
Alt + R	Opens the *Reports* menu.
Alt + V	Opens the *Smart VacPrep* menu.
Alt + W	Opens the *Window* menu.
Shift + F9	Opens the shortcut menu of (1) selected component on analyzer schematic when manual control is enabled or (2) onscreen reports.
Ctrl + N	Opens a new sample file.
Ctrl + O	Opens the *File Selector* window.
Ctrl + P	Opens the *File Selector* to start a report from a selected .SMP file.
Ctrl + S	Saves the open file.
F1	Opens the online help operator manual.
F2	Opens the *File Selector* window.
F3	When in the *File Selector* window, opens the file search box.
F4	When in the *File Selector* window, opens the address bar.

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Keyboard Shortcuts (continued)

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

OPTION PRESENTATION

Options > Option Presentation

> **CFR Note** For 21CFR11 environments, see *Option Presentation for 21CFR11 Environments on page 2 - 13*.

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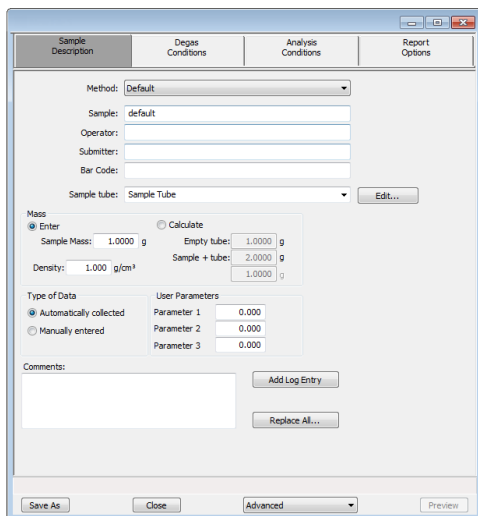
Option Presentation Display

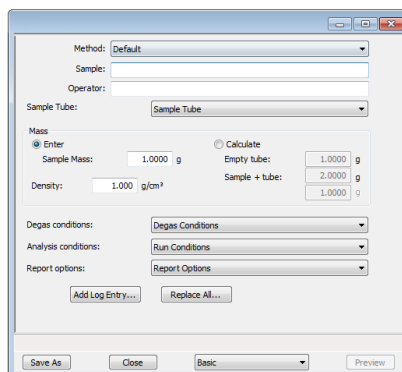| Presentation Display	Description
Advanced	Displays all parts of sample and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Restricted	Displays the sample file in a single window similar to the *Basic* display option with certain functions disabled. A password is set when the *Restricted* option is selected. That same password must be entered to change to the *Basic* or *Advanced* display option. This display type is typically used in laboratories where analysis conditions must remain constant — such as the pharmaceutical industry. The *Advanced* option is not available at the bottom of the window when using the *Restricted* display option.
Show Splash Screen	Enables (or disables) the splash screen upon application startup.
Show Degas Conditions	When enabled, displays the *Degas Conditions* tab when using *Advanced* option presentation and the Degas Conditions drop-down list when using *Basic* or *Restricted* option presentation. This option may be deselected to hide the *Degas Conditions* tab if not using a SmartPrep or Smart VacPrep.

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

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

![Micromeritics logo](mi micromeritics®)

**Advanced
option presentation**

**Basic / Restricted
option presentation**

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

OPTION PRESENTATION FOR 21CFR11 ENVIRONMENTS

Options > Option Presentation

Option Presentation Display

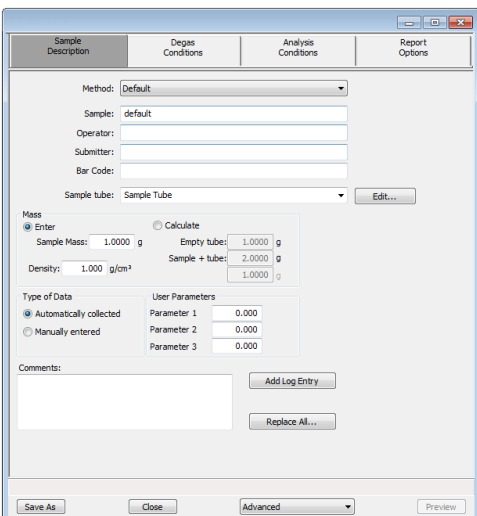| Presentation Display	Description
Show Degas Conditions	When enabled, displays the *Degas Conditions* tab when using *Advanced* option presentation and the Degas Conditions drop-down list when using *Basic* option presentation. This option may be deselected to hide the *Degas Conditions* tab if not using a SmartPrep or Smart VacPrep.
Show Splash Screen	Enables (or disables) the splash screen upon application startup.

CFR Note **For members of the Developer group only.** To change the view from *Advanced* (for Developers) to *Basic* (for Analysts), click the drop-down arrow at the bottom of the window. Select either *Advanced* (when in *Basic* view) or *Basic* (when in *Advanced* view).
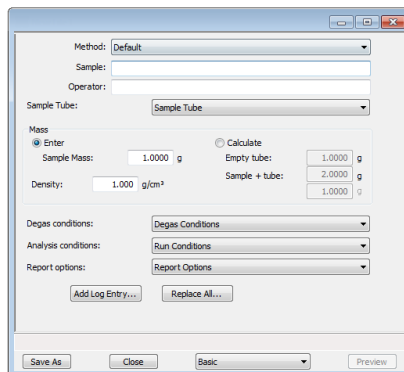
Option Presentation Display

Presentation Display	Description
Advanced	Displays all parts of sample and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	This display option is used after the parameter files have been created by Developer group members. The previously entered or default parameter files are then accessible using drop-down lists.

The following examples show the same sample file in *Advanced* and *Basic* display.

![micromeritics logo]

**Advanced view
for the Developer group**

**Basic view
for the Analyst group**

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

The **Save** button is disabled on sample files with a *Complete* status. When the **Preview** button is used to view reports for sample files with an unsaved status, the report will have a *Preview* watermark. The **Save As** and **Print** buttons on the report window are also disabled.

LIBRARIES

Options > Manage Libraries

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

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

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

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

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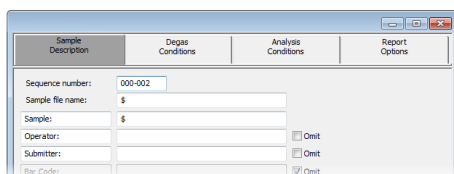
METHODS

Options > Default Method

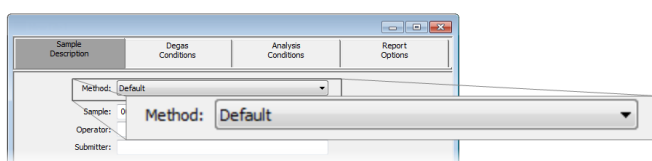## *File > Open > [.MTH File]*

> **CFR Note**
>
> For 21CFR11 environments, this section is applicable only to members of the Developer group, however, members of the Analyst group may find information in this section helpful. Sample file information that is available to Analysts is created by a member in the Developer group using information in this section.

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

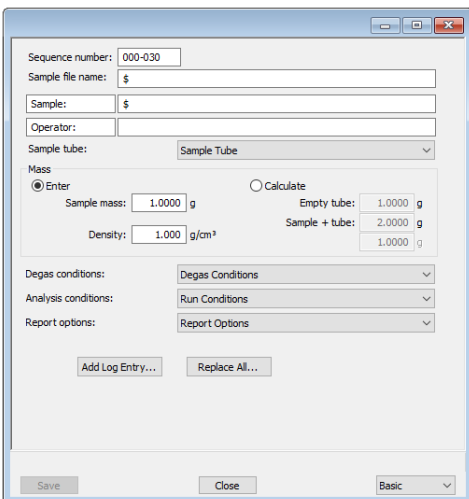The *Method* drop-down list displays only those methods applicable to the open sample file type.

Default Method

Sample Information File

Developer group view in a 21CFR11 environment

Analyst group view in a 21CFR11 environment

Default Methods

Field or Button	Description
Sequence Number *[text box]*	Specify a default numeric string to be used as a prefix in the *Sample* field when a new sample file is created. This number increments with each sample file created.
Sample file name *[text box]*	Enter a format for the sample identification. The entry in this field becomes a part of the saved sample file name. Include the $ symbol to have the sample file number included as part of the identification.
Sample Operator Submitter Bar Code *[text box]*	These field labels may be renamed and the new label becomes a part of all new sample files.

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CONFIGURE THE ANALYZER

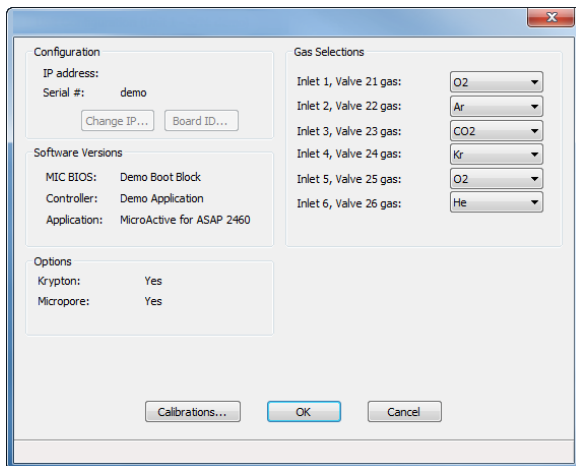### UNIT CONFIGURATION

Unit [n] > Unit Configuration

> **CFR Note** In 21CFR11 environments, this feature is applicable to members of the Developer group only.

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

The gases connected to the inlets must be specified in the analysis program. If the gas is changed on one of the inlets, the same change must be made on the *Unit Configuration* window.

Unit Configuration

| Field or Button	Description
Calibrations *[button]*	Displays calibration information for analyzer components.
Configuration *[group box]*	Displays the IP address used by the analysis program, serial number, and type of analyzer. **IP address.** Displays the IP address of the analyzer. **Change IP** *[button].* Click to display the *Unit IP Setup* window. Use to change the IP address and subnet mask assigned during installation. Do not edit these fields unless instructed by a Micromeritics Service Representative.

Unit Configuration (continued)

Field or Button	Description
	Board ID *[button]*. Click to display information from the electronic circuit boards in the analyzer. These parameters cannot be edited.
Gas Selections *[group box]*	Displays ports for gas selections.
Software Versions *[group box]*	Displays the software versions of the MIC BIOS, controller, and analysis program.

UNIT SELECTIONS

Options > Units

Use to specify how data should appear on the application windows and reports. This menu option is not available if using *Restricted* option presentation in a standard installation environment.
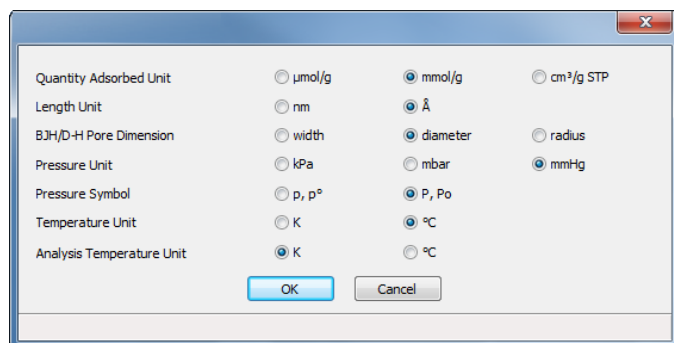
INSTRUMENT STATUS

SHOW DASHBOARD

Unit [n] > Show Dashboard

The dashboard displays the following:

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

Data for the dashboard comes from the logged diagnostic data. The dashboard is automatically kept current as the relevant diagnostic data items are updated. The gauges will be updated even if the dashboard window is not open.
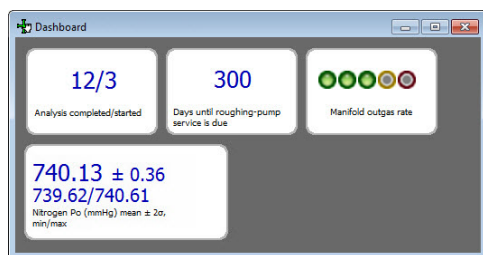
> Red numbers on the dashboard require attention. To reset the dashboard numbers, right click on the dashboard setting, then click **Reset**.

Dashboard Gauges

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

Dashboard Gauges (continued)

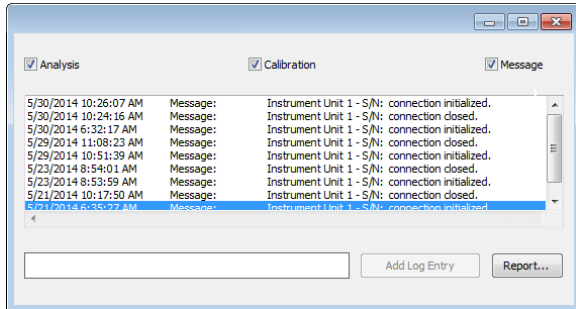| Field or Button	Description
Manifold outgas rate	Provides the qualitative indication of the outgas rate in the dosing manifold. LED images constitute a bidirectional bar graph of the outgas rate. The gauge is updated when the *Analysis Manifold Test* is run. See ***Start Diagnostic Test on page 9 - 1***. • Three green LEDs are lit if outgas rate is below 30% of outgas rate limit. • At 30%, the left LED turns off. • At 60%, the center LED turns off. • At 90%, three green LED lights turn off and the center yellow LED turns on. • At 110% and above, only the red LED turns on and attention is required.
Nitrogen P_0	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 P_0 is logged with nitrogen as the adsorptive and a bath temperature of 77±2 K.

SHOW INSTRUMENT LOG

Unit [n] > Show Instrument Log

CFR Note In 21CFR11 environments, see *System Audit Trail on page 2 - 32*.

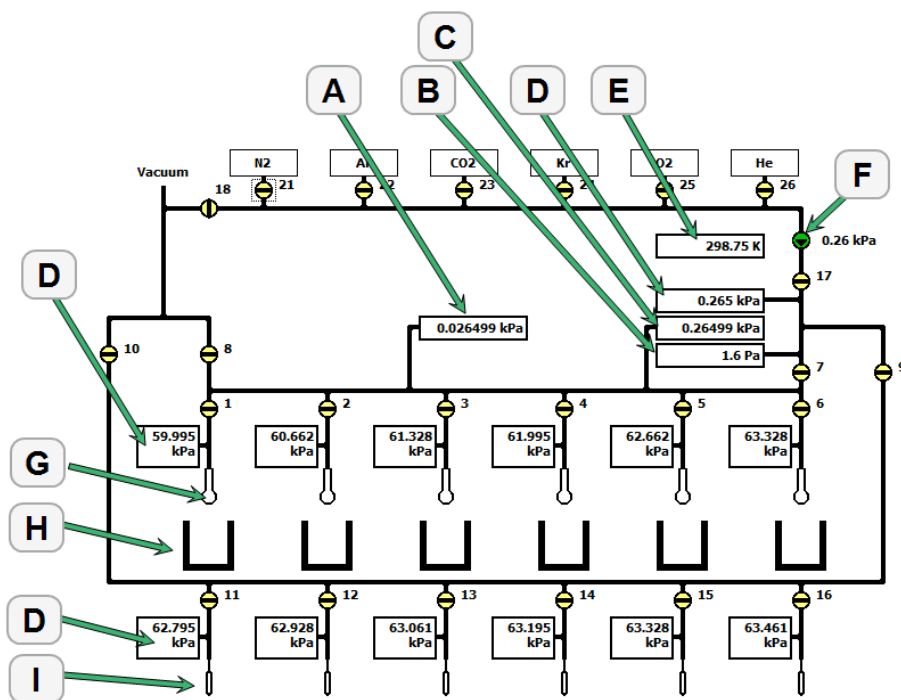Use to display a log of recent analyses, calibrations, errors, or messages.

Instrument Log

Field	Description
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.
Analysis/ Calibration/ Message *[check box]*	Select the logs to display.
Report *[button]*	Click to select print destination and report start date.

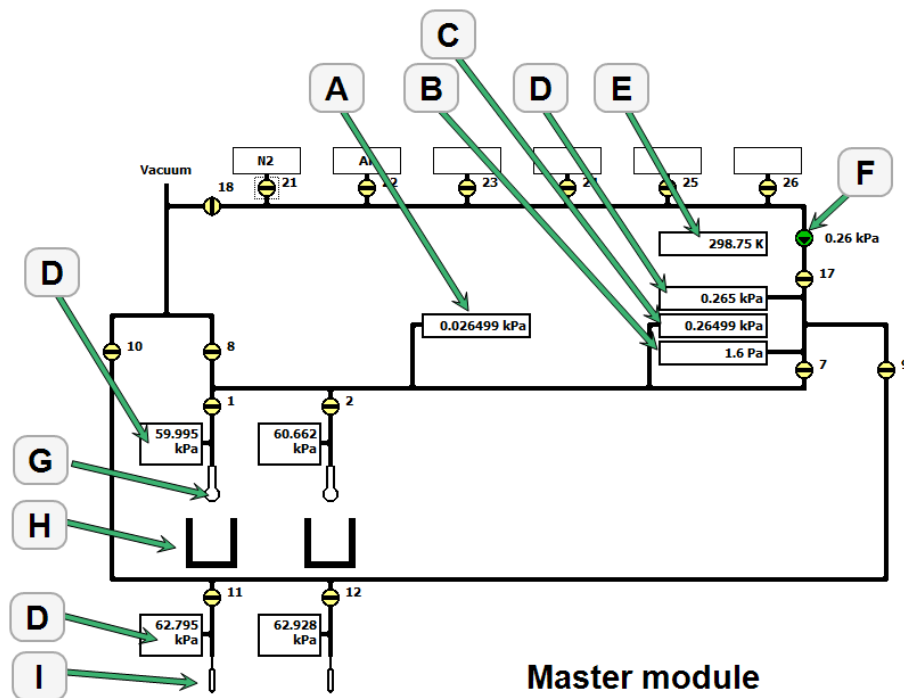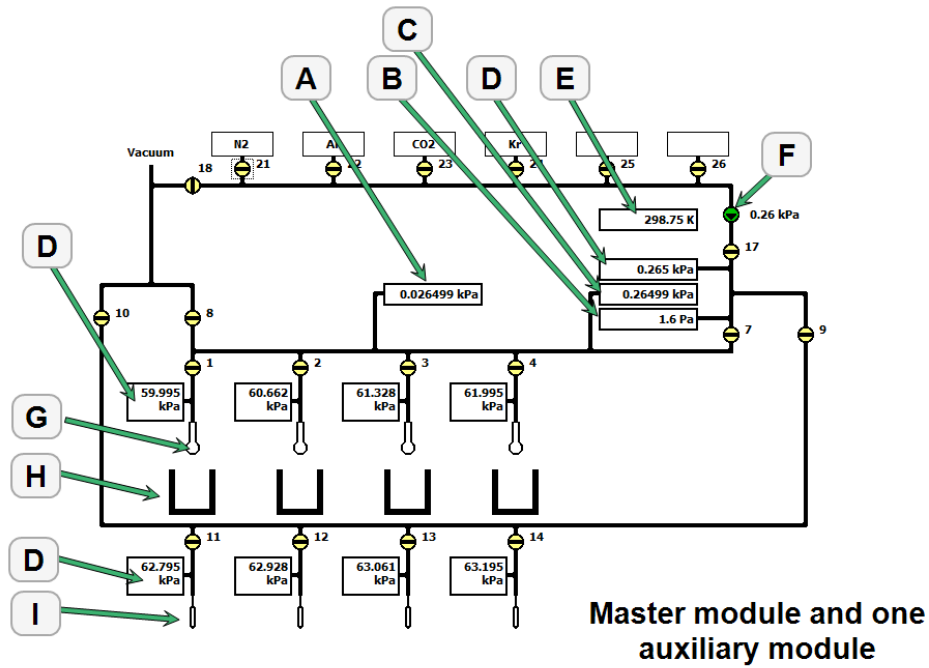**For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

SHOW INSTRUMENT SCHEMATIC

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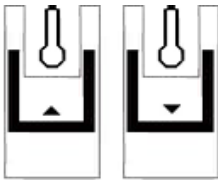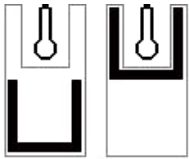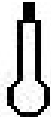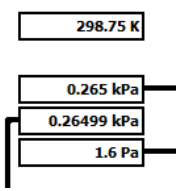
Use to display an analyzer schematic. To operate the valves and elevator from this window, manual control must be enabled (**Unit [n] > Enable Manual Control**).
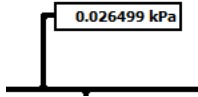
Master module and two auxiliary modules

Master module and one
auxiliary module

Master module

Analyzer Schematic Icon Table

Icon or Symbol	Description
	Open Valve. Green indicates an open valve.
	Closed Valve. Yellow indicates a closed valve. When manual control is disabled, closed valves appear white.
	Servo Valve. Closed.
	Servo Valve. Open.
	Elevator.
	The arrow inside the dewar icon indicates the direction of dewar movement.
	The elevator icon indicates the position of the dewar.
	Sample Tube. Cannot be manually controlled.
	P_0 **(Psat) tube.**
Transducers	Each sample port and P_0 port contains a 1000 mmHg transducer. The transducer readings display next to the ports.
	298.75 K — Displays the temperature, the 1000 mmHg, 10 mmHg transducer readings, and vacuum gauge pressure.
	0.265 kPa / 0.26499 kPa / 1.6 Pa — The 10 mmHg transducer (for krypton or Micropore installations).

Analyzer Schematic Icon Table (continued)
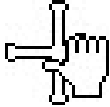
Icon or Symbol	Description
	0.026499 kPa — Displays the micropore transducer reading. This transducer is optional and is shown only if installed.

Analysis Valve Descriptions

Valve	Description
1 through 6	Sample ports
7	Lower manifold isolation
8	Sample ports unrestricted vacuum
9	P_0 ports access
10	P_0 ports unrestricted vacuum
11 through 16	P_0 ports
17	Upper manifold isolation
18	Gas inlets unrestricted vacuum
21 through 26	Gas inlet port valves
Unmarked	Servo
A	Micropore transducer
B	Vacuum gauge
C	10 mmHg transducer
D	1000 mmHg transducer
E	Analysis manifold temperature
F	Servo valve
G	Sample tube
H	Elevator
I	P_0 tube

Instrument Schematic Shortcut Menus

Schematic Shortcuts

Schematic Shortcut Icon	Description
Valve options	**Close.** Closes the selected valve.
	Open. Opens the selected valve.
	Pulse. Use to quickly turn the valve on and off allowing the operation to proceed in small increments.
	For Servo valve:
	• **Set.** Use to set the servo valve target pressure and dose or evacuate.
	• **Close.** Closes the servo valve. The valve symbol changes to solid black.
Elevator options	Right click the elevator icon, then select:
	Raise. Select *Raise* to raise the elevator. When it is moving, press the keyboard space bar to stop the movement (or right click and select *Stop* from the menu).
	Lower. Select *Lower* to lower the elevator. When it is moving, press the keyboard space bar to stop the movement (or right click and select *Stop* from the menu).
	Stop. Stops the elevator from moving.

SHOW STATUS

Unit [n] > Show Analysis Status

Use to show the current status for each port.

CONVERT FILES

File > Convert > [*.MGD file]

- Converts sample files created with an ASAP 2400, ASAP 2405, or ASAP 2420 system to files compatible with the ASAP 2460 system.
- Converts StarDriver files (.MGD extension) to a sample file with a .SMP file extension. Only those files with a .MGD file extension display in the *Name* column.

EXPORT FILES

File > Export

See also:

Exported Data Example on page C - 1

Provides the option to print the contents of one or more sample or parameter files to either the screen, a printer, or to a file. Data can be exported as a .PDF, .REP, .TXT, .XML, 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 these file formats.

LIST FILES

File > List

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

Select one or more files from the file selector, then click **List**.

No.	File Name	Date	Time	Description	Status
			File Listing		
1	co_pulse.smp	9/26/2017	5:12:26 PM	CO pulse chemisorption on 0.5 Pt Alumina	Complete
2	h2_o2_h2.smp	9/26/2017	5:12:26 PM	H2 O2 H2 titration on Pt Alumina	Complete
3	mfi-fit2.smp	9/26/2017	5:12:27 PM	Heat of desorption - fit peaks - strong chemi	Complete
4	mfi-fit1.smp	9/26/2017	5:12:26 PM	Heat of desorption - fit peaks - weak sites	Complete
5	mfi-hod1.smp	9/26/2017	5:12:27 PM	NH3 - heat of desoprtion - weak sites	Complete
6	mfi-hod2.smp	9/26/2017	5:12:27 PM	NH3 - Heat of desorption - strong chemisorption	Complete
7	mfi-01.smp	9/26/2017	5:12:26 PM	NH3 TPD	Complete
8	mfi-fit.smp	9/26/2017	5:12:26 PM	NH3 TPD - with peak deconvolution	Complete
9	bet.smp	9/26/2017	5:12:26 PM	Silica Alumina BET 2X	Complete
10	000-193.SMP	9/26/2017	11:28:12 AM	Silver Oxide TPR	Complete
11	ago_tpr.smp	9/26/2017	5:12:26 PM	Silver oxide TPR	Complete
12	n2o_puls.smp	9/26/2017	5:12:27 PM	Silver surface area using N2O	Complete
13	v2o5.smp	9/26/2017	5:12:27 PM	TPR of Vanadia	Complete

ABOUT THE SOFTWARE IN 21CFR11 ENVIRONMENTS

> **See also:**
>
> *Option Presentation for 21CFR11 Environments on page 2 - 13*

> The Micromeritics Confirm applications for 21CFR11 environments require an operating system of Windows 10 Professional or Windows 10 Enterprise or higher. Management of users and groups is performed in Windows Users and Groups.

The Micromeritics Confirm application enables laboratory managers to develop analysis methods, enforce industry standards, and produce audit trails. It also enables laboratory analysts to perform analyses and produce reports.

USER PERMISSIONS

Confirm User Name	Description
mic_confirm_user	*mic_confirm_user* is the user name used by all installations. • This user should have complete control over the installation directory. • The application is launched under this user name and has this user's privileges to the windows file system. • This user should not be used by anyone or any other software that is not a Micromeritics application. • The system administrator has the option of modifying this account so that the password never expires. Alternatively, if the password does expire while the application is running, the application automatically changes the password for this account.

Confirm Group Name	Description
Developer Group	The default Developer group name is *mic_[analyzer model number]_developer*. Members of the Developer group: • have rights to all functions of the Micromeritics application - including Advanced option presentation which allows the creation and modification of methods, sample files, and parameter files.

Confirm Group Name	Description
	• can run an analysis. • can also be assigned Administrator rights which control the user pro-files.
Analyst Group	The default Analyst group name is *mic_[analyzer model number]_analyst*. Members of the Analyst group: • have access to the Basic presentation option only. • may create sample files from pre-defined methods and can modify only a limited number of input fields.

CREATE NEW FOLDER

File > Create New Folder

CFR Note	For 21CFR11 environments only.

Provides the option to create and name a new folder in the Confirm application folder. This option may not be available depending on how the IT administrator configures Windows permissions.

SYSTEM AUDIT TRAIL

CFR Note	For 21CFR11 environments only.

File > System Audit Trail
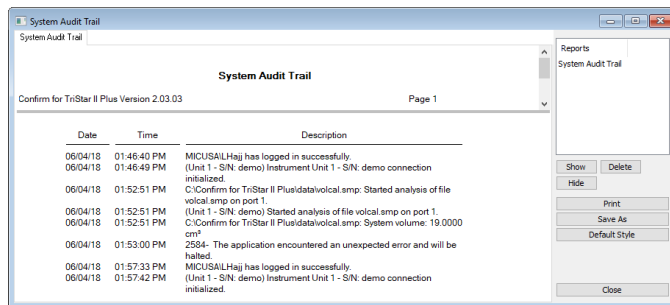
Lists the current user, successful and failed application user login attempts, and contains a description of all the changes made to sample files.

Contains an audit trail of all system initializations, user login attempts, and sample analyses.

SOFTWARE UNINSTALL

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

To uninstall the software, double click the *uninstall.exe* file located in the software installation directory, then follow the prompts.

UNINSTALL IN A 21CFR11 ENVIRONMENT

CFR Note In a 21CFR11 environment, upon uninstalling the Confirm application, the system administrator should go into Windows Users and Groups to remove the Confirm users and groups. See the Confirm Administrator Guide (part number 004-42821-01).

Depending on the network, Windows may not allow the uninstall.exe program to run. If this happens. follow these steps:

1. In Windows Users and Groups, verify that the current user is not a member of the analyst group or developer group. If so, remove the user from the group(s). Log OFF, then log back ON to the computer.
2. In Windows Explorer, in the Confirm installation directory, double-click the *uninstall.exe* file to run the uninstall program.

SOFTWARE UPDATES

Log in to your customer portal to access and download the latest analyzer software version.

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

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

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

CFR Note Existing Confirm application users and groups are not affected by software updates. Any changes to Confirm users and Confirm groups must be made using Windows Users and Groups.

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3 ABOUT SAMPLE FILES

Sample files include the information required by the analyzer to perform analyses and collect data. A sample file identifies the sample, guides the analysis, and specifies report options and may be displayed in either *Advanced*, *Basic*, or *Restricted* presentation display mode. See *Option Presentation on page 2 - 11* or *Option Presentation for 21CFR11 Environments on page 2 - 13*.

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

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

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

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

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CREATE SAMPLE FILES

File > New Sample > [.SMP File]

File > Open > [.SMP File]

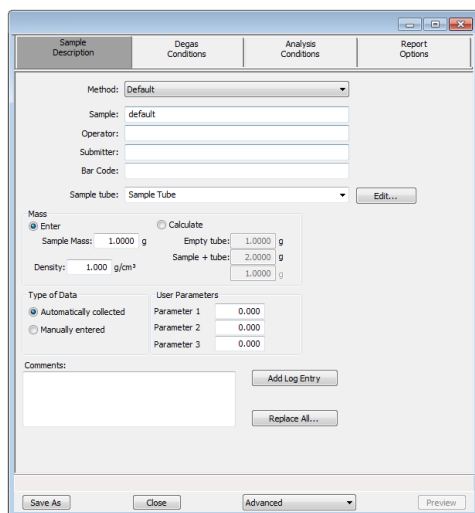|	
CFR Note	For 21CFR11 environments, this section is applicable only to members of the Developer group, however, members of the Analyst group may find information in this section helpful. Sample file information that is available to Analysts is created by a member in the Developer group using information in this section.

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

Specify or change the default display option by selecting ***Options > Option Presentation*** or select *Basic* or *Advanced* from the drop-down list at the bottom of the window.
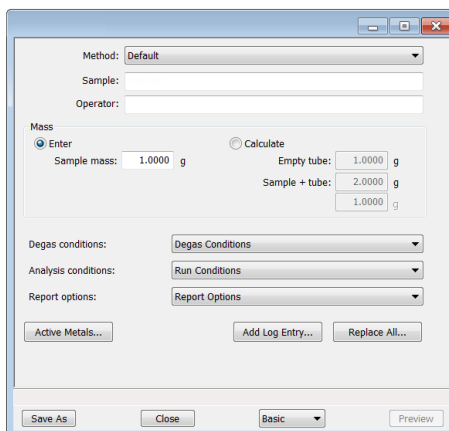
Sample files must be initially created in *Advanced* option presentation. After the sample files are saved, the files are accessible in *Basic* and *Restricted* option presentation.

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

Sample Tube parameters are edited on the *Sample Description* tab.

Example of tabbed file editor for Advanced or Developer (Cfr) view

Example of file editor for Basic or Analyst (Cfr) view

> 🖉 A bar code reader may be used to enter text into many of the fields on the *Sample Description* window. Use a mouse to click in the field first where information is to be entered then scan the bar code with the bar code reader.

Sample Files

Field or Button	Description
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.
Comments *[text box]*	Enter comments about the sample or analysis. Comments display in the report header.
Mass *[group box]*	If mass = 1, the reported surface area equals the total surface area but it is always shown as m^2/g. If an accurate mass is entered, the reported surface area is normalized per gram of sample. Choose whether to enter mass manually or have the system automatically calculate mass. Enter a value for sample mass. Mass can be changed any time before, during, or after analysis. **Enter.** Enables the *Sample mass* field. Enter a value for the sample mass. **Calculate.** Enables the *Empty tube* and *Sample + tube* fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass: $$Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$$ **Density.** Value is used for the calculated free space method only. Use 0.000 for a blank analysis.
Method *[drop-down box]*	Select a method from the drop-down list.
Operator *[text box]*	Enter operator identification information. This field label may have been renamed or may not display if modified in ***Options > Default Methods***.
Sample *[text box]*	Enter a sample description.
Sample Tube *[drop-down box]*	Select a sample tube file from the drop-down list, or click **Edit** to modify or create a new sample tube file.
Submitter *[text box]*	Enter submitter identification information. This field label may have been renamed or may not display if modified in ***Options > Default Methods***.

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Sample Files (continued)

Field or Button	Description
Type of Data *[group box]*	**Automatically collected.** Select if the type of data will be automatically collected by the system while an analysis is running. **Manually entered.** Use to enter data manually that was collected from another source. If *Manually entered* is selected, the Isotherm Report becomes available in the *Basic/Advanced* drop-down list for pasting or importing data into the file. See *Manually Enter Data on page 3 - 7*.
User Parameters *[group box]*	These fields are primarily used for the SPC (Statistical Process Control) reporting to specify sample characteristics or its manufacturing process but may be used for other data by entering specific analysis conditions or sample criteria. The entered parameters display on the *Summary Report*. Some fields may not display (or may have a different field label) if modified in the method from which the sample file was created, either through **Options > Default Method** or **File > Open > Method**. See *SPC Report on page 7 - 4*.

> **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**
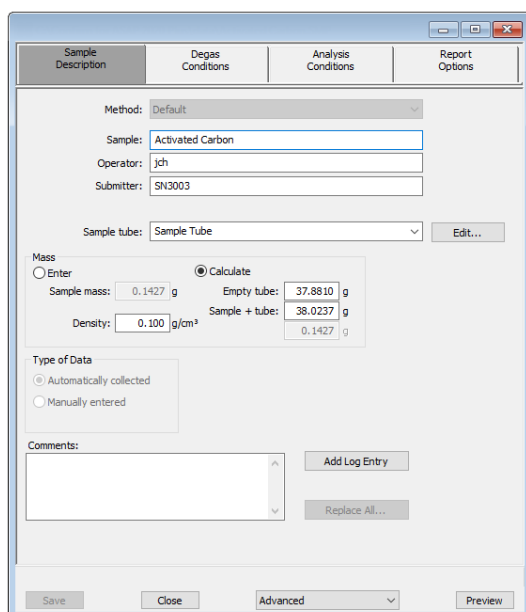
OPEN A SAMPLE FILE

File > Open > [.SMP File]

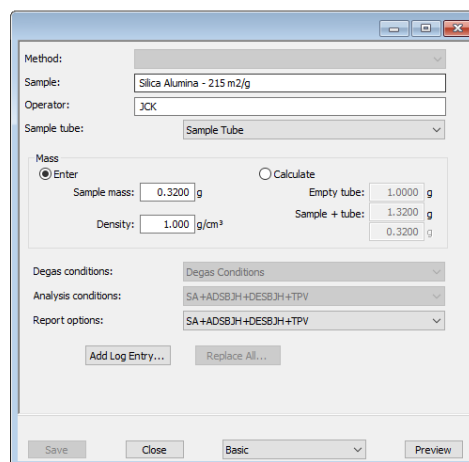> When working with an existing sample file, consider making a copy of the sample file to maintain the original configuration options.

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

Example of tabbed file editor for Advanced or Developer (Cfr) view

Example of file editor for Basic or Analyst (Cfr) view
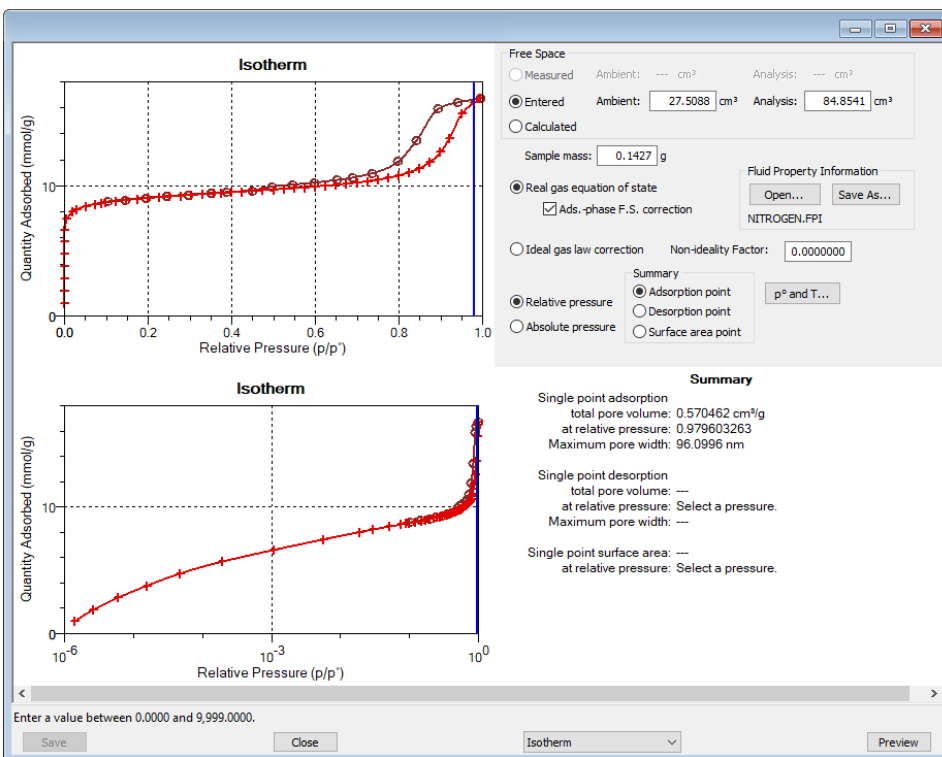
Example of a Report window

If a sample file with a *Complete* status is opened, to return to the tabbed file editor, select *Advanced* or *Basic* from the drop-down at the bottom of the window.

> **CFR Note** In 21CFR11 environments, this feature is applicable to members of the Developer group only.

MANUALLY ENTER DATA

> **CFR Note** In 21CFR11 environments, this feature is applicable to members of the Developer group only.

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

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

COPY AND PASTE MANUALLY ENTERED DATA

1. Open a sample file with a *Complete* status. The file will open to the interactive reports window.
2. Right click in the graph area of the interactive reports window, then select *Copy data*.
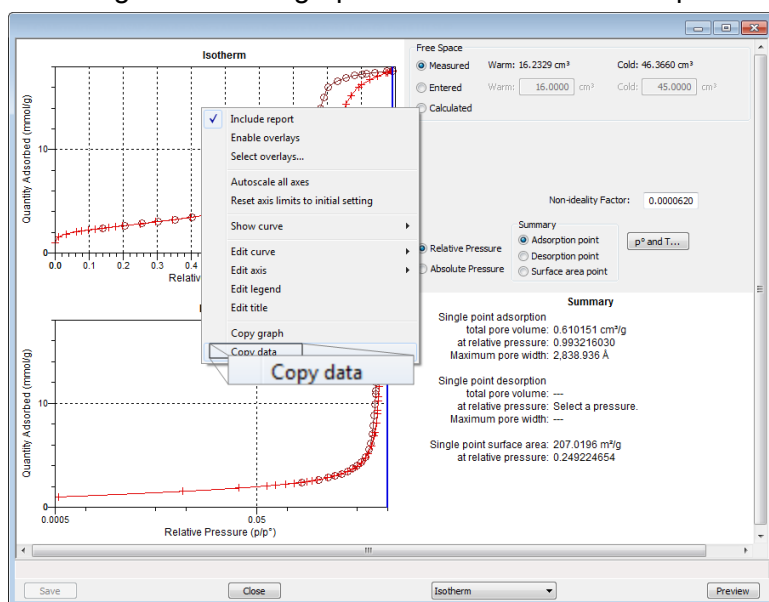
3. Open another sample file using *Advanced* option presentation.
4. On the *Sample Description* tab, select *Manually entered* in the *Type of Data* group box.
5. Click the *Advanced* down arrow at the bottom of the window, then select *Isotherm*.
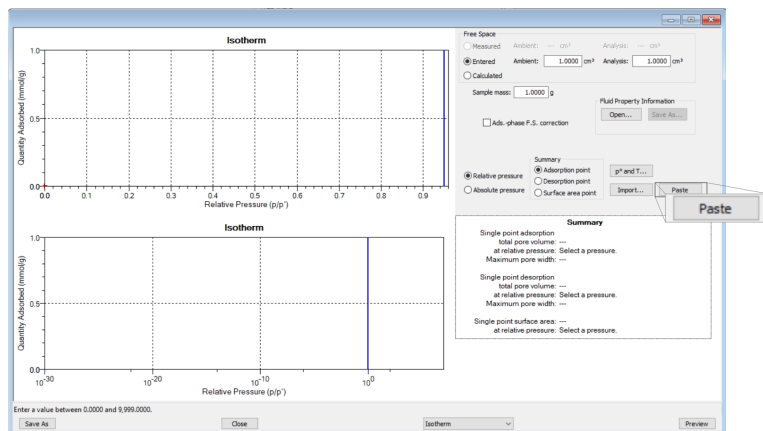
6. Ensure that all parameter fields are set appropriately, then click **Paste**.

IMPORT MANUALLY ENTERED DATA

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

ASCII text file format rules

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

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

Sample Physical Adsorption ASCII Text File

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

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

To import the ASCII text file

1. Open a new sample file in *Advanced* option presentation.
2. On the *Sample Description* tab, select *Manually entered*.
3. Click the *Advanced* down arrow at the bottom of the window, then select *Isotherm*.
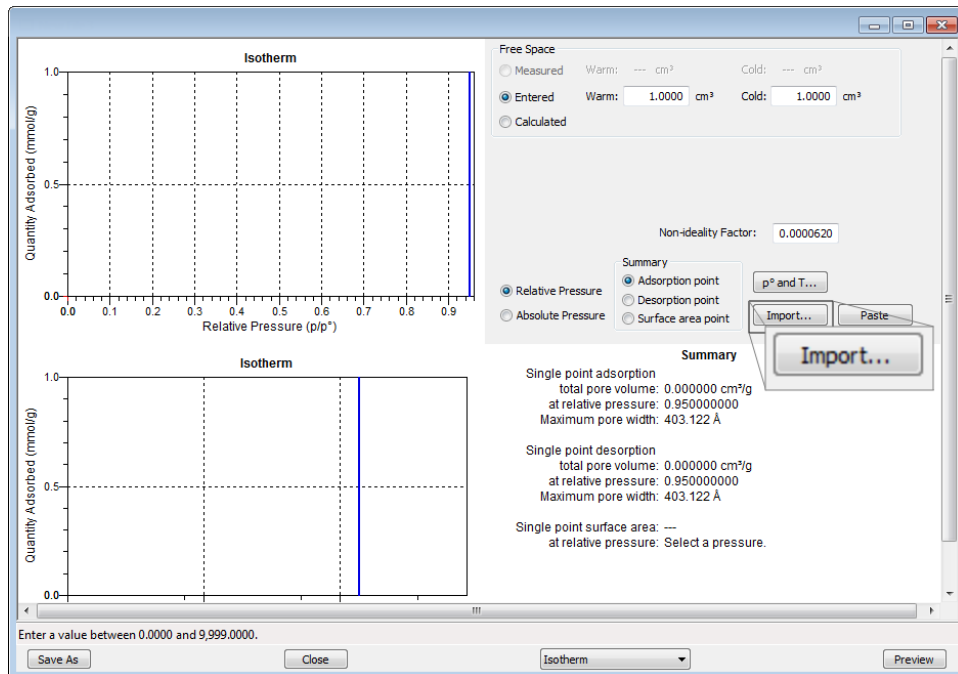
4. Ensure that all parameter fields are set appropriately, then click **Import**.
5. Open the .TXT file. The data from the original sample file is imported and displayed. If an error message appears instead, verify that the .TXT file format is correct.

4 PARAMETER FILES

> **CFR Note** In 21CFR11 environments, this section is applicable only to members of the Developer group, however, members of the Analyst group may find information in this section helpful. Parameter file information that is available to Analysts is created by a member in the Developer group using information in this section.

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

Methods include both analysis conditions and report options, offering the most convenient way to repeat most analyses.

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

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

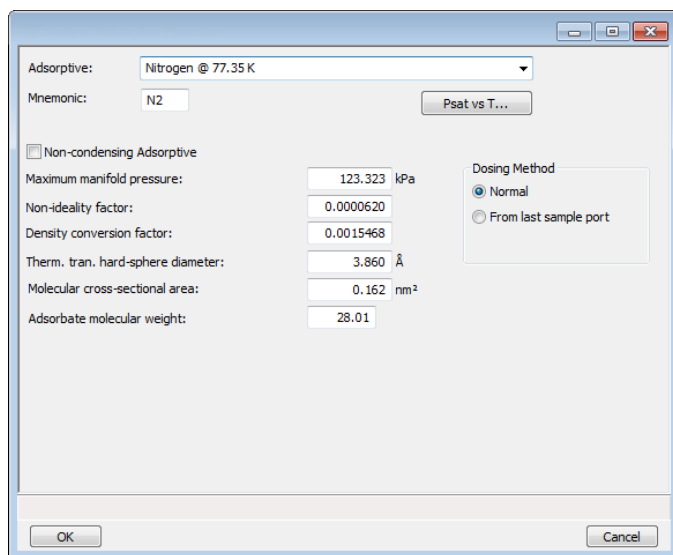**Parameter File Types**

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

![mi micromeritics® logo]

ADSORPTIVE PROPERTIES

File > Open > [.ADP File]

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

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

Adsorptive Properties

Field or Button	Description
Adsorbate molecular weight *[text box]*	The molecular mass is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot.
Adsorptive *[text box]*	Name of the adsorptive gas whose properties are being defined.
Density Conversion Factor *[text box]*	Factor determined by obtaining the ratio of the gas density (STP) to the liquid volume. This field is disabled if *Non-condensing Adsorptive* is selected.

Adsorptive Properties (continued)

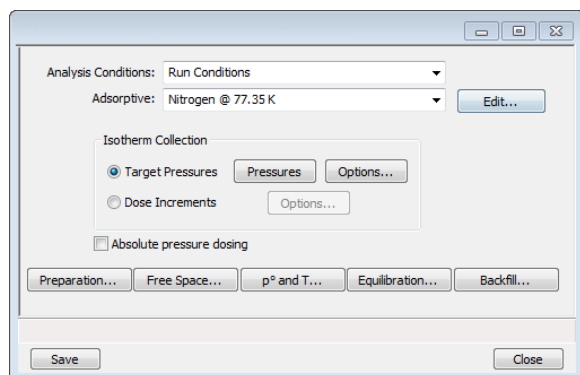| Field or Button	Description
Dosing Method *[group box]*	**Normal.** Dose from a pressurized tank of gas attached to a gas inlet port. **From last sample port.** For krypton analysis only, the krypton is stored and dosed on the last port (2, 4, or 6 depending on system configuration). • **Charge from inlet.** Use to have the tube automatically charged with condensate from a gas inlet port after the dewar is raised. • **Purify adsorptive.** Use to have the condensate in the tube purified after charging by evacuating the gas over the condensate. If *Charge from inlet* is selected, select *Purify adsorptive* to have non-condensing contaminants automatically removed from the dosing tube prior to analysis. After the adsorptive has condensed in the Psat tube or sample port, the remaining gas in the tube will be evacuated to remove noncondensing contaminants. A small amount of the purified adsorptive condensate will then return to gas phase to restore equilibrium pressure in the tube.
Maximum manifold pressure *[text box]*	The highest pressure to which the manifold will be dosed. To avoid damage to the analyzer, this number is limited to 925 mmHg. Low pressure sources will require lower numbers. For gases to be used for dosing after charging a tube from a gas inlet, enter the maximum pressure for dosing from the inlet, not from the tube of condensate.
Mnemonic *[text box]*	Enter the mnemonic name for the adsorptive. If this gas is connected to a gas inlet port, this mnemonic must be entered in the *Unit Configuration Gas Selection* for the inlet port. See *Unit Configuration on page 2 - 18*
Molecular cross-sectional area *[text box]*	The area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations.
Non-condensing Adsorptive *[check box]*	Select if using a non-condensing analysis gas. When selected, the *Density conversion factor* field and the **PSAT vs T** button are disabled.
Non-ideality factor *[text box]*	Compensates for the forces of attraction between molecules in a real gas. This value is used for a calculated free space.
Psat vs T *[button]*	Click to edit the *Psat vs Temperature* table. The table contains saturation pressures and their corresponding temperatures. To edit, click in a field and enter the value.

Adsorptive Properties (continued)

Field or Button	Description
Therm. tran. hard-sphere diameter *[text box]*	An estimate of molecular size used in calculating the thermal transpiration correction.

> **For fields and buttons not listed in this table, see** *Common Fields and Buttons on page 2 - 4*.

ANALYSIS CONDITIONS
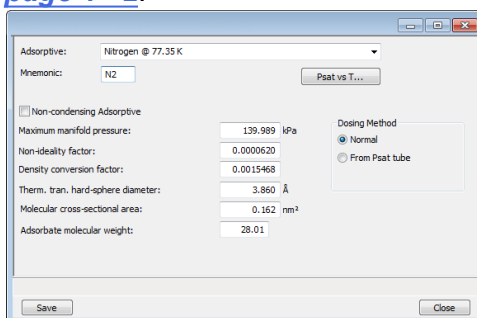
File > Open > [.ANC File]

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

Analysis conditions specify the parameters used to guide an analysis.
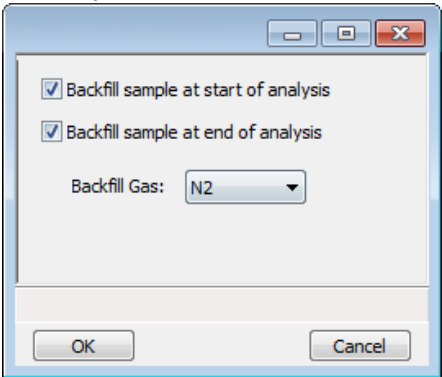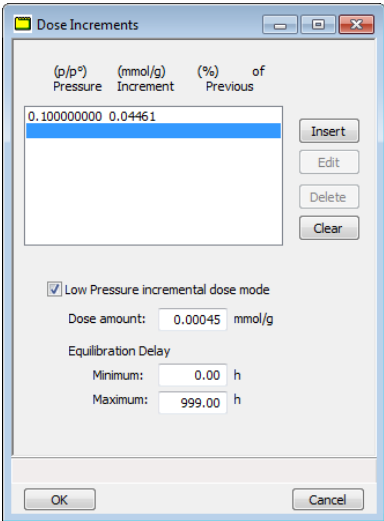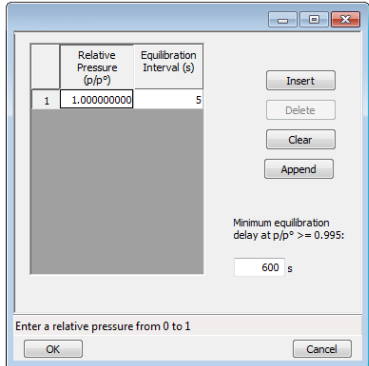

Analysis Conditions
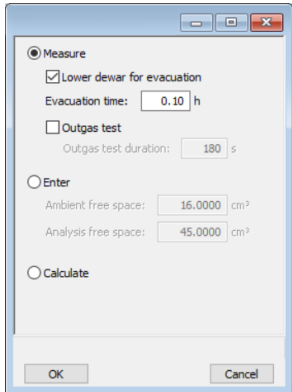
Field or Button	Description
Absolute pressure dosing [check box]	Specifies pressure targets in mmHg, mbar, or kPa instead of relative pressure. If this option is selected, the *Relative Pressure* labels and entries change to *Absolute Pressure* in the selected pressure units. This option is typically 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.
Adsorptive [drop-down box]	Select an *Adsorptive Properties* file from the list of defined gases to be used for analysis. After selection, click **Edit** to modify adsorptive properties. See *Adsorptive Properties on page 4 - 2*.

Analysis Conditions (continued)

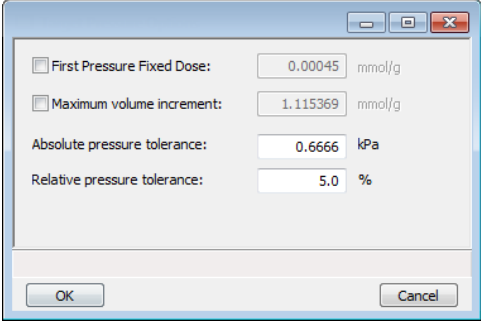| Field or Button	Description
Analysis Conditions *[drop-down box]*	Use to browse for an *Analysis Conditions* file that contains analysis condition parameters to be used in the analysis.
Backfill *[button]*	Select gas for backfilling the sample tube at the start and end of an analysis. ☑ Backfill sample at start of analysis ☑ Backfill sample at end of analysis Backfill Gas: N2 OK Cancel The options are disabled if the file status is *Entered*, *Complete*, or *Analyzing*.
Dose Increments *[selection]*	The sample is dosed repeatedly with a fixed amount of gas and isotherm points are collected after equilibrating each dose. Dose Increments (p/p°) (mmol/g) (%) of Pressure Increment Previous 0.100000000 0.04461 Insert Edit Delete Clear ☑ Low Pressure incremental dose mode Dose amount: 0.00045 mmol/g Equilibration Delay Minimum: 0.00 h Maximum: 999.00 h OK Cancel **Options** *[button].* Each entry in the table defines how much to dose for a segment of the isotherm. In this form of dosing, there is no predefined pressure table. Define target ranges for up to 10 dose increments. The ending relative pressure and the dose amount (entered or as the fraction of the previous quantity adsorbed) for each target range are shown.

Analysis Conditions (continued)

Field or Button	Description
Equilibration *[button]*	Provides options to specify the equilibration interval and delay time. **Minimum equilibration delay at p/po > = 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. **Relative Pressure (p/p^o) or Absolute Pressure.** The pressure the equilibration interval will be applied.
Free Space *[button]*	Use to enter the free space measurement type. **Measure:** • **Lower dewar for evacuation.** If the dewar is to be lowered for evacuation, select this option and enter the length of time for evacuation after the free space meas-

![micromeritics logo]

Analysis Conditions (continued)

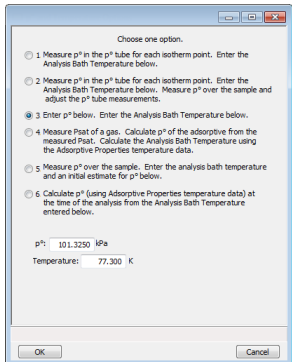| Field or Button	Description
	urement in the *Evacuation time* field. If using a cryostat, the operator must manually move the cryostat assembly when prompted.
	• **Evacuation time.** The length of evacuation time prior to free space measurement.
	• **Outgas test.** Checks for system leaks or sample outgassing. After free space is measured, the dewar is lowered and the sample evacuated for the specified amount of time. The leak test is performed after evacuation. If the pressure rises more than 0.025 mmHg within the time specified in the *Outgas test duration* field, outgassing is present. If a leak is found, the leak test repeats nine times, with 30 minutes evacuation between tests. If the 10th leak check fails, the analysis stops and the operator is notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak occurs.
	Enter. Measures free space after analysis ends. Enter the estimated free space measurements.
	• **Ambient free space.** Empty sample tube gas capacity measured at room temperature.
	• **Analysis free space.** Empty sample tube gas capacity measured with the dewar raised.
	Calculate. Use to have the free space measurement calculated using the sample and tube parameters.
Options *[button]*	Available when the *Target Pressures* option is selected. **First Pressure Fixed Dose.** This option is most frequently used when performing a

Analysis Conditions (continued)

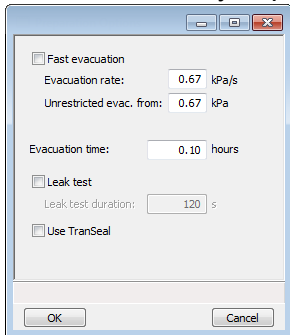| Field or Button	Description
	standard nitrogen analysis of mesoporous materials such as catalysts. If the first pressure table point is low and the gas uptake of the sample is expected to be high, this option can shorten the time required to reach the first point on the pressure table. The sample is dosed repeatedly at low pressures with a specified amount of gas until the first pressure point is reached. This initial dosing quickly meets the adsorptive demand of the sample. 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. Enter the amount of gas to be added to the sample for each dose cycle. **Maximum volume increment.** Select to determine when additional data points are collected between target pressures in regions of adsorption. When the maximum increment has been adsorbed since the last collected data point, another point is equilibrated and collected. During desorption, this field is treated as a maximum volume decrement value. 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. This field is disabled if *First pressure fixed dose* is selected. **Absolute / Relative pressure tolerance.** Values used to determine how close the actual pressure must be to each target pressure from the pressure table. At lower pressures, the relative tolerance value is lower. At higher pressures, the absolute tolerance value is lower. **Experiment 1.** There might be an absolute tolerance of

Analysis Conditions (continued)

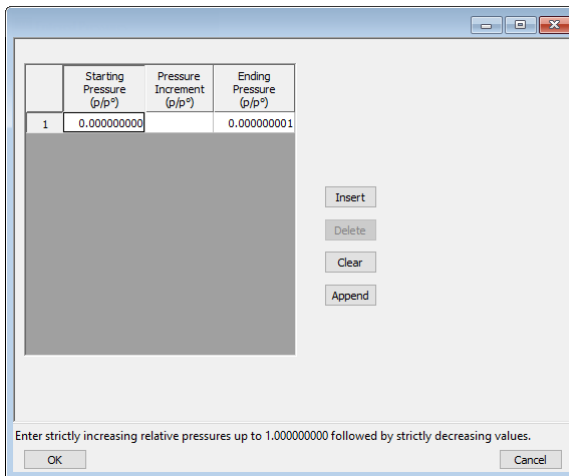| Field or Button	Description
	5 mmHg, a relative tolerance of 5%, and a target pressure of 40 mmHg; 5% of 40 mmHg is 2 mmHg. Since 2 mmHg (relative tolerance) is lower than 5 mmHg (absolute tolerance), 2 mmHg is used. Therefore a minimum pressure of 38 mmHg (40 - 2) must be attained to collect data for a target pressure of 40 mmHg.
	Experiment 2. There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 200 mmHg; 5% of 200 mmHg is 10 mmHg. Since 5 mmHg (absolute tolerance) is lower than 10 mmHg (relative tolerance), 5 mmHg is used. Therefore a minimum pressure of 195 mmHg (200 - 5) must be attained to collect data for a target pressure of 200 mmHg.
	Normally, surface area measurement points are widely spaced and the resulting measurement is not very sensitive to the precise location of points so wider tolerances may be used. Unnecessarily tight tolerances lengthen the analysis.
	Low Pressure incremental dose mode.
	Select when performing an analysis of microporous materials. At low pressures on Type 1 isotherms, the pressure points are very closely spaced, making a useful pressure table difficult to define. When enabled, equilibrium points are measured at approximately equal intervals on the quantity adsorbed axis. Each dose is fully equilibrated and recorded as a data point.
	In this mode, the sample is successively dosed with a specified amount of gas until the first pressure point is reached. The first point is the threshold value, triggering the transition from *Incremental Dose Mode* to *Pressure Table Mode*. When the first pressure table value is reached*, Incremental Dose Mode* is disabled, and points are recorded in accordance with the specified pressure table. Because the data points recorded during *Incremental Dose Mode* may define most of the analysis, one point on the pressure table can be sufficient and serve as the end point for the analysis.
	• **Dose amount.** The amount of gas to be added to the

Analysis Conditions (continued)

Field or Button	Description
	sample for each data point until the first point on the pressure table is reached. This field is enabled when *Low pressure incremental dose mode* is selected. ● **Equilibration Delay.** Enabled when *Low Pressure incremental dose mode* is selected. **Minimum.** Prevents premature equilibration caused by reduced percentage sensitivity to pressure changes at the lowest pressures. **Maximum.** Prevents the effects of long term temperature or pressure drift, which may cause the analyzer to wait an excessive length of time for equilibration.
p° 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 window. Options and descriptions: 1. **Analysis bath temperature** (not used for krypton analyses). Measures the p_0 on a continuous basis. Allows the measurement of each data point without slowing the analysis. 2. **Analysis bath temperature** (not used for krypton analyses). Measures the p_0 on a continuous basis and over the sample, then adjusts the measured p_0 in the sample tube to agree with the p_0 over the sample. 3. The p_0 and analysis bath temperatures or an analysis

Analysis Conditions (continued)

Field or Button	Description
	bath temperature only (when *Absolute pressure dosing* is selected on the *Analysis Conditions* window). This method uses the entered values.
	4. **Psat gas.** If this is a krypton analysis, select the *Nitrogen @ 77.35K* option from the *Adsorptive* drop-down box. Click **Psat vs T** to edit the values of the Psat vs T table. Editing the values in the current table does not affect the original table.
	5. **Analysis bath temperature.** Analysis bath temperature and an estimate for the initial p_0 (not used for krypton analyses). Measures the p_0 over the sample. After the p_0 is measured, the value is reapplied to all data points.
	6. **Analysis bath temperature.** This method calculates the p_0 at the time of analysis using the entered temperature.
	If *From last sample tube* (for krypton analysis) on the analysis window and a p_0 option other than 3, 4, or 6 is selected, an error message is displayed when the analysis begins.
Preparation *[button]*	Use to enter analysis preparation details.
	Evacuation rate. The rate for restricted evacuation.
	Evacuation time. 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.
	Fast Evacuation. Select for samples (such as pellets) that do not fluidize or shed particles during evacuation.

Analysis Conditions (continued)

Field or Button	Description
	Leak Test. Enables the system to check for leaks or sample outgassing before the analysis. The leak test allows sample pressure to rise during the test. If the pressure rises more than 0.15 mmHg, the analysis does not proceed and the operator is notified. While leak testing slightly increases analysis time, it prevents the continuation of analysis and collection of erroneous data if a leak exists. Select to enable the *Leak test duration* field. **Leak test duration.** Enter the duration of the leak test. **Unrestricted evac. from.** The pressure at which unrestricted evacuation is to begin. This option is enabled when *Fast evacuation* is not selected. **Use TranSeal.** Select if using the TranSeal to transfer the sample from the preparation port to the analysis port under vacuum.
Pressures *[button]*	Available when the *Target Pressures* option is selected. Use to edit the *Entered Pressures* table. The pressure table consists of relative pressure points at which isotherm data are to be collected. An optional pressure increment can be entered in the *Pressure Increment* column, which will cause additional points to be collected at intervals of the pressure increments up to the relative pressure specified in the *Ending Pressure* column.

Analysis Conditions (continued)

Field or Button	Description
	The relative pressure points may span the entire range of 0.000000000 to 0.000000001 p/p^0. There must be one adsorption branch (strictly increasing pressures) followed optionally by one desorption branch (strictly decreasing pressures).

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.
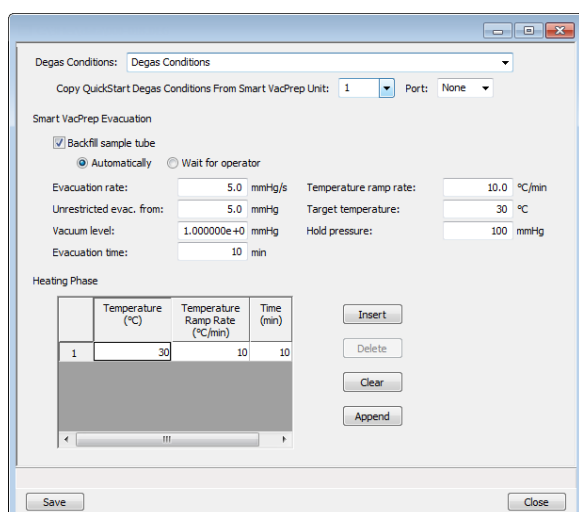
DEGAS CONDITIONS

File > Open > [.DEG File]

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

> ✏️ Use this option only when the SmartPrep or Smart VacPrep is installed. Degassing is a required step in preparation for an analysis. The *Degas Conditions* tab provides settings that will be automatically applied during the degassing procedure when using the Smart VacPrep.
>
> Log in to your customer portal to access the Smart VacPrep Operator Manual.

Degas Conditions

Field or Button	Description
Copy QuickStart Degas Conditions from Smart VacPrep unit *[drop-down box]*	Use to copy the degas conditions settings from the selected Smart VacPrep unit and port.
Degas Conditions *[drop-down box]*	Use to browse for a .DEG file that contains degas condition parameters to be used in the analysis.

Degas Conditions (continued)

Field or Button	Description
Heating Phase *[table]*	This option is applicable when degassing with either a Smart VacPrep or a SmartPrep. Enter up to five stages of degas conditions. **Temperature.** Temperature at which the sample is to be held while degassing. **Time.** How long the sample is to be held at the specified temperature before beginning to cool down. **Temperature Ramp Rate.** The rate at which the temperature will change while advancing to the hold temperature.
Smart VacPrep Evacuation *[group box]*	**Backfill sample tube.** Indicate if the sample tube should be backfilled automatically or wait for operator response. **Evacuation rate.** Rate used for evacuation. **Evacuation time.** Length of time for preliminary evacuation before proceeding with the *Heating Phase* temperature schedule. The timer starts when the vacuum level is reached. **Hold pressure.** Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the *Hold* pressure. This prevents damage to the sample structure due to 'steaming' and /or elutriation due to excessive escaping gas velocity. **Target temperature.** Targeted temperature for evacuation. **Temperature ramp rate.** Rate at which the temperature is to change when advancing to the target pressure. **Unrestricted evac. from.** Pressure at which the unrestricted evacuation is to begin. **Vacuum level.** Evacuation time starts when the vacuum level is reached.

🖉 **For fields and buttons not listed in this table, see** *Common Fields and Buttons on page 2 - 4*.

REPORT OPTIONS

File > Open > [.RPO File]

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

> **See also:**
>
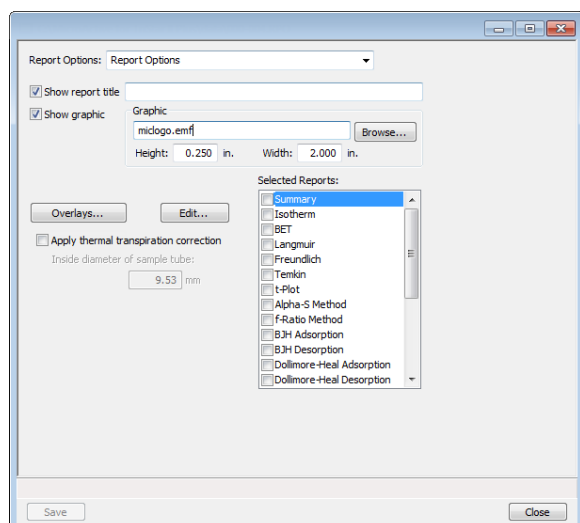> - *About Reports on page 7 - 1*
> - *Selected Reports on page 8 - 1*

Log in to your customer portal to access MicroActive Report Tutorials and the Calculations document.

Additional reports are available using the *Reports* menu.

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

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

Report Options files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis. See *Graph and Sample Overlays on page 7 - 26*.
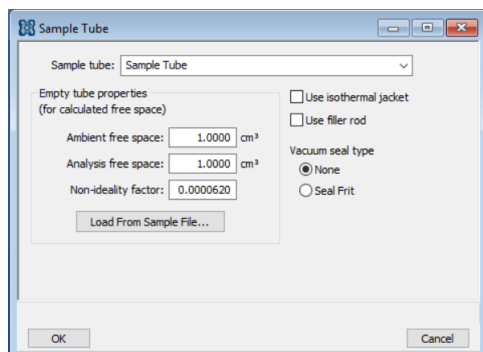
Report Options

Field or Button	Description
Apply thermal transpiration correction *[check box]*	Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Do not use filler rods in the sample tube when applying correction for thermal transpiration. Always use thermal transpiration when performing micropore analyses. **Inside diameter of sample tube.** Enabled when *Apply thermal transpiration correction* is selected. Enter the inside diameter of the sample tube used in the analysis. See the *Thermal Transpiration Correction* section of the *Calculations* document. Log in to your customer portal to access the *Calculations* document.
Overlays *[button]*	See *Graph and Sample Overlays on page 7 - 26*.
Report Options *[drop-down box]*	Browse for a .RPO file that contains report options parameters to be used in the report.
Selected Reports *[group box]*	Select the report names to include in the report. For BJH reports, BJH pore dimension can be calculated in pore width (w), pore radius (R), or pore diameter (D). Go to **Options > Units** to specify default calculations.
Show graphic *[text box]*	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title *[check box]*	Select and enter a report title to appear on the report header.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

SAMPLE TUBE

File > Open > [.STB File]

Sample Tube files specify information about the sample tube.

Sample Tube

Field or Button	Description
Ambient free space *[text box]*	Empty sample tube gas capacity measured at room temperature.
Analysis free space *[text box]*	Empty sample tube gas capacity measured with the dewar raised.
Load from Sample File *[button]*	Loads parameters from the selected sample file.
Non-ideality factor *[text box]*	Compensates for the forces of attraction between molecules in a real gas. This value is used for a calculated free space.
Sample tube *[drop-down box]*	It is a good practice to label each sample tube with a unique identification. Enter that information here. This information will also appear in the *Sample Tube* drop-down list on the *Sample Description* tab.
Use filler rod *[check box]*	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. It is generally a good practice to use filler rods for samples having less than 100 square meters of total surface area.

Sample Tube (continued)

Field or Button	Description
Use isothermal jacket *[check box]*	Select if an isothermal jacket is to be used. An isothermal jacket maintains a constant temperature profile along the sample tube stem during an extended analysis of more than 1 or 2 hours.
Vacuum seal type *[group box]*	Select the seal type to be used.

> **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

5 DEGASSING

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

Log in to your customer portal to access the Smart VacPrep Operator Manual or the SmartPrep Operator Manual.

DEGAS ON THE SMARTPREP

> **See:**
>
> ***Degasser Options on page 1 - 5***

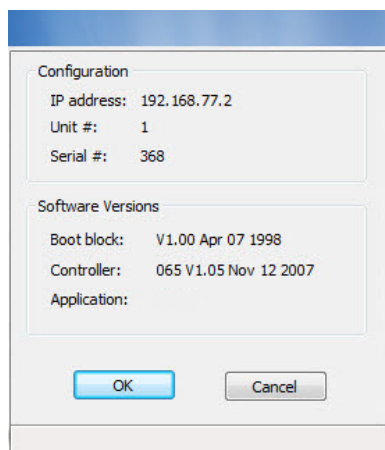> If a SmartPrep is not connected to the analyzer, the SmartPrep menu items are not available.

Log in to your customer portal to access the SmartPrep Operator Manual.

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:

[OK]   [Cancel]

SHOW SMARTPREP STATUS

Unit [n] > Degas > Show SmartPrep Status

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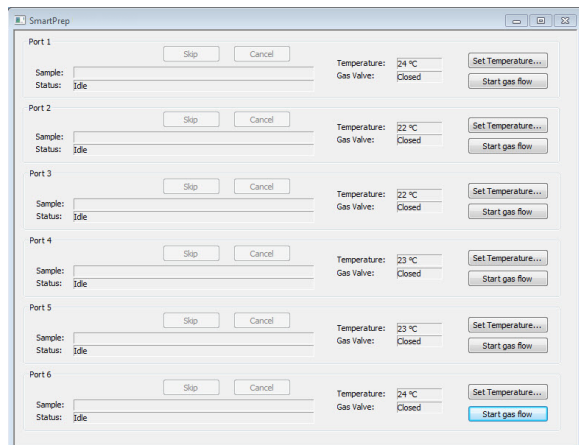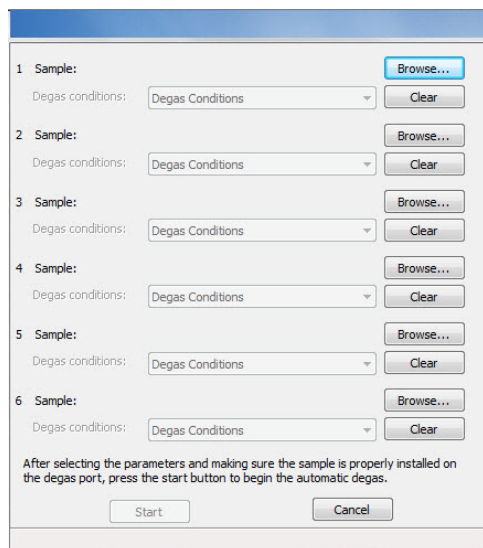

Show SmartPrep Status

Button	Description
Cancel *[button]*	Discards any changes or cancels the current process.
Set Temperature *[button]*	Use to set the temperature of the selected port.
Skip *[button]*	Use to bypass the current stage.
Stop Gas flow *[button]*	Stops the gas flow to the selected port.

START SMARTPREP DEGAS

Unit [n] > Degas > Start SmartPrep Degas

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

Start SmartPrep Status

Button	Description
Browse *[button]*	Searches for a file.
Cancel *[button]*	Discards any changes or cancels the current process.
Clear *[button]*	Clears the sample file selection for a port.
Start *[button]*	Starts degassing.

DEGAS ON THE SMART VACPREP

> **See:**
>
> *Degasser Options on page 1 - 5*

Log in to your customer portal to access the Smart VacPrep operator manual.

TRANSFER A DEGASSED SAMPLE TO AN ANALYSIS PORT

> **See also:**
>
> *Worksheets on page F - 1*

The sample tube must be removed from the degas port, weighed, and then installed onto the analysis port for analysis.

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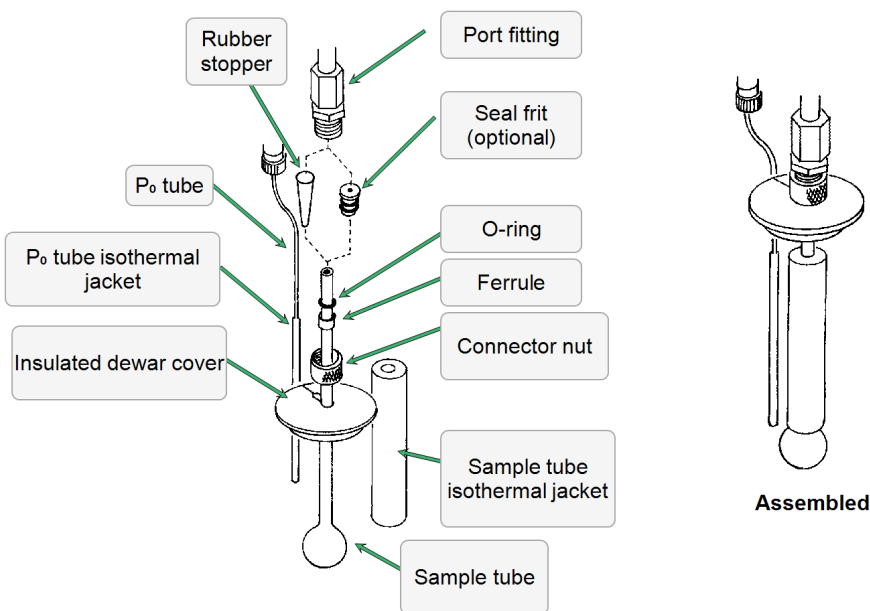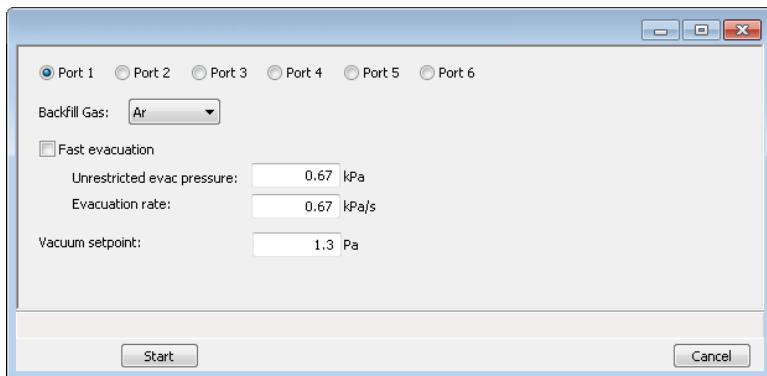

1. Allow the sample tube to cool.

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

2. Carefully remove the heating mantle clip and the heating mantle from the sample tube.
3. Hold the sample tube, loosen the port connector nut, and remove the sample tube from the degas port.
4. Remove the connector nut, ferrule, and O-ring from sample tube stem.
5. Weigh the sample tube set. Use the *Sample Data Worksheet* to determine the sample mass.
6. Slide an isothermal jacket down over the sample tube stem until it touches the sample tube bulb.
7. Place the connector nut, ferrule, and O-ring onto the sample tube stem.
8. Remove the stopper and immediately attach the sample tube to the analysis port, pushing it fully up into the port. Secure it in place by screwing the connector nut onto the analysis port connector; hand tighten the connector nut. If a Seal Frit was used, it does not have to be removed.
9. Place the dewar cover over the sample tube stem just above the isothermal jacket.

EVACUATE PORTS

Unit [n] > Evacuate Ports

Evacuate Ports

Field or Button	Description
Port[s] *[group box]*	Select the ports to evacuate.
Fast evacuation	**Unrestricted evac. pressure.** Pressure at which the unrestricted evacuation is to begin. **Evacuation rate.** Rate at which unrestricted sample evacuation should begin. **Vacuum setpoint.** The vacuum level to be achieved before timed evacuation begins.

> **For fields and buttons not listed in this table, see _Common Fields and Buttons on page 2 - 4_.**

6 PERFORM AN ANALYSIS

> **CFR Note** In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

DEWAR PRECAUTIONS

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

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

When handling dewars containing liquefied gases or cryogenic liquids:

- Wear protective equipment:
  - goggles or face shield
  - an insulated or rubber apron
  - insulated gloves

- When pouring liquefied gases from one container to another:
  - cool the receiving container gradually to minimize thermal shock
  - pour the liquified gas slowly to prevent splashing
  - vent the receiving container to the atmosphere

FOR GLASS DEWARS

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

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PREPARE FOR ANALYSIS

The steps in this topic properly prepare the equipment for an analysis. It is recommended to perform the tasks in the provided order.

> **See also:**
>
> - *Dewar Precautions on the previous page*
> - *Select a Sample Tube on the facing page*
> - Step 1 - *Clean and Label Sample Tubes on page 6 - 4*
> - Step 2 - *Create the Sample File on page 6 - 6*
> - Step 3 - *Determine the Sample Mass for Physical Adsorption on page 6 - 6*
> - Step 4 - *Degas the Sample on page 6 - 8*
> - Step 5 - *Sample Tube Installation on page 6 - 9*
> - Step 6 - *Fill and Install the Dewar on page 6 - 11*

SELECT A SAMPLE TUBE

A sample tube set consists of:

- Sample tube
- Stopper or Seal Frit
- Filler rod

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

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

Filler rods help to ensure accuracy in samples with lower total surface areas by reducing the free space volume. It is generally a good practice to use filler rods for samples having less than 100 square meters of total surface area. Filler rods are unnecessary for samples with total surface areas greater than 100 square meters.

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

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

> If a Seal Frit is not used, insert a stopper immediately after removing the sample from the degas port.

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

CLEAN AND LABEL SAMPLE TUBES

> ✏️ The equipment images in this topic may differ slightly from your equipment, however the instructions are the same unless otherwise noted.

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

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

1. Preheat drying oven to 110 ºC.
2. Verify that the ultrasonic cleaning unit is clean.
3. Use 5 grams of Alconox (or other suitable detergent) per 500 mL of warm water and fill the ultrasonic unit with enough water to cover the sample tubes and filler rods (if used). If too much detergent is used, it may be difficult to rinse from the sample tubes. Ensure the detergent is dissolved before placing the sample tubes and filler rods into the water.
4. Fill the sample tubes with warm water and place them in the ultrasonic cleaning unit, then place the filler rods in the unit. Turn on the ultrasonic cleaning unit for approximately 15 minutes.

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

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

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

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

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

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

CREATE THE SAMPLE FILE

See:

Create Sample Files on page 3 - 2

DETERMINE THE SAMPLE MASS FOR PHYSICAL ADSORPTION

See also:

Sample Data Worksheet for Gas Adsorption on page F - 2

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

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

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

Analysis results are expressed in units of surface area per gram of sample; therefore, it is important the true sample mass be known.
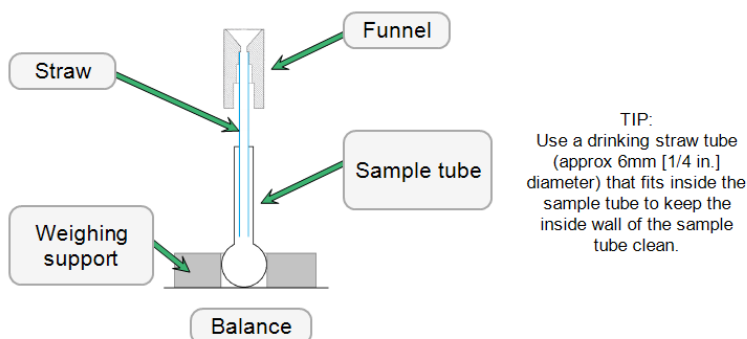
Follow the instructions on the *Sample Data Worksheet* and complete all fields to find the true sample mass.

1. Record the sample tube identification on the *Sample Data Worksheet.*
2. Place the sample weighing support on the balance. Tare the balance and allow it to stabilize at zero (0).
3. Place the empty sample tube set (empty sample tube and stopper) on the sample weighing support and place it on the balance.
4. Record the stabilized mass on the *Sample Data Worksheet* . Remove the sample tube set from the balance.

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

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

TIP:
Use a drinking straw tube (approx 6mm [1/4 in.] diameter) that fits inside the sample tube to keep the inside wall of the sample tube clean.

9. Replace either the rubber stopper, Seal Frit, Check Seal, or TranSeal.
10. On the *Sample Data Worksheet,* record the following:
    a. Mass of the sample tube set with the sample.
    b. Subtract the *Mass of empty sample tube set* from the *Mass of sample tube set plus sample.*

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DEGAS THE SAMPLE

See also:

- *Degas Conditions on page 4 - 15*
- *Degassing on page 5 - 1*
- *Sample Data Worksheet for Gas Adsorption on page F - 2*

If using the Smart VacPrep degasser, go to **Smart VacPrep > Unit [n] > Start Degas**, then degas the sample using menu commands and information entered on the *Degas Conditions* tab. Log in to your customer portal to access the Smart VacPrep Operator Manual.

For instructions on degassing on the SmartPrep, see *Degas on the SmartPrep on page 5 - 1*.

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.

After degassing is complete, perform the following steps:

1. Weigh the sample tube set containing the sample. Record the mass on the *Sample Data Worksheet for Gas Adsorption on page F - 2* as *Mass of Sample tube set plus sample (After Degas)*.
2. Subtract the *Mass of empty sample tube set (Before Degas)* from the *Mass of Sample tube set plus sample (After Degas)* to obtain the sample's mass. Record this value as *Mass of sample (After Degas)*.

SAMPLE TUBE INSTALLATION

If using...	Then...
A rubber stopper	Remove it.
An isothermal jacket	Slide the jacket down over the stem of the sample tube until it touches the sample tube bulb.
A filler rod	Hold the sample tube horizontally and carefully slide the filler rod into the tube. If using a hanging filler rod, the rod attaches to the Seal Frit. Filler rod Sample tube **CAUTION** Do not hold the rod vertically and drop the rod into the tube; this could break the rod and/or tube.

1. Remove the closure from the sample tube.
2. If a filler rod is used, slide the filler rod slowly into the sample tube.
3. Slide an isothermal jacket down over the sample tube stem until it touches the sample tube bulb.
4. Place the connector nut, ferrule, and O-ring onto the sample tube stem.

O-ring
(1/4 in. from top)

Ferrule

Connector nut

Isothermal
jacket
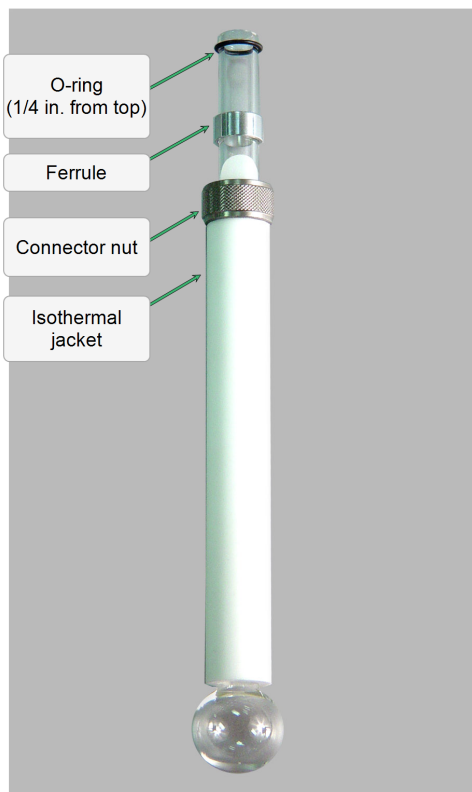
5. Attach the sample tube to the analysis port, pushing it fully up. Turn the connector nut clock-
    wise to hand tighten.

Connector nut
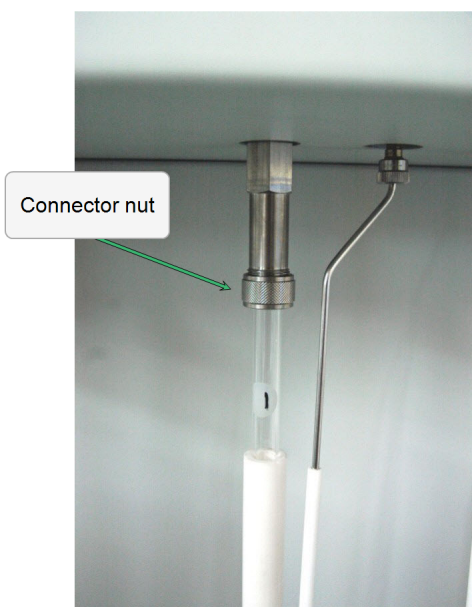
FILL AND INSTALL THE DEWAR

> ✏️ The equipment images in this topic may differ slightly from your equipment, however the instructions are the same unless otherwise noted.

> 🔬 **See also:**
>
> *Dewar Precautions on page 6 - 1*

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

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

---

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

Wetness or frozen condensation indicates bath liquid level

Level indicator mark

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

PERFORM A SAMPLE ANALYSIS

CFR Note	In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

Unit [n] > Start Analysis

Before beginning an analysis, ensure the tank pressure for the gas regulator is at least 200 psig. Pressures less than 200 psig may cause the sample to be inadequately saturated, resulting in inaccurate data or termination of analysis.

Evacuation through the **Unit > Evacuate Port** option can be performed on any idle port while analyses are in progress.


Standard Analysis Guidelines

- Standard analyses cannot be performed if a high throughput or krypton analysis is in progress.
- One analysis can be started at a time.
- A sample can be added to any idle port and an analysis started without disturbing the analyses being performed on other ports.
- Samples can be removed from any of the six ports without disturbing the analyses being performed on other ports.
- The sample dosing method should be *Normal*.
- All analyses must use the same gas.
- If saturation pressure is being measured, all analyses must use the same Psat gas.

Sample Analysis

Field or Button	Description
Density / Mass / Sample + Tube / Empty Tube *[text box]*	Enter values for the sample's mass and density. These values may be edited after analysis.
New *[button]*	Creates a new sample file.

> For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.
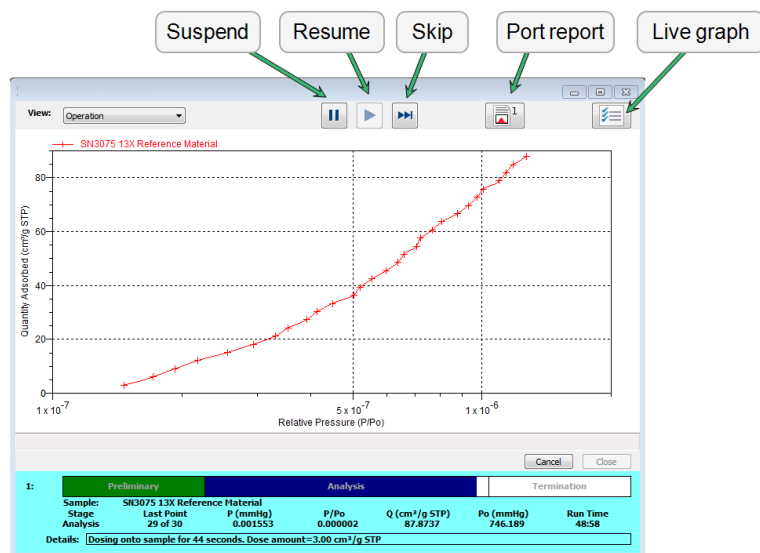
Sample Analysis Graph

Field or Button	Description
Live Graph Settings *[button]*	Select Thermal transpiration, x-axis Quantity (relative or absolute pressure), and the x-axis Scale (linear or logarithmic).
Report after analysis *[button]*	Generates reports to the selected destination when the analysis is complete.
Report Port *[button]*	Generates a report on data being collected. The reports are displayed on the computer monitor only.
Resume *[button]*	Restarts the suspended analysis.
Skip *[button]*	Moves to the next step. Select the ports to skip. In 21CFR11 environments, steps cannot be skipped.

Sample Analysis Graph (continued)

Field or Button	Description
Status window	Displays the last point pressure and relative pressure for each port.
Suspend *[button]*	Suspends an analysis in progress.

> **For fields and buttons not listed in this table, see _Common Fields and Buttons on page 2 - 4_.**

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PERFORM A HIGH THROUGHPUT ANALYSIS

Unit [n] > Start High Throughput Analysis

CFR Note	In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

Use to perform up to six simultaneous high throughput analyses.

High Throughput Analysis Guidelines
- All ports must be idle in order to start an analysis.   - All analyses must use the same analysis gas.   - All analyses that measure Psat must use the same Psat gas (which may be different from the analysis gas).   - The sample file must specify *Normal* as th*e Dosing Method* in the *Adsorptive Properties*.   - From one to six analyses can be started simultaneously.   - Samples cannot be removed from or added to ports until the full set of analyses has completed.

The steps for performing a high throughput analysis are the same as the Krypton analysis. See *Perform a Krypton Analysis on the facing page*.

PERFORM A KRYPTON ANALYSIS

Unit [n] > Start Krypton Analysis

CFR Note — In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

Krypton analyses are available only if the krypton option is installed.

See also:

- *Dewar Precautions on page 6 - 1*
- *Perform a High Throughput Analysis on the previous page*
- *Perform a Micropore Analysis on page 6 - 20*
- *Perform a Sample Analysis on page 6 - 13*
- *Prepare for Analysis on page 6 - 2*

Data collection is done sequentially — one analysis starts and completes before the next analysis begins.

Use to perform up to five simultaneous krypton analyses.

Krypton Analysis Guidelines

- All ports must be idle in order to start an analysis.
- All analyses must use krypton gas.
- From one to five analyses can be started simultaneously. The last port is used to store krypton for dosing (Port 2, 4, or 6 depending on system configuration).
- An empty sample tube must be installed in the last port (2, 4, or 6 depending on system configuration).
- The sample file must specify *From last sample port* as the *Dosing Method* in the *Adsorptive Properties*.
- Samples cannot be removed from or added to ports until the full set of analyses has completed.

> The dewar below the port used to store and purify krypton must be at least 50% full of the analysis bath liquid and an empty sample tube must be installed on the port before starting a krypton analysis.

1. Install an empty sample tube on the port to be used to store krypton (the last port: 2, 4, or 6) and place a dewar cover over the sample tube stem and the P_0 tube stem.

2. Go to *Unit > Start Krypton Analysis*. For each port to be used, either click **Browse** and select a sample file or click **New** to create a new sample file.

3. Verify the information populated into the sample identification. This information is pulled from the sample file. The *Density* value is applicable only if using the *Calculate* method for the free space determination.

4. Edit the P_0 and *Bath temperature* fields, if necessary.

5. Click **Report after analysis** to generate reports automatically when the analysis is complete. On the *Report Settings* window, select the report destination. Click **OK** to return to the previous window.

6. 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.
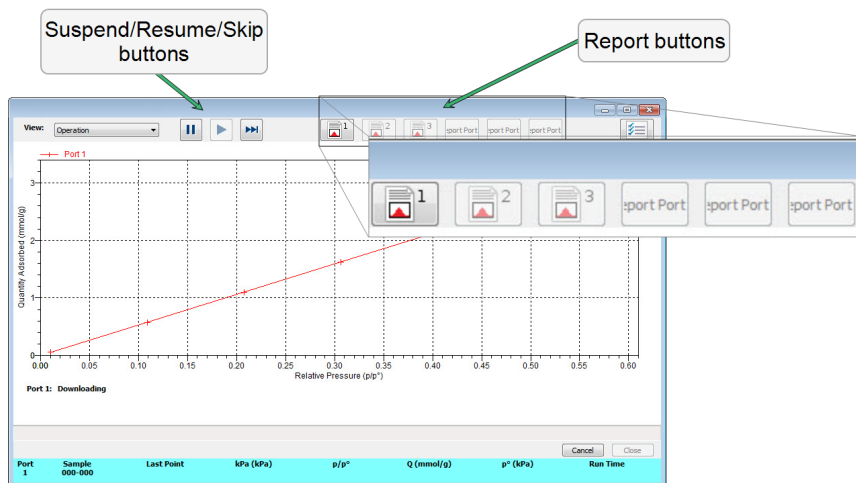
| ⚠ | Use the **Skip** function with caution; the ASAP 2460 performs multiple steps for a given task. Skipping certain steps may cause corruption of data, instrument damage, or personal injury. |

7. To view an analysis report, click the appropriate **Report Port** button.

8. When the analysis is complete, the **Next** button is displayed. Click **Close** to exit or **Next** to perform another analysis.

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PERFORM A MICROPORE ANALYSIS

Unit [n] > Start Micropore Analysis

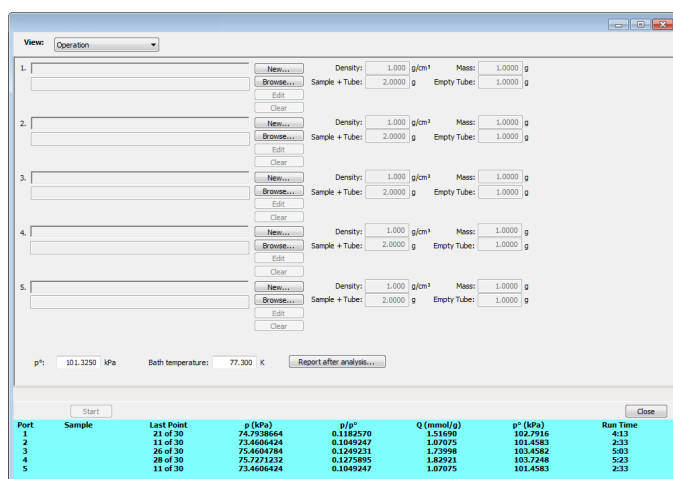| CFR Note	In 21CFR11 environments, users are required to login to the Confirm application to start an analysis. Once the analysis window is opened, manual control is disabled until the analysis has completed. During analysis, pausing and resuming is allowed, however, steps cannot be skipped.

	Micropore analyses are available only if the Micropore option is installed.

Micropore Analysis Guidelines

- All ports must be idle in order to start an analysis.
- All analyses must use the same analysis gas.
- All analyses that measure Psat must use the same Psat gas (which may be different from the analysis gas).
- The sample dosing method should be Normal.
- From one to six analyses can be started simultaneously.
- Samples cannot be removed from or added to ports until the full set of analyses has completed.

The steps for performing a MicroPore analysis are the same as the krypton analysis. See *Perform a Krypton Analysis on page 6 - 17*.

7 ABOUT REPORTS

Reports > Open Report > [.REP file]

Opens a saved report.

Reports > Close Reports

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

Reports > Start Report

Generates a report on a sample analysis.

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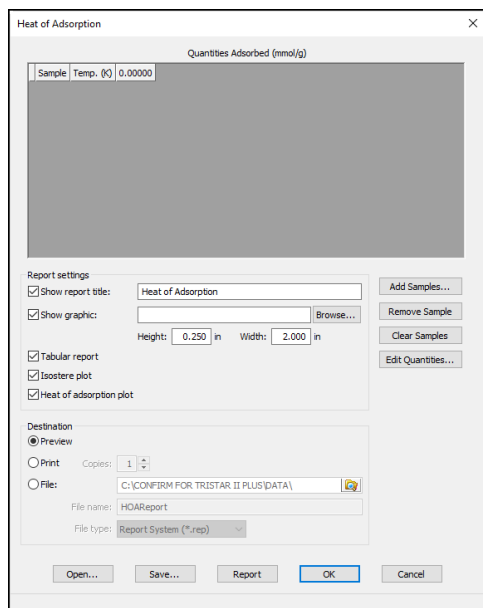
START REPORTS

Reports > Start Report

Starts the selected report. Select a file from the *Files* list. Ensure the selected file has a status of either *Complete* or *Analyzing*.

HEAT OF ADSORPTION REPORT

Reports > Heat of Adsorption

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

Heat of Adsorption Report

Field or Button	Description
Add Samples *[button]*	Adds a sample file to the table.
Clear Samples *[button]*	Removes all entries from the table.
Edit Quantities *[button]*	Use to specify the range of surface coverage to include in the report.

Heat of Adsorption Report (continued)

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

> For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

SPC REPORT

Reports > SPC Report Options

Use to generate reports with various *SPC* (Statistical Process Control) options. All selected variables must be computed for each sample file used in an SPC report; therefore, it is more efficient to select only the necessary variables.
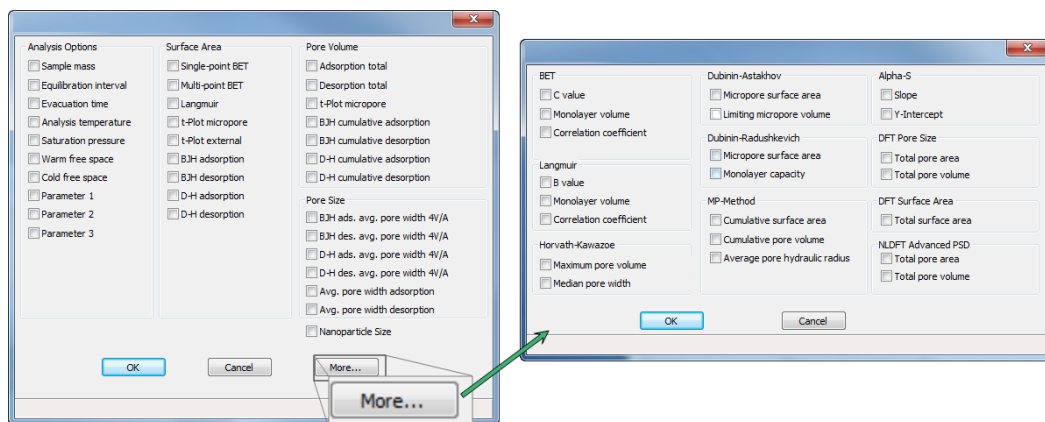
The selected items display as graph variable selections in **Reports > Regression Report** and graph selections in **Reports > Control Chart**. If report options for NLDFT Advanced PSD are required, click **More**.

REGRESSION REPORT

Reports > Regression Report

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

Regression Report

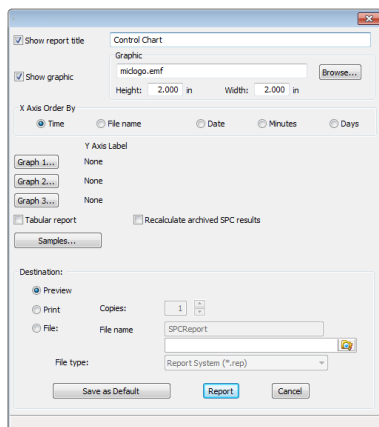| Field or Button	Description
Autoscale *[check box]*	When enabled, allows the x- and y-axes to be scaled automatically.
Axis Range *[text box]*	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if *Autoscale* is selected.
Label data *[check box]*	Use to label the points on the plot to correspond with the values in the sample files.
Recalculate archived SPC results *[check box]*	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. If an earlier application version will not be reused, this warning message can be safely disregarded.
	When this option is selected, this message will display:

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Regression Report (continued)

Field or Button	Description
	Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.
	If *Do not show me this message again* is selected, the message cannot be redisplayed without Micromeritics assistance.
	The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Samples *[button]*	Select additional sample files to add to the report.
Save as Default *[button]*	Click to save selected report options as default report settings.
Show graphic *[check box]*	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title *[check box]*	Select and enter a report title to appear on the report header.
Tabular report *[check box]*	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X- and Y-Axis Variable *[drop-down box]*	Use to designate the x- and y-axes variables. The variables in the drop-down lists are those selected in the ***Reports > SPC Report Options*** window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.
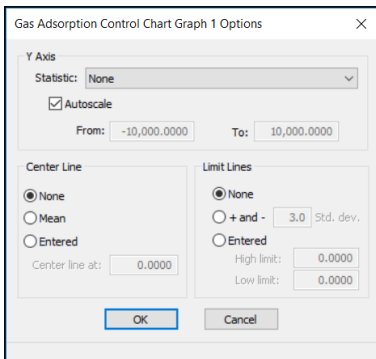
CONTROL CHART REPORT

Reports > Control Chart

Generates a Statistical Process Control *(SPC)* chart report which plots the changes in a statistic.

Control Chart Report

Field or Button	Description
Graph [n] *[button]*	Defines the y-axis of each graph. **Statistic.** Displays the SPC variables selected on the ***Reports > SPC Report Options*** window. The selected variable will be plotted for each selected sample. This selection also becomes the y-axis label. **Autoscale.** Allows the y-axis to be scaled automatically. To specify a range, deselect this option and enter a range in the *From* and *To* fields.

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Control Chart Report (continued)

Field or Button	Description
	Center Line. Displays placement options for the center line in the graph. Select *Entered* to specify placement of the line or *Mean* to place the center line at the calculated mean value for the selected samples. **Limit Lines.** Displays limiting lines options. Lines can be placed at some multiple of the standard deviation or at specified positions (*Entered*). When *Entered* is selected, enter the *High limit* and *Low limit* fields with appropriate values.
Recalculate archived SPC results *[check box]*	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. If an earlier application version will not be reused, this warning message can be safely disregarded. When this option is selected, this message will display: **Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.** If *Do not show me this message again* is selected, the message cannot be redisplayed without Micromeritics assistance. The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Report *[button]*	Generates the report.
Samples *[button]*	Select additional sample files to add to the report.

Control Chart Report (continued)

Field or Button	Description
Show graphic *[check box]*	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title *[check box]*	Select and enter a report title to appear on the report header.
Tabular report *[check box]*	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X Axis Order by *[group box]*	Select the order in which x-axis statistics are placed. Sort by: **Time.** Time the files were analyzed. **File name.** Alphanumeric order. **Date.** Date the files were analyzed. **Minutes.** Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. **Days.** Number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

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MICROACTIVE REPORTS

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

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

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.

Log in to your customer portal to access MicroActive Report Tutorials.

INTERACTIVE REPORTS

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

1. When opening a sample file that contains analysis data, a window with the following information will display:

   - a linear plot and log plot of the data collected during analysis
   - a summary of the analysis giving a single total pore volume and surface area
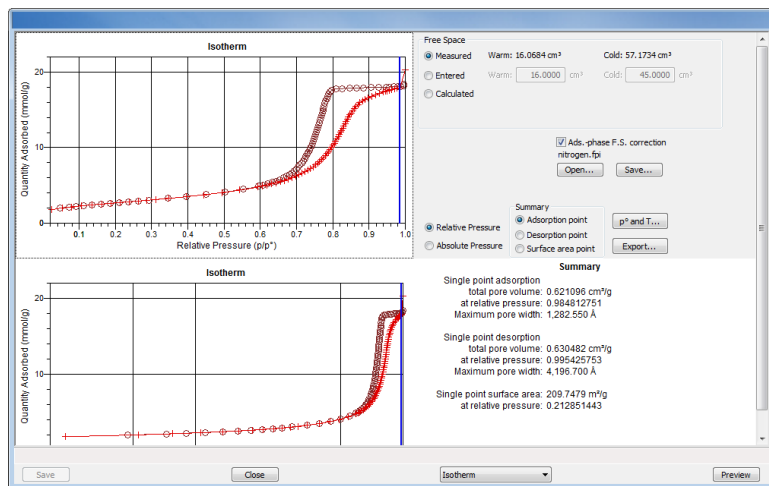
2. To view the plots in either relative or absolute pressure, select either the *Relative Pressure* or *Absolute Pressure* option.
3. To view the reports selected for generation during the analysis, click **Preview**.
4. From the drop-down list at the bottom of the window:

   - change the option presentation of the sample information window to either *Basic* or *Advanced* to modify certain file parameters, or
   - select another plot from the list and edit the data contained in the plot.

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

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

6. Click **Save**.

EVALUATE REPORT RESULTS

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

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

Analysis data can be evaluated by:

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

VIEW THE VALIDATION REPORT

The *Validation* report shows whether the data collected during an analysis are within typical ranges. Select the types of reports to include by selecting the report in the *Validation Report Options* window.
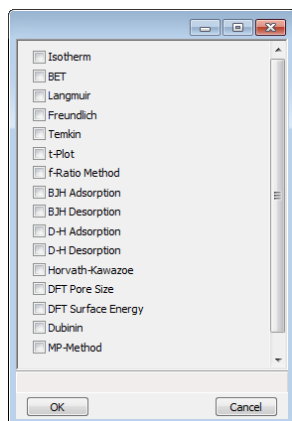
When a selected report is generated, if errors occur, a message is displayed across the top portion of the report and a unique symbol displays on the graph.

INSPECT THE ISOTHERM PLOT

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

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

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

EVALUATE THE ISOTHERM TABULAR DATA SET

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

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

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

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

REVIEW REDUCED DATA

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

PHYSICAL PARAMETERS

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

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

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

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

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

using which, the Kelvin equation[3] reduces to

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

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

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

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

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

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

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

The t-Plot method plots quantity adsorbed (Va) against thickness (t) derived from a thickness equation, and the Dubinin transform plots quantity adsorbed against log(P/P_0)n. All of these data reduction methods were first proposed for specific applications. The user must make a judgment as to the applicability of the method to a gas-solid system.

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

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

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

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

BET C-VALUE

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

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

DATA ANALYSES BY THE BJH METHOD

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

REPORT FEATURES AND SHORTCUTS

> **CFR Note**
>
> In 21CFR11 environments, members of the Analyst group must click **Preview** on the sample file window to access this screen.

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

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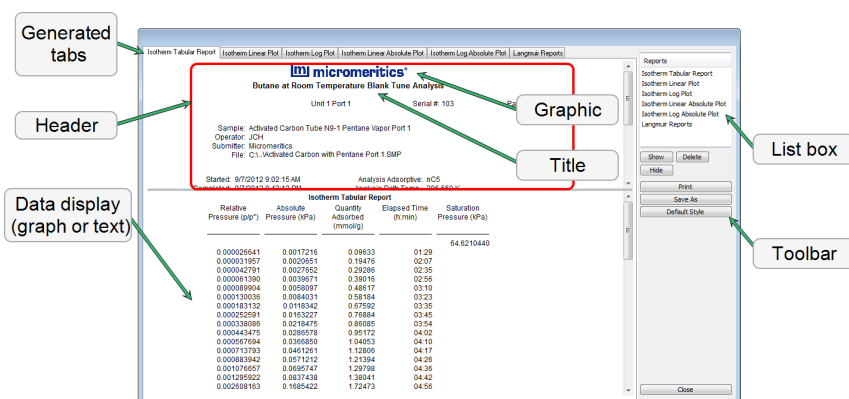

If configured, the report header can also contain a graphic and/or a title.

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

![micromeritics logo]

REPORT HEADER SHORTCUTS

Display header shortcuts by right clicking in the report header.
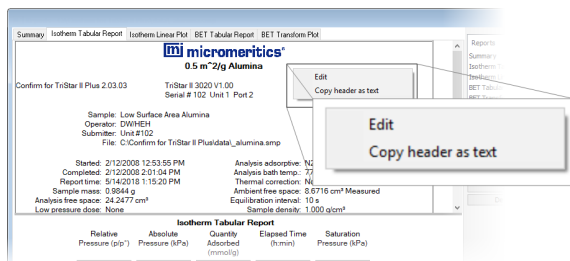
Report Header Shortcuts

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

REPORT TOOLBAR

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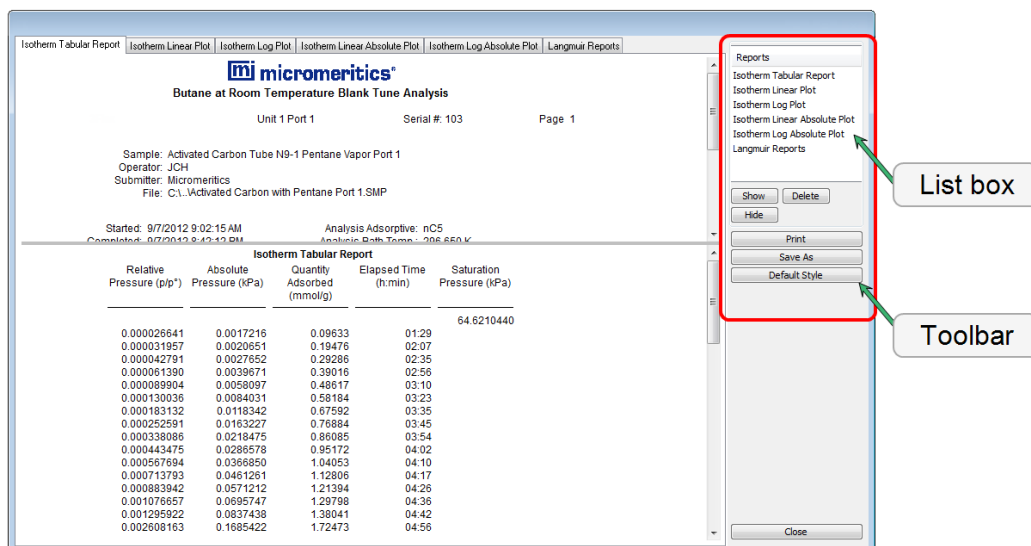

Report Toolbar

Field or Button	Description
Default Style *[button]*	Specify default report parameters for fonts and curve properties.
Delete *[button]*	Deletes the selected report in the *Reports* list box. 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.
Print *[button]*	Displays the *Print* window for report output.
Reports *[group box]*	Contains a list of all generated reports. The same reports display as tabs at the top of the report header unless the report has been hidden using the **Hide** button.
Show *[button]*	Displays the selected or hidden report in the *Reports* list box.

> ✏️ **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

TABULAR REPORT FEATURES AND SHORTCUTS

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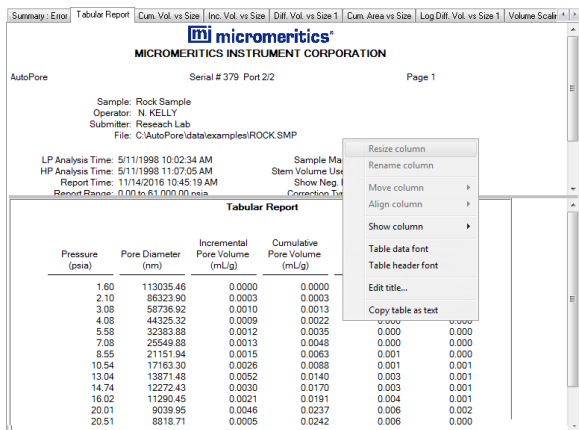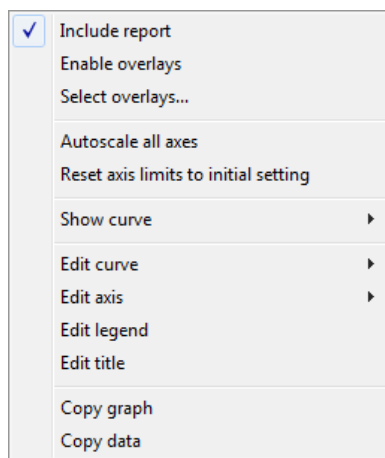

Tabular Report Shortcuts

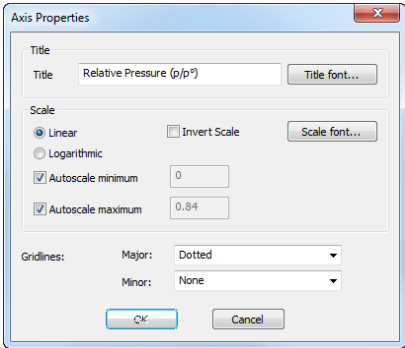| Field or Button	Description
Align column	Select to change the column alignment to either left, right, or centered.
Copy table as text	Use to copy the report contents to the clipboard as tab-delimited text. It can then be pasted into another document.
Edit title	Use to edit the report title and/or title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

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.
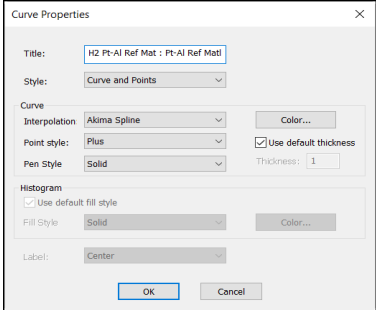
GRAPH FEATURES AND SHORTCUTS

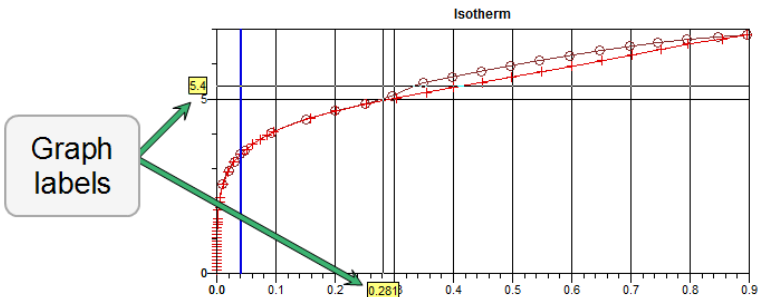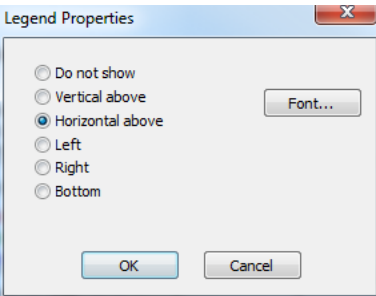Right click in the graph area to display graph report shortcuts.

✓	Include report
	Enable overlays
	Select overlays...
	Autoscale all axes
	Reset axis limits to initial setting
	Show curve ▶
	Edit curve ▶
	Edit axis ▶
	Edit legend
	Edit title
	Copy graph
	Copy data

Graph Shortcuts

Field or Button	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Copy graph	Copies the graph to the clipboard. It can then be pasted into other software programs.
Edit axis	Use to edit the selected axis properties. **Axis Properties** Title Title: Relative Pressure (p/p°) Title font... Scale ◉ Linear ☐ Invert Scale Scale font... ◯ Logarithmic ☑ Autoscale minimum 0 ☑ Autoscale maximum 0.84 Gridlines: Major: Dotted Minor: None OK Cancel **Grid lines.** Use to change how to display major / minor grid lines. **Scale.** *[group box]* • **Autoscale minimum/maximum.** *[check box]* To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box.

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Graph Shortcuts (continued)

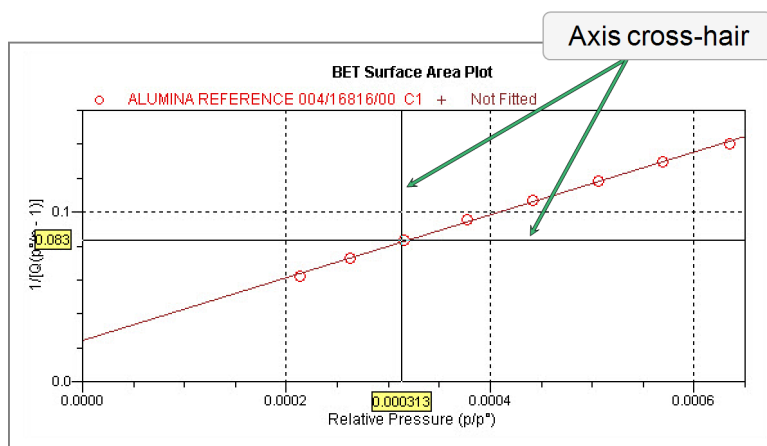| Field or Button	Description
	• **Invert scale.** *[check box]* Use to invert the scale. • **Linear/Logarithmic.** *[selection]* Select the option to scale the graph as linear or logarithmic. • **Scale font.** *[button]* Use to modify the font for the scale label. Deselect *Use default font* to enable font options. **Title.** *[text box]* Use to edit the selected axis label. **Title font.** *[button]* Use to modify the font for the selected axis label. Deselect *Use default font*. Select new font attributes for report data. Enable *Use default font* to reset default fonts.
Edit curve	Use to edit selected curve properties. **Color.** *[button]* Click to change the curve color. **Curve.** *[group box]* Use to change the interpolation, point style, and pen style for the selected curve. These options are disabled if *Use default fill style* is selected in the *Histogram* group box. **Histogram.** *[group box]* Enabled only if *Histogram* is selected in the *Style* drop-down list. Use to specify the type of fill, fill color, and label position for the selected curve. **Label.** *[drop-down box]* Select where the graph point labels will display (left, right, center, etc.) on the SPC report.

Graph Shortcuts (continued)

Field or Button	Description
	Style. *[drop-down box]* Use to select another style for the collected data curve. **Title.** *[text box]* Use to change the title of the selected curve. **Use default thickness.** *[check box]* Uses the default curve thickness. Deselect to enter a new thickness number in the *Thickness* text box.
Edit legend	Use to change the legend location and font.
Edit title	Select to change the report title.
Enable overlays	If overlays have been selected, this option displays (or hides) the overlays.
Include report	When selected, places a checkmark to the left of the report in the *Select Reports* list box on the *Report Options* tab.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.

Graph Shortcuts (continued)

Field or Button	Description
Select overlays...	Displays the option to select files to overlay onto the active graph. To view the overlays, click *Enable Overlays* on the shortcut menu.
Show curve	Displays a list of all curves. Select the curve(s) to display.

> **For fields and buttons not listed in this table, see** *Common Fields and Buttons on page 2 - 4*.
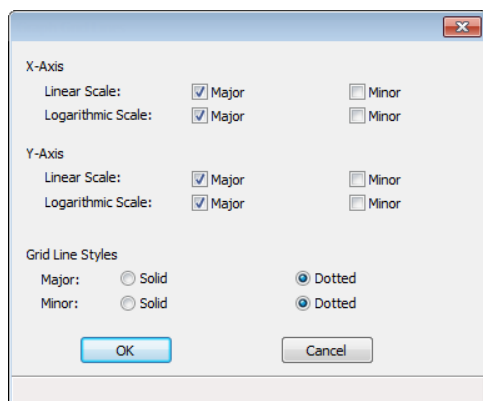
Axis Cross-Hair

Left click on the graph to view the cross-hair coordinates.

Graph Grid Lines

Options > Graph Grid Lines

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

Graph Grid Lines

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

> ✏️ **For fields and buttons not listed in this table, see _Common Fields and Buttons on page 2 - 4_.**

Zoom Feature

Use the zoom feature to examine graph details. Click, hold, and drag the left mouse button on the graphical area to be enlarged. A box will display in the area to be enlarged. To return to normal view, right click in the graph and select _Autoscale all axes_.

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GRAPH AND SAMPLE OVERLAYS

CFR Note   In 21CFR11 environments, this feature is applicable to members of the Developer group only.

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

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

This feature is available only when using *Advanced* option presentation. Go to **Options > Option Presentation > Advanced**.

GENERATE PORE SIZE DISTRIBUTION GRAPH OVERLAYS

The following reports in the physical adsorption applications can produce graphical results for a sample material's pore size distribution:

> **See also:**
>
> - *BJH Adsorption/Desorption Report Options on page 8 - 6*
> - *DFT Pore Size Report Options on page 8 - 12*
> - *Dollimore-Heal Adsorption/Desorption Report Options on page 8 - 15*
> - *Horvath-Kawazoe Report Options on page 8 - 29*

Two methods can be used to import and overlay report data into another interactive graph using shortcut menu options:

- **Import ASCII text data.** Data can be imported from an ASCII text file into the interactive graph. The ASCII text file must follow certain rules.
- **Copy/paste.** Data can be copied from one sample file (source) and pasted into another sample file (target).

Pore size distribution report overlays menu selections are:

- **Copy data.** Used to copy data sets.
- **Paste data.** Used to paste data sets.
- **Edit Imported Data.** Used to edit data sets.
- **Display Imported Data.** Used to hide or show the imported data.

IMPORT ASCII TEXT DATA

ASCII text file format rules

- The header must consist of one line to include title, two unit specifications, and distribution type:

    - Accepted pore dimension units are: A, nm, um
    - Accepted pore volume units are: cm3/g, cm^3/g, ml/g
    - Accepted distribution types are: cumulative, incremental

    Two examples of a header format:

        My Title (A, cm3/g, incremental)
        My Title (A, cm3/g, cumulative)

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

Sample ASCII Text File

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

Window appearance will vary depending on the selected report. This function can be performed on samples files with a *Completed* status or during an analysis.

1. Create the ASCII text file using the proper format as indicated above.
2. In the analyzer application, go to **File > Open**. Select a sample file to overlay graphs on to. Click **Open** (or double-click the file name).
3. Right click in the graph area and select *Edit imported data*.
4. In the *Select Imported Overlays* window, if the ASCII text file does not display, click **Import** to locate the file. Select the ASCII text file in the *Select Imported Overlays* window, then click **OK**. If an error message will display instead, verify that the .TXT file format is in the correct format.
5. To include the overlay data in a printed report, see ***Print Pore Size Distribution Overlay Data in Reports on the facing page***.

OVERLAY PORE SIZE DISTRIBUTION DATA USING COPY/PASTE

1. Open a source sample file and a target sample file; both should have a *Complete* status. The report will open to the interactive reports window.
2. In the source sample file, right click on the graph and select *Show Curve*. Deselect any differential curve data to hide them in the graph.
3. Right click in the graph area again and select *Copy Data*.
4. Change to the target sample file, right click the graph, and select *Paste data*. The graph now displays overlayed data from the source sample file.

   Typically, one new graph will appear showing both the cumulative and differential curves. To show or hide individual curves, right click the graph and select (or deselect) *Display imported data*.

5. Ensure that all parameter fields are set appropriately, then click **Paste**.
6. To include the overlay data in a printed report, see *Print Pore Size Distribution Overlay Data in Reports below*.

Print Pore Size Distribution Overlay Data in Reports

1. Open the sample file containing the overlay data and select *Advanced* from the drop-down list at the bottom of the window.
2. Click the *Report Options* tab.
3. In the *Selected Reports* list box, select the cumulative, differential, or incremental intrusion graph to show the imported distribution data, then click **Edit** (or double-click the selected report).
4. In the *Overlay* drop-down box, select *Imported*.
5. Click **OK** to close the window.
6. Click **Preview** on the *Report Options* tab. Click **Print** in the reports toolbar section to display print options.
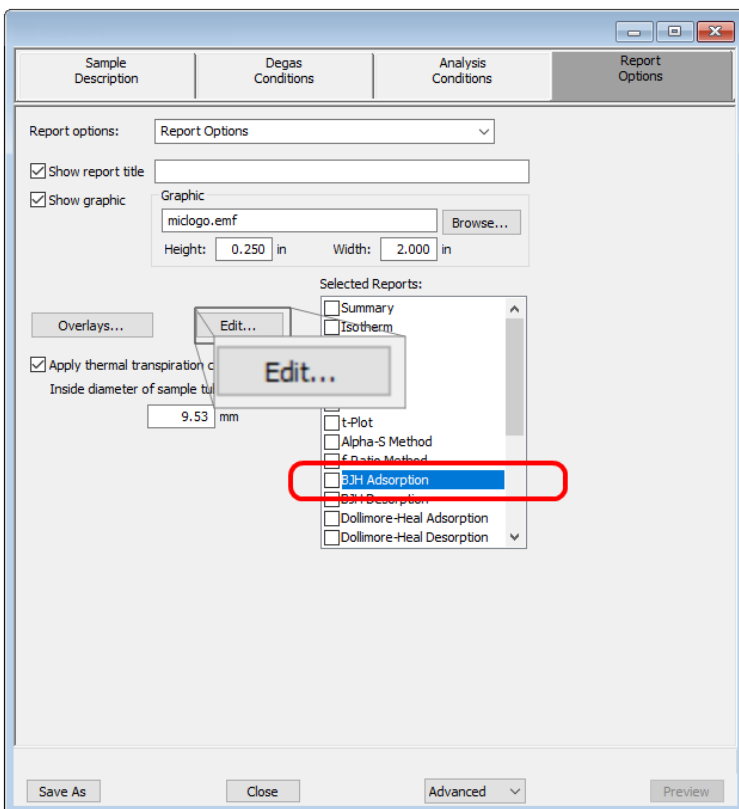
OVERLAY MULTIPLE SAMPLE FILES

CFR Note In 21CFR11 environments, this feature is applicable to members of the Developer group only.

To overlay the same type of graph on multiple samples:

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

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

5. On the *Report Options* tab, click **Overlays**.
6. On the *Plot Overlay Sample Selection* window, move up to 25 files from the *Available Files* box to the *Selected Files* box:
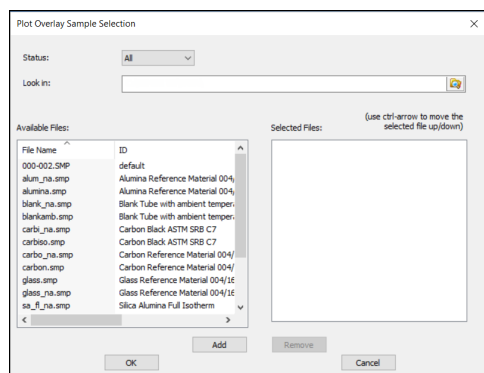
7. Click **OK**.
8. To view the report, click **Preview**.

Plot Overlay Sample Selection

Field	Description
Status *[drop-down box]*	Select the status of files to be combined.
Look in *[button]*	Click the **Browse** icon to change the file folder location.
Available Files *[selection]*	Lists files that meet the selected criteria. Select the files to be combined, then click **Add**. The selected files are moved to the *Selected Files* list box.
Selected Files *[selection]*	Lists the files selected to be combined. Click **OK** to combine the files.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

IMPORT ASCII PORE DISTRIBUTION DATA

IMPORT AN ASCII TEXT FILE USING GRAPH SHORTCUTS

1. Create an ASCII text file. See *Manually Enter Data on page 3 - 7*.
2. Open a report with a *Complete* status.
3. Select a pore-size distribution report from the drop-down list at the bottom of the window.
4. Right click on the graph and select *Edit imported data* on the shortcut menu.

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

5. To hide or show imported data, right click in the graph area and use the *Display imported data* option on the shortcut menu.

COPY/PASTE AN ASCII TEXT FILE USING GRAPH SHORTCUTS

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

COPY/PASTE GRAPH DATA FROM ANOTHER GRAPH

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

REPORT EXAMPLES

BET SURFACE AREA PLOT

BET SURFACE AREA

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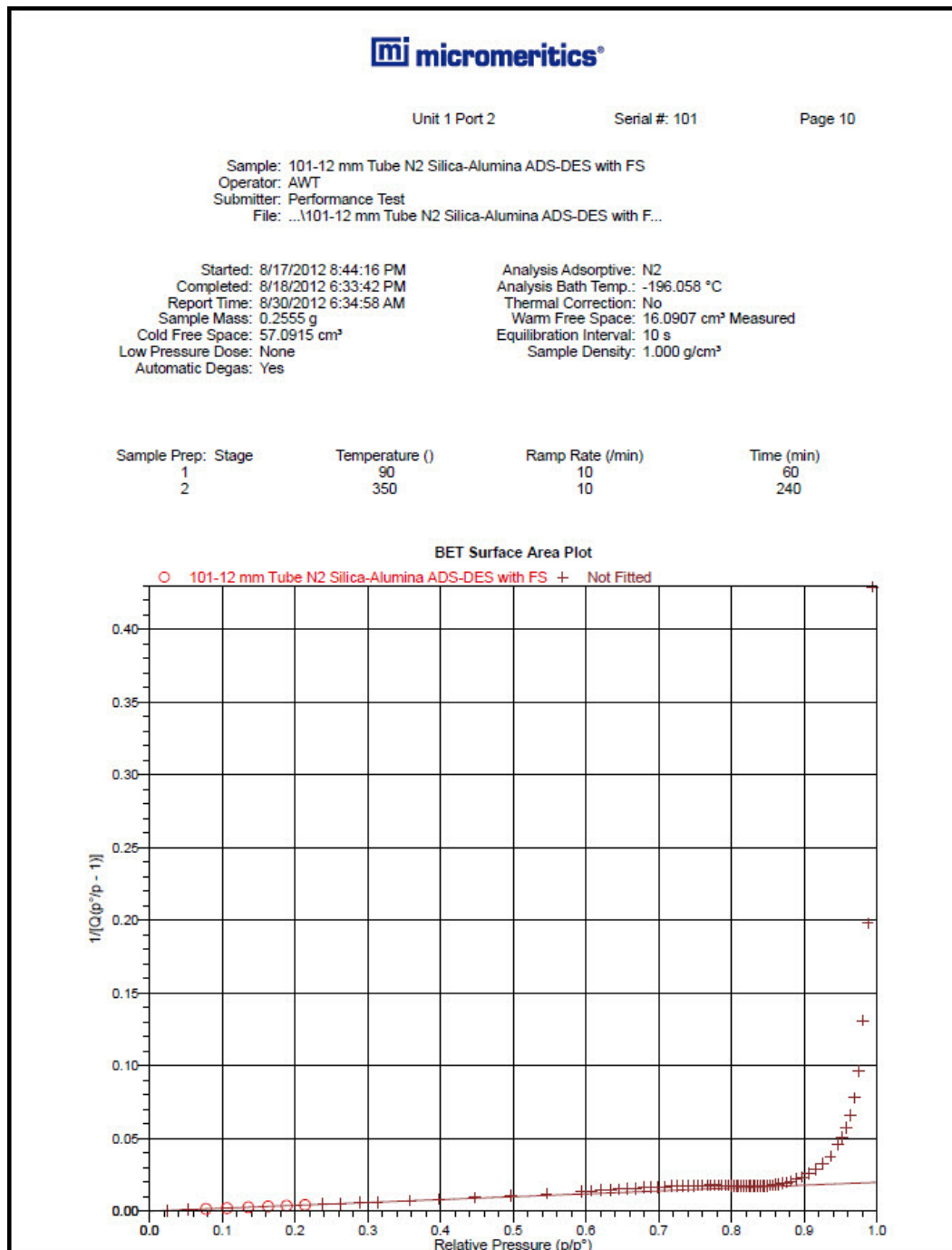
| Unit 1 Port 2 | Serial #: 101 | Page 9 |

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

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

BET Surface Area Report

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

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

BJH ADSORPTION - CUMULATIVE PORE VOLUME REPORT

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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
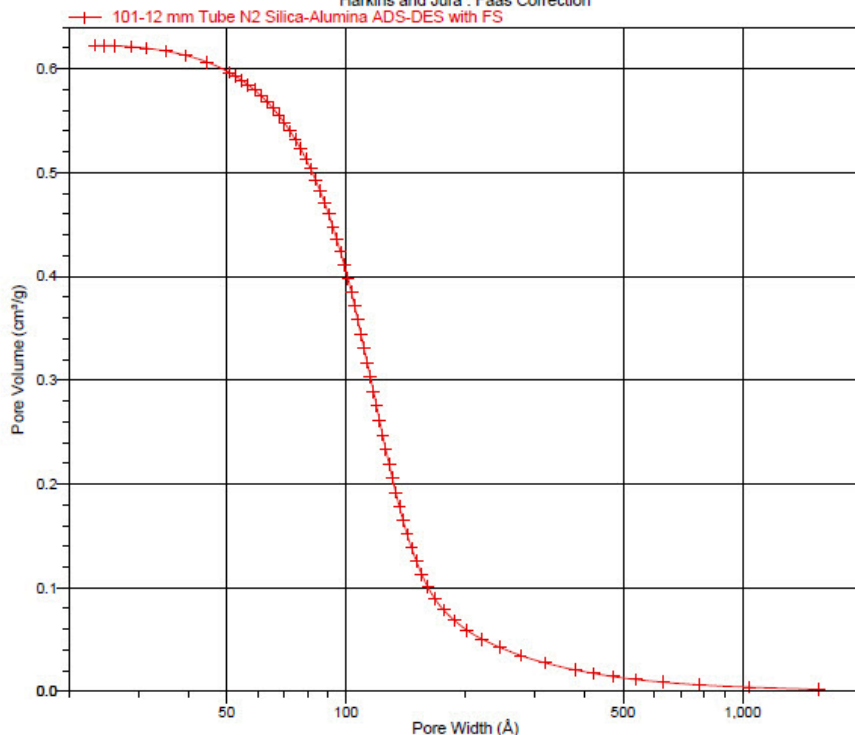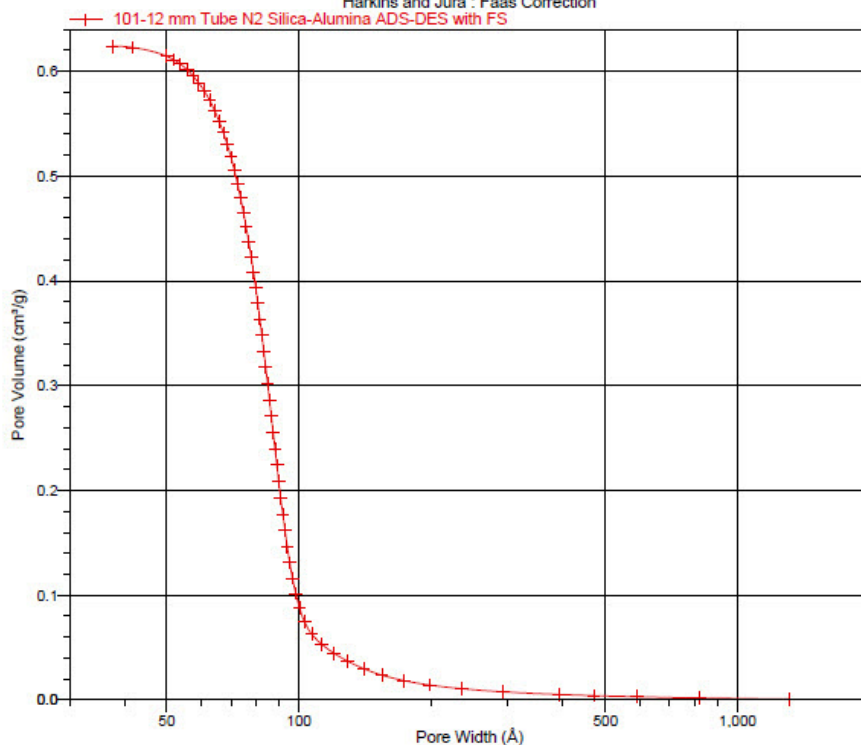File: ...\101-12 mm Tube N2 Silica-Alumina ADS-DES with F...

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
— 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS

BJH DESORPTION - CUMULATIVE PORE VOLUME REPORT

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ISOTHERM LINEAR PLOT

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Unit 1 Port 2	Serial #: 101	Page 8

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

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

Isotherm Linear Plot

—+— 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS - Adsorption
—○— 101-12 mm Tube N2 Silica-Alumina ADS-DES with FS - Desorption

t-Plot Report

Unit 1 Port 2	Serial #: 101	Page 11

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

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

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8 SELECTED REPORTS

CFR Note In 21CFR11 environments, this feature is applicable to members of the Developer group only.

To edit reports, open the *Sample* file then select the *Report Options* tab. Highlight the report name in the *Selected Reports* list box and click **Edit**.

Log in to your customer portal to access MicroActive Report Tutorials.
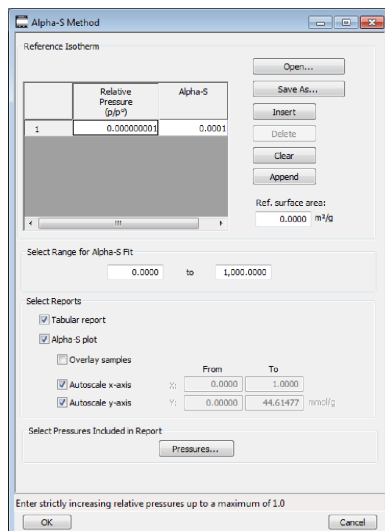
ADVANCED REPORT OPTIONS

See:

Advanced Reports - Python Module on page A - 1

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ALPHA-S METHOD REPORT OPTIONS

The *Alpha-S* plot converts the standard adsorption isotherm into a dimensionless isotherm using the quantity adsorbed at a relative pressure of 0.4.
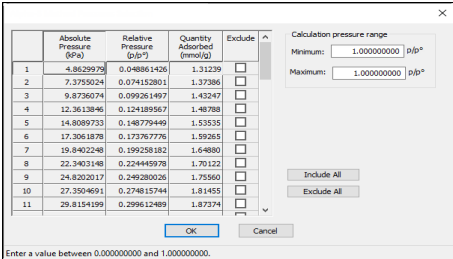
One predefined curve is located in the *Reference* file directory. Use the table buttons to enter relative pressure and the alpha-s values.
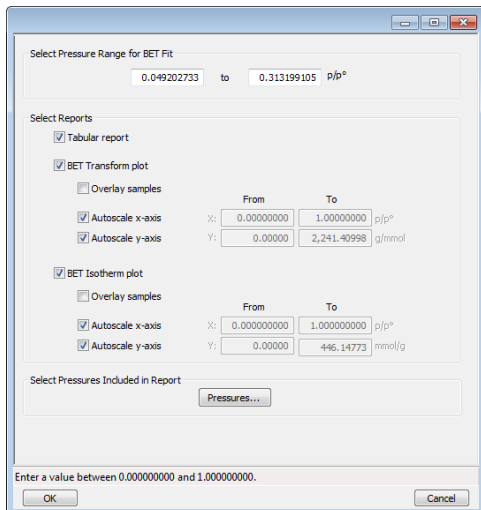
Alpha-S Method Reports

Field or Button	Description
Open *[button]*	Use to import values from an existing thickness curve (.ALS). 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.

Alpha-S Method Reports (continued)

Field or Button	Description
Pressures [button]	Use to select a pressure range for report calculations and points for exclusion from calculations. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
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 [group box]	Enter minimum and maximum relative pressures to determine the fit.
Selected Reports [group box]	**Alpha-S Plot.** Use to plot data in graph format. • **Autoscale x-axis.** The x-axis field shows the relative pressure. • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. • **Overlay samples.** Use to overlay sample files on the plot. **Tabular Report.** Use to have a tabular report of data generated.

> **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

BET SURFACE AREA REPORT OPTIONS

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

BET Reports

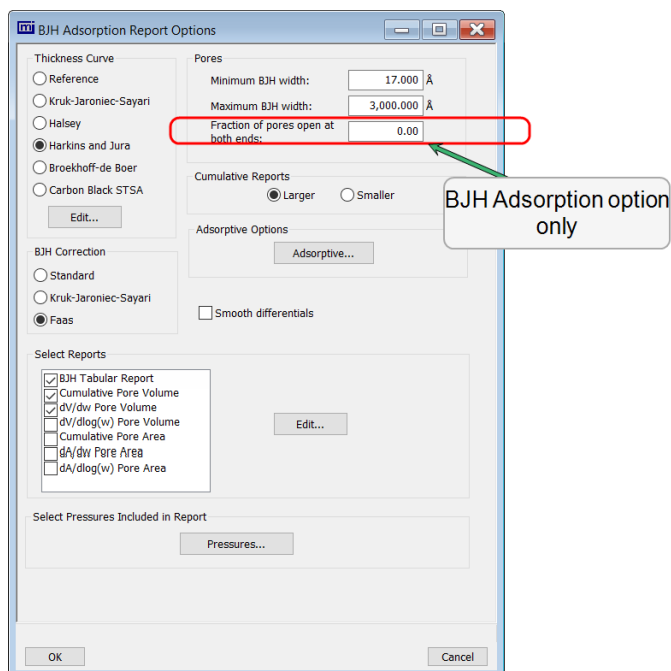| Field or Button	Description
Pressures *[button]*	This option is available when the sample file has a status of *Analyzing* or *Complete*. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table if not using the *Use Interpolation* option. **Exclude All.** Select to exclude all pressure points in the table.

BET Reports (continued)

Field or Button	Description
	Include All. Select to include all pressure points in the table.
	Insert Predefined. Click to insert a predefined (default) set of points into the report. *Use Interpolation* must be selected to enable this button. This button displays for BET reports only.
	Use Interpolation. Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only.
Select Pressure Range for BET fit *[text box]*	Enter values to indicate the fitted pressure range.
Selected Reports *[group box]*	**BET Isotherm plot.** Uses BET monolayer volume and constant to produce an isotherm.
	• **Autoscale x-axis**. Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET.
	• **Autoscale y-axis**. The y-axis field shows the quantity of gas adsorbed.
	• **Overlay samples.** Use to overlay sample files on the BET isotherm plot.
	BET Transform plot. Use to generate a traditional BET surface area plot used to determine monolayer volume and BET C constant.
	• **Autoscale x-axis**. Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET.
	• **Autoscale y-axis**. The y-axis field shows BET transformation.
	• **Overlay samples.** Use to overlay sample files on the BET transform plot.
	Tabular report. Use to have a table of measured and calculated values generated.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

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 *BJH Adsorption Report Options* and *BJH Desorption Report Options* are identical unless otherwise specified.
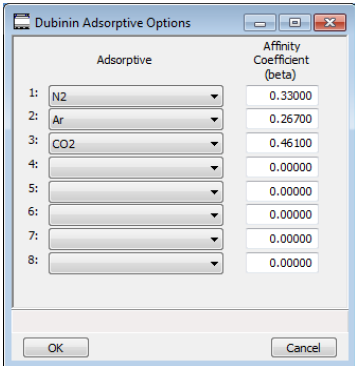
> An incomplete pore distribution may be generated if a thickness curve selection is not a good match for the sample being analyzed.
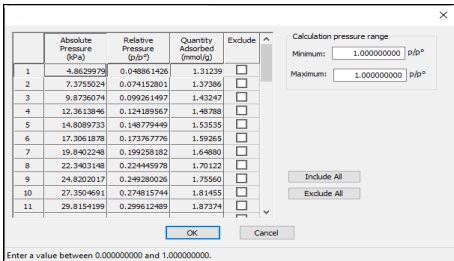

BJH Adsorption/Desorption Reports

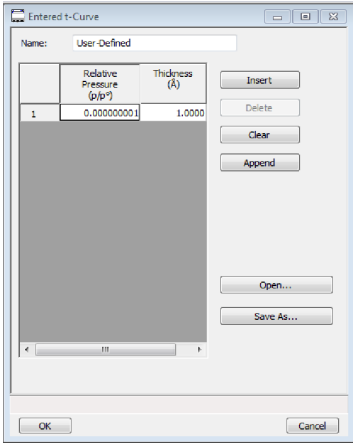| Field or Button	Description
Adsorptive *[button]*	Displays the *Adsorptive Options* window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.

BJH Adsorption/Desorption Reports (continued)

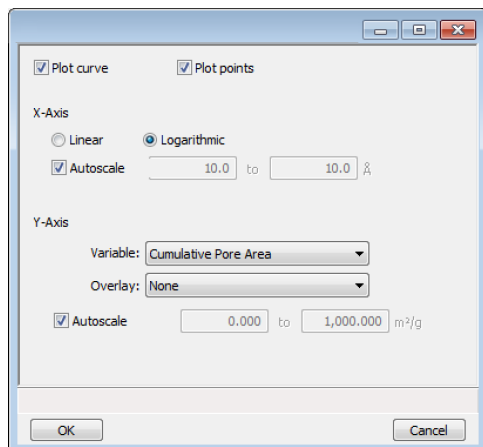| Field or Button	Description
BJH Correction *[group box]*	Select the type of correction to apply to calculations. The selected type will display in the report header. **Faas.** Good for statistical thickness curves. **Kruk-Jaroniec-Sayari.** Good for reference thickness curves. **Standard.** Uses original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

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BJH Adsorption/Desorption Reports (continued)

Field or Button	Description
Pressures *[button]*	Use to select a pressure range for report calculations and points for exclusion from calculations. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Select Reports *[group box]*	Select the report names to include in the report. Highlight the report name, then click **Edit** to modify report parameters.
Smooth differentials *[check box]*	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.
Thickness Curve *[group box]*	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed. **Reference.** Select *Reference*, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory.

BJH Adsorption/Desorption Reports (continued)

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

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

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BJH 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, then click **Edit**.
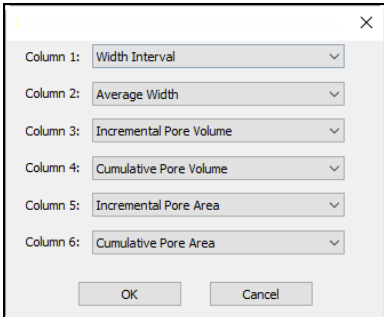
BJH Plot Reports

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

> **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**
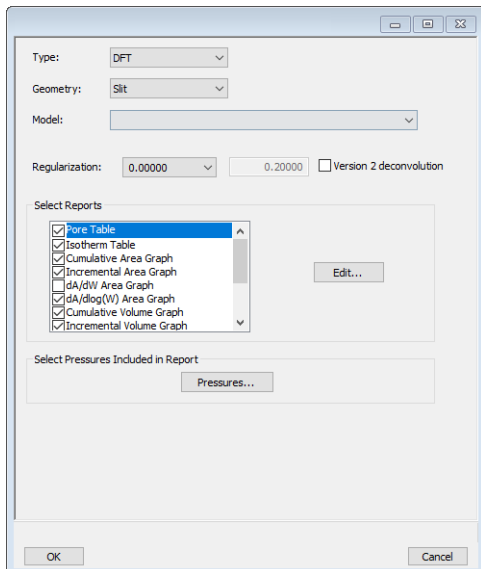
BJH Tabular Report Options
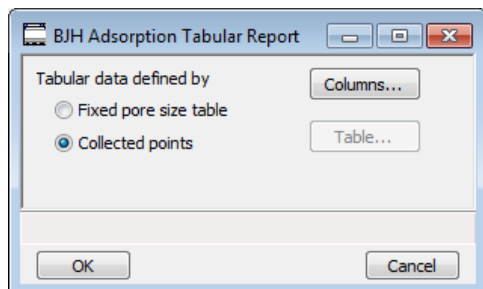
Highlight *BJH Tabular Report* in the *Selected Reports* list box on the *BJH Adsorption Report Options* window, then click **Edit** to specify the method of data reduction.
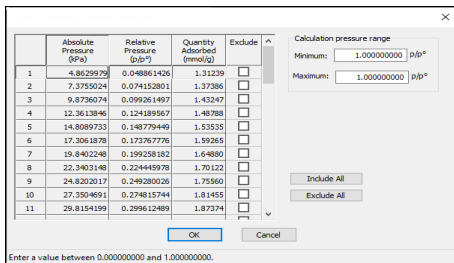
BJH Tabular Reports

Field or Button	Description
Collected points *[selection]*	Use to include all relative pressure points collected. Refer to the **Columns** button below.
Columns *[button]*	Select the data types to include in the report. *Column [n]* indicates the column order and data contents for the report.
Fixed pore size table *[selection]*	Use to specify exact pore sizes for volume or area data. Click **Table** to modify the fixed pore size table. Refer to **Table** and **Columns** buttons elsewhere in this table.
Table *[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.

✏️ **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**
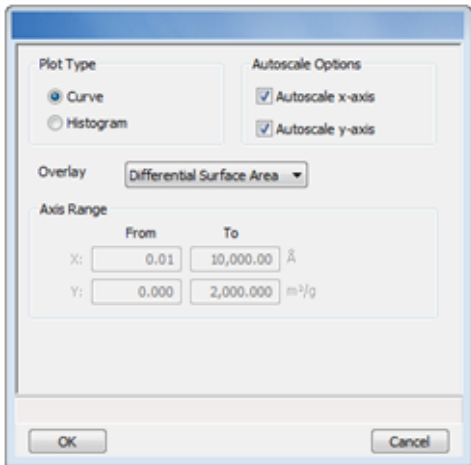
DFT PORE SIZE REPORT OPTIONS

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

DFT Pore Size Reports

Field or Button	Description
Geometry *[drop-down box]*	Select the pore shape.
Model *[drop-down 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.
Pressures *[button]*	Use to select a pressure range for report calculations and points for exclusion from calculations. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*.

DFT Pore Size Reports (continued)

Field or Button	Description
	Exclude All. Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Regularization *[drop-down 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.
Select 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. **Autoscale Options.** Use to autoscale the x-axis and/or y-axes. **Axis Range.** *From/To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and/or y-axes. • **x-axis.** Shows the pore size. • **y-axis.** Shows the area. **Overlay.** Select an overlay for the report. **Plot Type.** Select the method for data display.

DFT Pore Size Reports (continued)

Field or Button	Description
Type *[drop-down box]*	**Classical.** Model based on the Kelvin equation and thickness for determining the pore size distribution. See *DFT Models on page B - 1*. **DFT.** Model based on the density functional theory.
Version 2 deconvolution *[check box]*	If enabled, adds improved performance for nonzero values of the DFT regularization. The DFT results will be slightly better than when the checkbox is not selected and when using a regularization parameter. When the checkbox is not selected, the legacy behavior of the DFT calculation used by earlier versions of the application is produced.

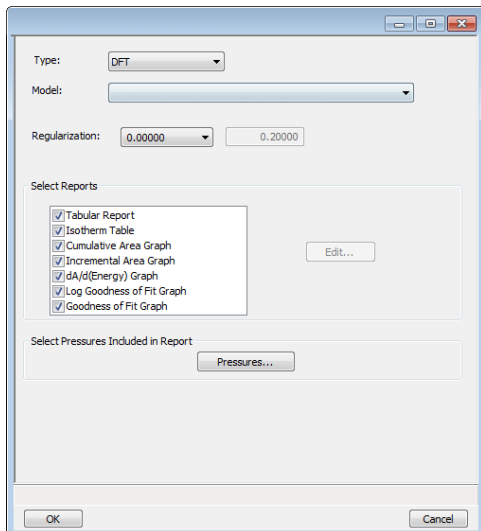> For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

DFT SURFACE ENERGY REPORT OPTIONS

> **See also:**
>
> *DFT Pore Size Report Options on page 8 - 12*

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

DFT Surface Energy Report Options fields and buttons are identical to the *DFT Pore Size Report Options*.

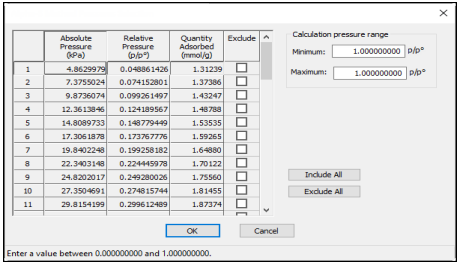## DOLLIMORE-HEAL ADSORPTION/DESORPTION REPORT OPTIONS

> **See also:**
>
> *BJH Adsorption/Desorption Report Options on page 8 - 6* for additional information on field and buttons for this report.

The *Dollimore-Heal Adsorption Report Option* and the *Dollimore-Heal Desorption Report Option* generate reports from both adsorption and desorption data.
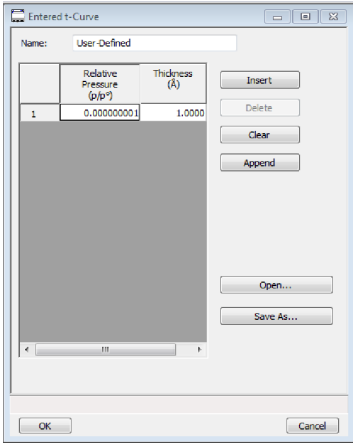
Dollimore-Heal Adsorption/Desorption Reports

Field or Button	Description
Adsorptive *[button]*	Displays the *Adsorptive Options* window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.

Dollimore-Heal Adsorption/Desorption Reports (continued)

Field or Button	Description
Cumulative Reports *[group box]*	**Larger.** Use to report the total volume found in pores larger than the current pore size. **Smaller.** Use to report the total volume found in pores smaller than the current pore size.
Pores *[group box]*	Enter the minimum and maximum diameter (radius or width) of pores to include in the BJH reports.
Pressures *[button]*	Use to select a pressure range for report calculations and points for exclusion from calculations. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Select Reports *[group box]*	Select the report names to include in the report. Highlight the report name, then click **Edit** to modify report parameters.
Smooth differentials *[check box]*	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.
Thickness Curve *[group box]*	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed. **Reference.** Select *Reference*, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory.

**Dollimore-Heal Adsorption/Desorption Reports (continued)**

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

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

DOLLIMORE-HEAL PLOT OPTIONS

> **See also:**
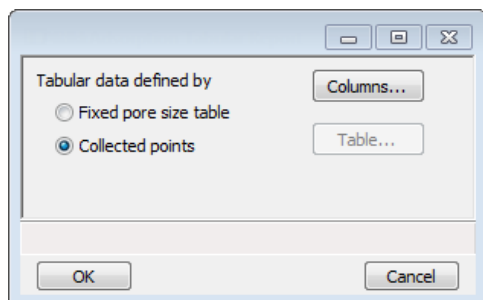>
> **BJH Plot Options on page 8 - 10**
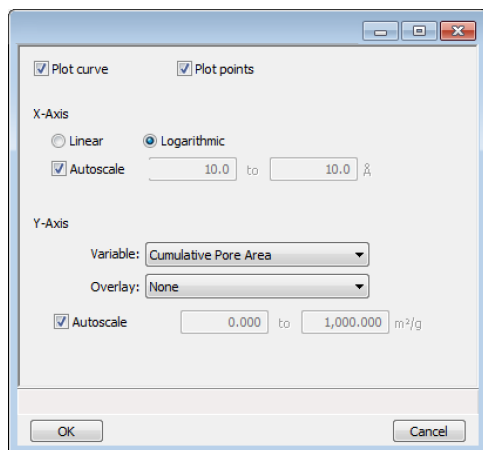
The fields for all plot options are identical for specifying plotting methods and customizing plots. Highlight any plot option in the *Selected Reports* list box in the *Dollimore-Heal Report Options* window, then click **Edit**. The fields and buttons for these reports are identical to the *BJH Plot Report Options*.

DOLLIMORE-HEAL TABULAR REPORT OPTIONS

> **See also:**
>
> **BJH Tabular Report Options on page 8 - 11**
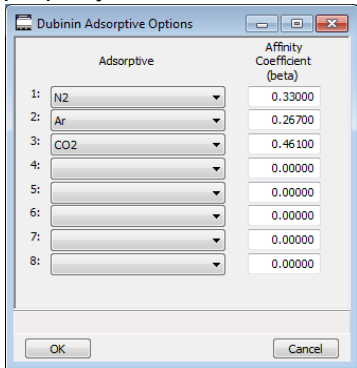
Tabular data defined by	
⊙ Fixed pore size table	Columns...
⦿ Collected points	Table...

OK   Cancel
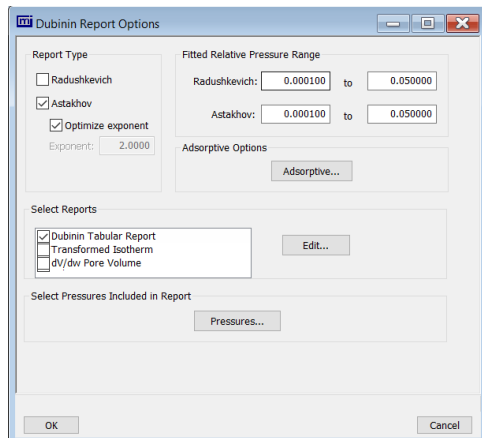
Dollimore-Heal Tabular Report Options are identical to the *BJH Tabular Report Options*.

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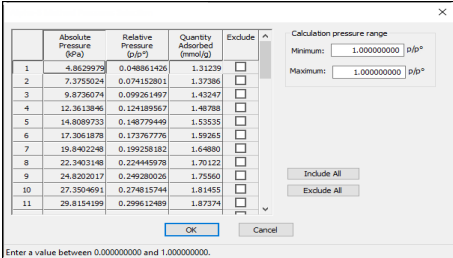
The *Dubinin* method provides pore volume distributions for microporous materials by making use of an expression for the adsorption potential.

Dubinin Reports

Field or Button	Description
Adsorptive *[button]*	Displays the *Adsorptive Options* window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.
Fitted Relative Pressure Range *[group box]*	Enter the minimum and maximum limits for Radushkevich or Astakhov relative pressures included in the line fit.

Dubinin Reports (continued)

Field or Button	Description
Pressures *[button]*	Use to select a pressure range for report calculations and points for exclusion from calculations. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Report Type *[group box]*	Select report types. If *Astakhov* is selected, either select *Optimize exponent* or enter an appropriate exponent value in the text box.
Select Reports *[group box]*	Select the reports to generate. Highlight the report, then click **Edit** to modify report options.

> **For fields and buttons not listed in this table, see** *Common Fields and Buttons on page 2 - 4*.

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DUBININ PORE VOLUME REPORT OPTIONS

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

This option plots differential pore volume as a function of pore width.
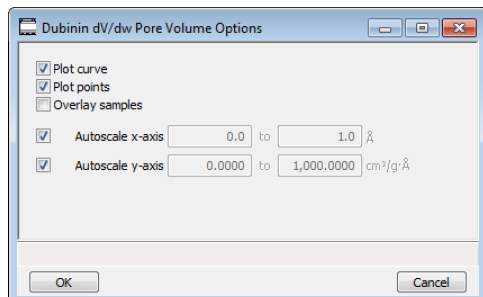
Dubinin Pore Volume Reports

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

> **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

DUBININ TABULAR REPORT OPTIONS

In the *Dubinin Report Options* window, highlight *Dubinin Tabular Report* in the *Selected Reports* list box, then click **Edit**. *Column [n]* indicates the column order and data contents for the report.
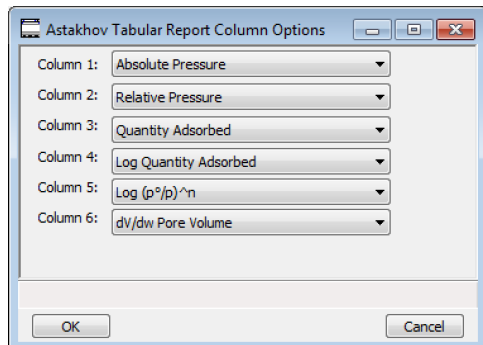
Log (p^o/p)^n. The value for *[n]* is the optimized exponent if *Optimize exponent* is selected on the *Dubinin Report Options* window. If not, then the value for *[n]* is the entered exponent value.

DUBININ TRANSFORMED ISOTHERM PLOT OPTIONS

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

The transformed Dubinin isotherm is the logarithm of quantity adsorbed as a function of the log of relative pressure raised to a power. Isotherms for which the Dubinin method is applicable produce straight lines when transformed in this way.
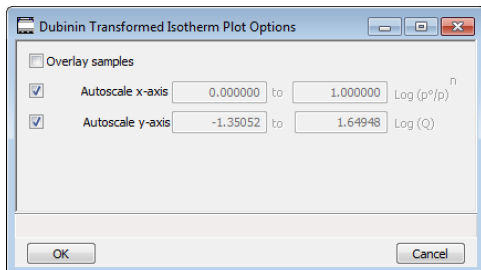
Dubinin Transformed Isotherm Plot Reports
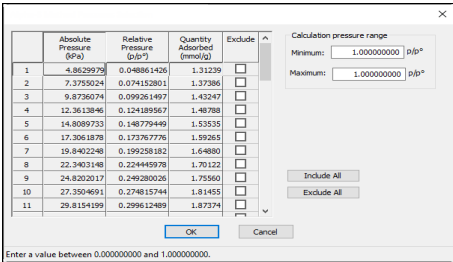
Field or Button	Description
Autoscale x-axis / Autoscale y-axis *[check box]*	Select an option to have the x- and/or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges. Deselect to enter beginning and ending values manually. **Autoscale x-axis.** Shows the quantity of gas adsorbed at standard temperature and pressure. **Autoscale y-axis.** Shows the log of relative pressure.
Overlay Samples *[check box]*	Use to overlay sample files on the plot.

> ✏️ **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

F-RATIO METHOD REPORT OPTIONS

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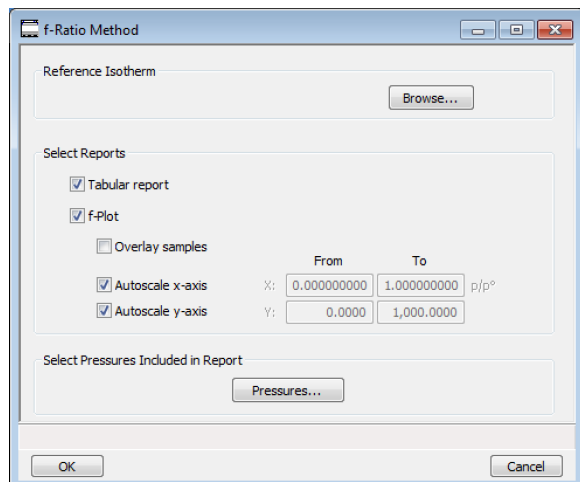
f-Ratio Reports

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

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f-Ratio Reports (continued)

Field or Button	Description
Reference isotherm *[group box]*	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.
Selected Reports *[group box]*	**Tabular Report.** Use to have a tabular report of data generated. ***f*-Plot.** Use to generate a normalized isotherm. ● **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. ● **Overlay samples.** Use to overlay sample files on the f-plot.

> **For fields and buttons not listed in this table, see _Common Fields and Buttons on page 2 - 4_.**

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**.

Freundlich Reports

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

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Freundlich Reports (continued)

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

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

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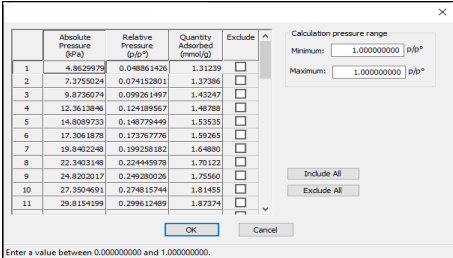
The *Horvath-Kawazoe* method plots individual peaks for different pore sizes even if the difference between one pore size and the next is only one angstrom (0.10 nm) or less.

Horvath-Kawazoe Reports

Field or Button	Description
Apply Cheng-Yang correction *[selection]*	Use to apply the Cheng-Yang correction to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath-Kawazoe derivation.
Interaction Parameter *[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 **Properties** to display the *Physical Properties* window). The interaction parameter is recalculated each time a parameter in the *Physical Properties* window is edited. **Entered.** Calculates using the value entered in the text box.
Pore Geometry *[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.

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Horvath-Kawazoe Reports (continued)
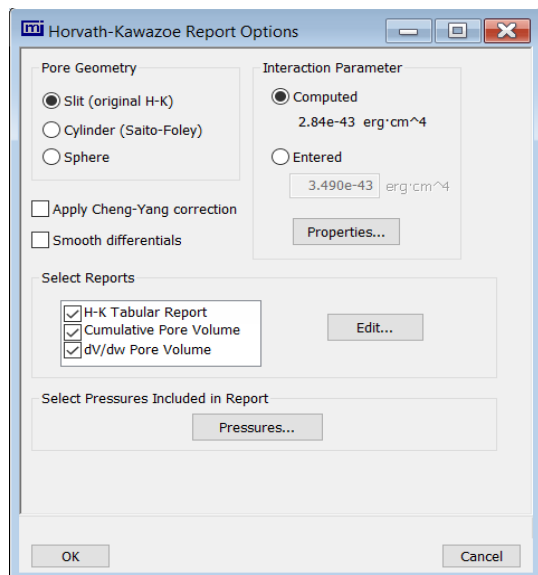
Field or Button	Description
Pressures *[button]*	Use to select a pressure range for report calculations and points for exclusion from calculations. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.
Properties *[button]*	Click to view or edit the constants describing the physical properties of the adsorbent and adsorptive. **Adsorbent**. Contains the parameters for the sample. If using *Computed* for the interaction parameter, all fields are enabled. If using *Entered*, only the values in the *Diameter* and *Diameter at zero energy* text fields may be edited. • **Density.** Enter the density per unit area of the sample. * • **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: $(2/5)^{1/6}$ × diameter. • **Magnetic susceptibility.** Enter the magnetic susceptibility of the sample. * • **Polarizability.** Enter the polarizability of the sample. * **Adsorptive.** Contains the parameters for the adsorptives. If using *Computed* for the interaction parameter, all fields are enabled. If using *Entered*, only the values in the *Diameter* and *Diameter at zero energy* text fields may be edited. • **Density.** Enter the density per unit area of the adsorptive. * • **Diameter.** Enter the diameter of the gas phase atom.

Horvath-Kawazoe Reports (continued)

Field or Button	Description
	• **Diameter at zero energy.** Enter the diameter of an atom at zero interaction energy: $(2/5)^{1/6} \times$ diameter. • **Magnetic susceptibility.** Enter the magnetic susceptibility of the adsorptive. * • **Mnemonic.** Select the mnemonic of the adsorptive gas in use. • **Polarizability.** Enter the polarizability of the adsorptive. * * Option is disabled if *Entered* is selected in the *Interactions Parameter* group box.
Select Reports *[group box]*	Select the types of reports to generate. Highlight the report, then click **Edit** to modify report parameters.
Smooth Differentials *[selection]*	Use to smooth all differential calculations, thus eliminating variations in the differential computation caused by noise in the input data.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

HORVATH-KAWAZOE PLOT OPTIONS

> **See also:**
>
> *BJH Plot Options on page 8 - 10* for additional information on fields and buttons for this report.
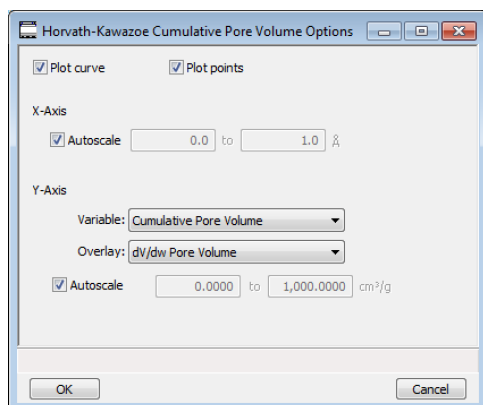
Highlight a plot option in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit** to customize the plotting method.
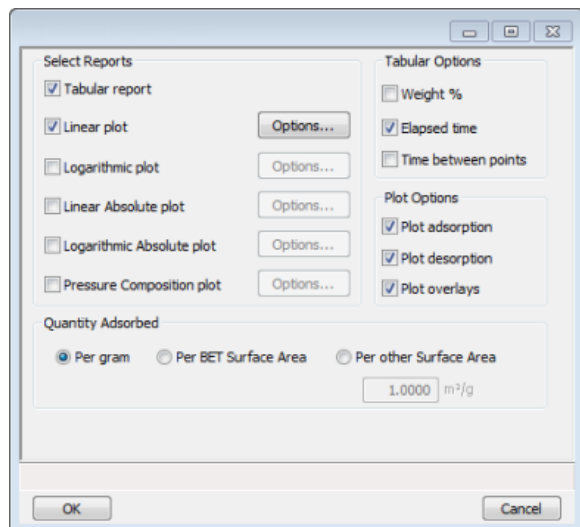
HORVATH-KAWAZOE TABULAR REPORT OPTIONS
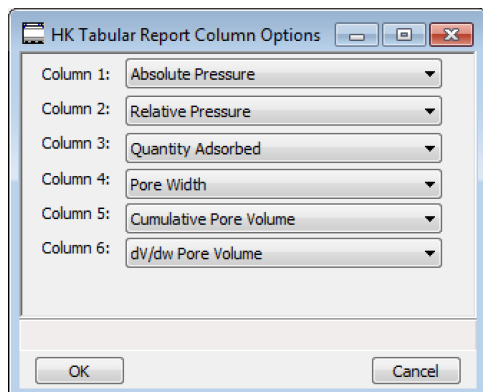
Highlight *H-K Tabular Report* in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit**. Select the data types to include in the report. *Column [n]* indicates the column order and data contents for the report.

ISOTHERM REPORT OPTIONS

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.

Isotherm Reports

Field or Button	Description
Options *[button]*	Click to display related linear plot options. All plot windows contain identical fields. **Autoscale x-axis.** Linear x-axes begin at zero. Logarithmic x-axes begin at an appropriate value. The x-axis field shows the relative or absolute pressure. **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. **Plot curve / Plot points.** Select to plot points on the graph.
Plot Options *[group box]*	Select the types of isotherm to plot.
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

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Isotherm Reports (continued)

Field or Button	Description
Selected Reports *[group box]*	Select each option to include on the final report. Click the Options button of a selected item to include plot curve, plot points, and to autoscale x- and y-axes.
Tabular Options *[group box]*	Select the options to include on the report. **Elapsed time.** Time elapsed during the analysis. **Time between points.** Time elapsed between points during the analysis. **Weight %.** Enter the mass percentage when plotting pressure composition.
Tabular Report *[group box]*	Select to include tabular data in the report.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

LANGMUIR REPORT OPTIONS

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.

Langmuir Reports

Field or Button	Description
Pressures *[button]*	This option is available when the sample file has a status of *Analyzing* or *Complete*. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points. **Calculation pressure range.** Enter the minimum and

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Langmuir Reports (continued)

Field or Button	Description
	maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table. **Use Interpolation.** Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only.
Select Pressure Range for Langmuir fit *[group box]*	Enter values to indicate the fitted pressure range.
Selected Reports *[group box]*	**Langmuir Isotherm Plot.** Uses the Langmuir monolayer volume and constant to produce an isotherm. • **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. • **Overlay samples.** Use to overlay sample files on the Langmuir isotherm plot. **Langmuir Transform Plot.** Use to generate a traditional Langmuir surface area plot used to determine monolayer volume constant. • **Autoscale x-axis.** Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir. • **Autoscale y-axis.** The y-axis field shows Langmuir transformation. • **Overlay samples.** Use to overlay sample files on the Langmuir transform plot.

> **For fields and buttons not listed in this table, see** *Common Fields and Buttons on page 2 - 4*.

MP-METHOD REPORT OPTIONS

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

MP-Method Reports

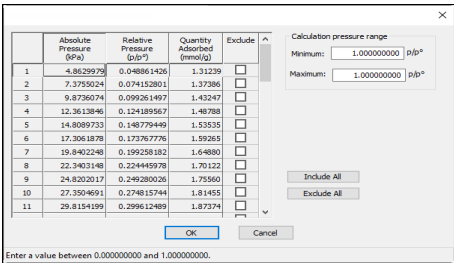| Field or Button	Description
Pressures *[button]*	Use to select a pressure range for report calculations and points for exclusion from calculations. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used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

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MP-Method Reports (continued)

Field or Button	Description
Select Reports *[group box]*	Select the reports to generate. Highlight the report, then click **Edit** to modify report options.
Thickness Curve *[group box]*	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve.

> **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

MP-METHOD PLOT REPORT OPTIONS

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

MP Method Plot Reports

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

> ![pencil icon] **For fields and buttons not listed in this table, see** *Common Fields and Buttons on page 2 - 4*.

MP-Method Tabular Report Options

In the *MP-Method Report Options* window, highlight *MP Tabular Report* in the *Selected Reports* list box, then click **Edit**. *Column [n]* indicates the column order and data contents for the report.
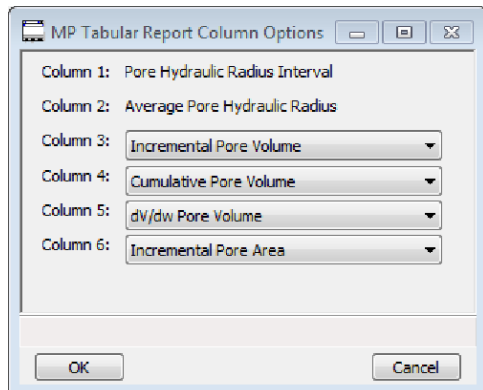
> The MP Method reports hydraulic radius only. If Pore size in diameter is selected on the Unit Selection window, pore size in radius will be reported.

NLDFT Advanced PSD Report

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

The *NLDFT Advanced PSD* report option provides the same calculations as the DFT Pore Size report option and more. The NLDFT report compares two sample files. The models that can be selected are restricted to only those models which have the same analysis temperature and analysis gas as the sample file that is open. For instance, if the sample file was analyzed with N2 at 77 degrees Kelvin, then only the N2 DFT models at 77 degrees Kelvin will be available in the *Model* drop-down list.
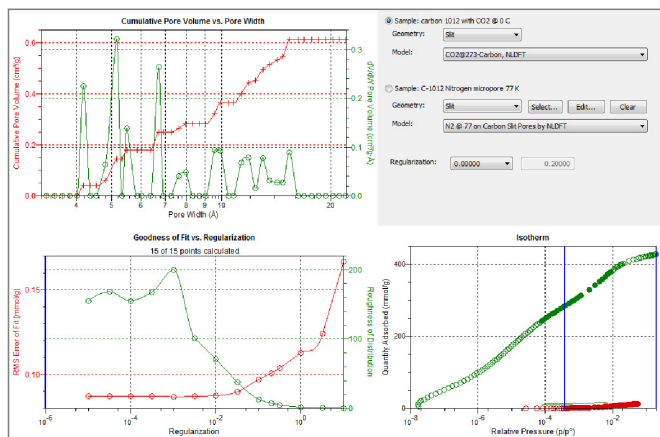
The model curve fit is shown in the lower right quadrant along with the adsorption isotherm. This curve fit is updated each time the calculation parameters change (selection of isotherm data points, choice of model, choice of regularization parameter).

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

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

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

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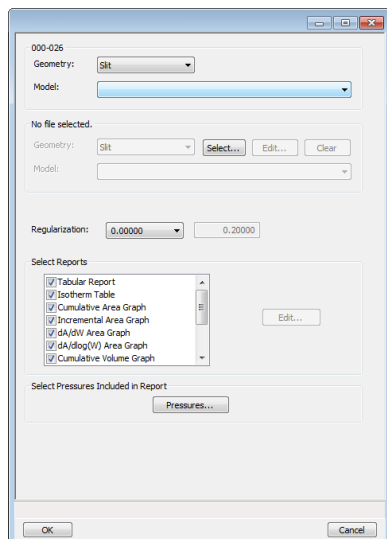
NLDFT Advanced PSD Reports

Field or Button	Description
Geometry [drop-down box]	Select the pore shape.
Model [drop-down 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.
Regularization [drop-down 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 mass for the smoothing during deconvolution. Larger values produce more smoothing.
Select Reports [group box]	Use to select the second sample file.

> For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

To run the NLDFT report:

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

First selected sample file

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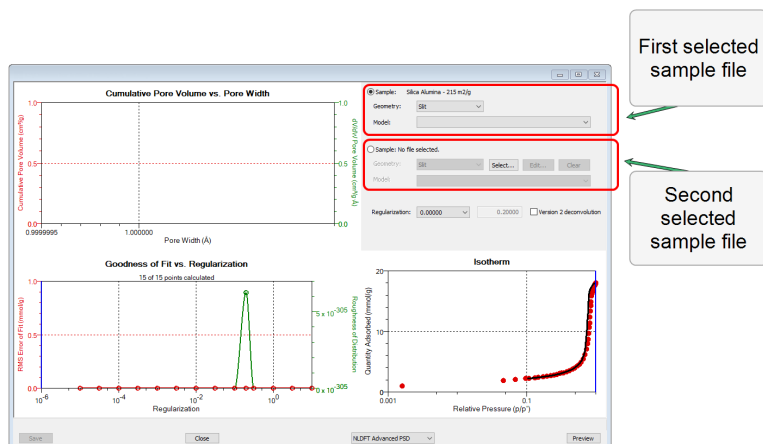
a. Select the *Geometry* and *Model* from the drop-down lists for the first sample file.

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

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

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

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

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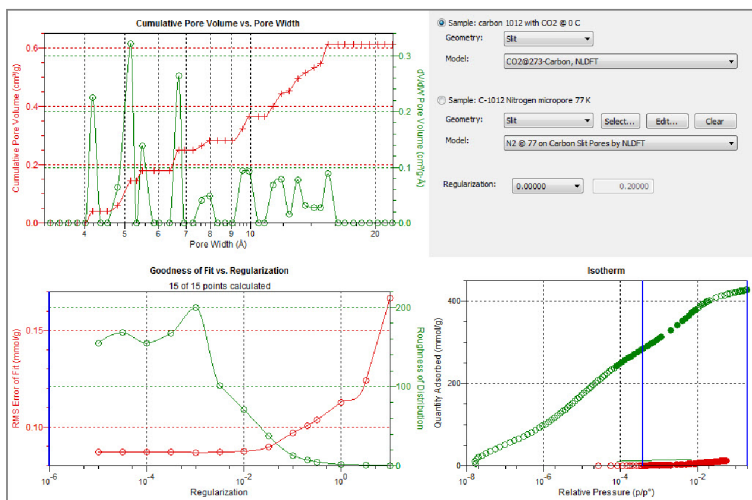
OPTIONS REPORT

Lists the conditions used to perform the analysis— such as:

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

> Options reports cannot be edited.

SAMPLE AUDIT TRAIL REPORT

CFR Note	For 21CFR11 environments only.

This report lists all changes and comments that have been applied to sample files with a *Completed* status.

SAMPLE LOG REPORT

CFR Note	Not applicable to 21CFR11 environments. See ***Sample Audit Trail Report above***.

This report provides information on:

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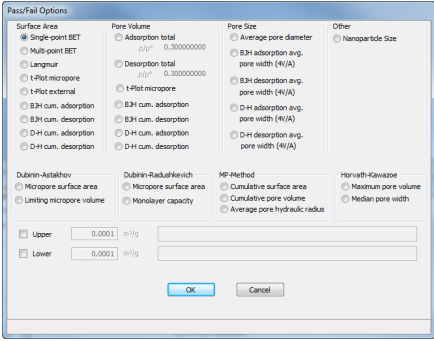
SUMMARY REPORT OPTIONS

The *Summary Report* for physical adsorption analyses provides a condensed summary of selected data results.
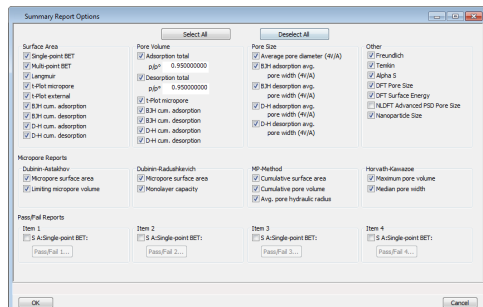
In the *Pore Volume* group box, if *Adsorption total* or *Desorption total* is selected, the *p/p^o* field is enabled. Enter the relative pressure used to calculate the total pore volume.

Summary Report

Field or Button	Description
Item [n] *[selection]*	Use to enable the first *Pass/Fail* item. Until the *Summary Report* is selected, *S A Single-point BET* will be displayed by default. When selected, click **Pass/Fail**, then select pass/fail criteria options. **Pass/Fail [n].** Click to display the *Pass/Fail Options* window for selection of pass/fail criteria. **S A: Single-point BET.** Use to enable **Pass/Fail [*n*]** in the *Item [n]* group box.

Summary Report (continued)

Field or Button	Description
	Upper/Lower. Specify upper and lower limits for the selected parameter. A range can be left open by not selecting the limit. In the text box to the right of *Upper / Lower*, enter operator instructions to be displayed if a failure is encountered.
Select All / Deselect All *[button]*	Selects (or deselects) all options.

> ✏️ **For fields and buttons not listed in this table, see** *Common Fields and Buttons on page 2 - 4*.
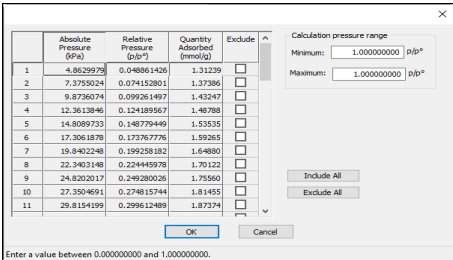
The *Summary Report* for dynamic analyses provides a condensed summary of the peaks and selected analysis parameters.

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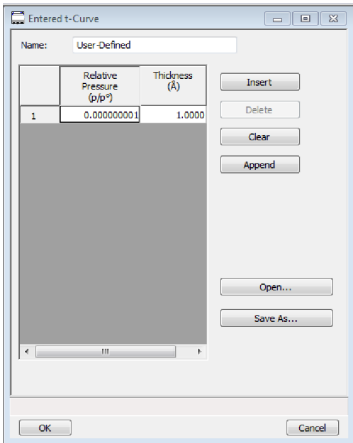
The *t*-Plot calculation allows quantitative analysis of the area and total volume ascribed to micropores. Matrix area (the area external to micropores) is directly determined and often proves to be a valuable way of characterizing complex mixed materials.

t-Plot Reports

Field or Button	Description
Fitted thickness range *[text box]*	Enter the minimum and maximum thicknesses (in angstroms or nano-meters) to include in the thickness curve. Go to ***Options > Units*** to specify default units.
Pressures *[button]*	Use to select a pressure range for report calculations and points for exclusion from calculations. **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table.To exclude a point from the calculations used to generate the report, select *Exclude*. **Exclude All.** Select to exclude all pressure points in the table. **Include All.** Select to include all pressure points in the table.

t-Plot Reports (continued)

Field or Button	Description
Selected Reports *[group box]*	**Tabular Report.** Use to have a tabular report of data generated. **t-Plot.** Use to have a graphical representation of data generated. • **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. • **Overlay samples.** Use to overlay sample files on the *t*-plot.
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.
Surface Area *[group box]*	Select the surface area value used for thickness calculations. BET is the most commonly used option.
Thickness Curve *[group box]*	Select the thickness curve, then click **Edit** to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option. **Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.** Select the thickness curve option, then click **Edit**. Modify the equation for the selected curve as needed. **Reference.** Select *Reference*, then click **Edit** to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the *Reference* directory.

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t-Plot Reports (continued)

Field or Button	Description
	To import values from an existing thickness curve (.THK file), click **Open**, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space or a tab.
t-Plot *[check box]*	Use to have a graphical representation of data generated. **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. **Overlay samples.** Use to overlay sample files on the *t*-plot.

> **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

TEMKIN REPORT OPTIONS

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

Temkin Reports

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

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Temkin Reports (continued)

Field or Button	Description
Selected Reports *[group box]*	**Tabular Report**. Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample. **Temkin Isotherm plot.** Overlays the Temkin isotherm with the analysis data. • **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. • **Overlay samples.** Use to overlay sample files on the isotherm plot. **Temkin Transform plot.** Plots a linear form of the Temkin transform plot. • **Autoscale x-axis**. The x-axis field shows the logarithm of pressure (ln). • **Autoscale y-axis.** The y-axis field shows the quantity of gas adsorbed. • **Overlay samples.** Use to overlay sample files on the transform plot.
Specify differential heat of adsorption *[check box]*	Select and enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all Temkin constants.
Specify monolayer capacity *[check box]*	Select and enter the monolayer capacity of the sample.

For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.

VALIDATION REPORT OPTIONS

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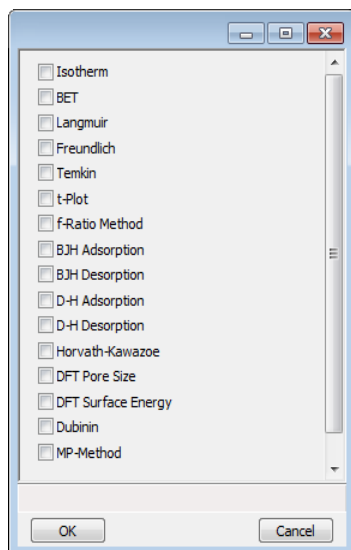
Blank Page

9 DIAGNOSTICS
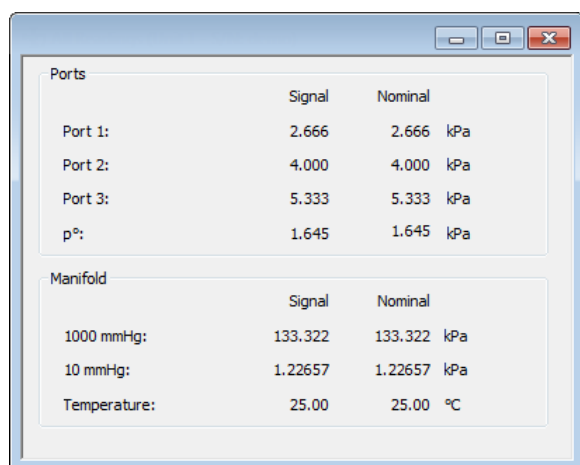
Unit [n] > Diagnostics

Use to display diagnostic readings, start diagnostic tests, and open saved diagnostic reports. Each test generates a file to the default directory name and path of *...\...\Service\userdiag* unless another directory name was specified. These reports can be sent to a Micromeritics Service Representative for examination.

SHOW ALL READINGS

Unit [n] > Diagnostics > Show All Readings

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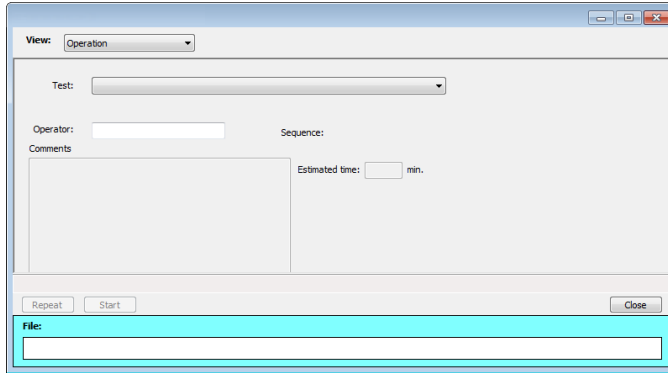
START DIAGNOSTIC TEST

Unit [n] > Diagnostics > Start Diagnostic Test

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

Start Diagnostic Test

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

> **For fields and buttons not listed in this table, see** *Common Fields and Buttons on page 2 - 4*.

10 CALIBRATION

Unit [n] > Calibration

> Service Test Mode is required for this test. See *Service Test Mode on page 11 - 31*.

A calibration file was created specifically for the analyzer and included with the accessories. It is not necessary to recalibrate the system unless it seems out of calibration.

Disabled calibration menu options can be accessed only with the assistance of an authorized Micromeritics Service Representative. Calibrations can be saved to a file and reloaded later.

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

A reference material analysis can be run (if necessary) to ensure the highest quality data. Each kit contains reference material and an instruction booklet. Follow the instructions in the reference material booklet to perform the analysis and review the results.

Generally it will not be necessary to change the data in the calibration file. However, if a condition occurs during the operational verification that requires changes to the calibration data, changes should be saved in a file. Calibration data files are retained in the analyzer history file and can be reloaded in the event that calibration data becomes corrupt.

PRESSURE OFFSET

Unit [n] > Calibration > Pressure Offset

This procedure evacuates the system and zeroes the pressure transducers. This calibration should only be performed by qualified service personnel. In order to perform this procedure, sample tubes must be attached to each port.
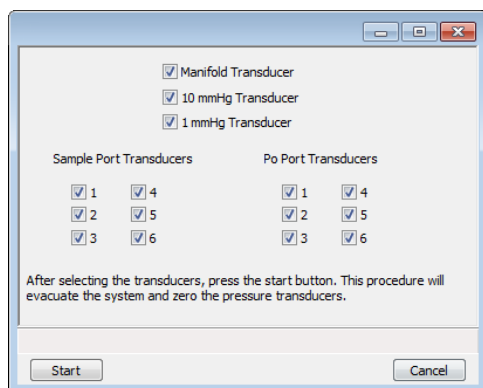
1. Install a blank sample tube or small plug on each applicable port.
2. Ensure that all applicable transducers are selected, then click **Start**. Click **OK** when the process is complete.

MATCH TRANSDUCERS

Unit [n] > Calibration > Match Transducers

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

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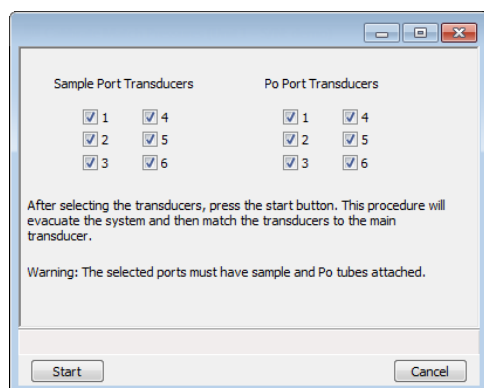
---

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

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SERVO VALVE

Unit [n] > Calibration > Servo Valve

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

> Ensure the pressure transducer has been calibrated before performing this procedure. Go to *Unit [n] > Unit Configuration* and view the calibration information. Contact your Micromeritics Service Representative if calibration dates are not listed.
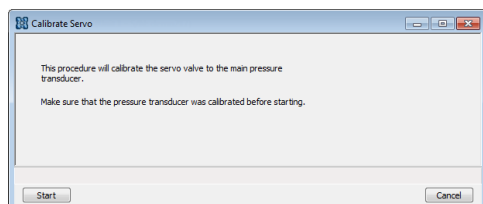
Click **Start**. The window closes when the calibration is complete. Click **Cancel** to stop the calibration process.

LOAD CALIBRATION FROM FILE

Unit [n] > Calibration > Load from File

Use to load a previously saved calibration file.

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

> Changing the calibration may affect the analyzer's performance.

SAVE CALIBRATION TO FILE

Unit [n] > Calibration > Save to File

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

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

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

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11 TROUBLESHOOTING AND MAINTENANCE

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. When unexpected results occur, some common operational problems not indicated on the window and their respective causes and solutions are provided:

Log in to your customer portal to access error messages. Parts and accessories can be found online at www.Micromeritics.com.

Most operational problems are caused by the following:

- 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

Elevator cannot be raised or lowered.

Cause: Dewar elevator stuck.

Action: Check for possible obstruction to elevator movement.

Elevator is noisy.

Cause: The elevator screw may need greasing.

Action: Contact your Micromeritics Service Representative.

Analysis valves cannot be operated.

Cause: Cable from computer to the analyzer is loose.

Action: Ensure the cable is seated properly.

Vacuum pump gurgles continuously.

Cause A: Sample tube connector nut is loose.

Action A: Turn the sample tube connector nut clockwise to tighten.

Cause B: Sample tube fitting is loose.

Action B: Tighten the fitting securely with a wrench.

Cause C: Sample tube O-ring is worn or cracked.

Action C: Replace the sample tube O-ring. See *Replace the Sample Tube O-ring on*

Cause D: Sample tube is cracked.

Action D: Replace with a new sample tube.

Cause E: No sample tube is loaded on a selected port.

Action E: Enure the port valve is closed. Install a plug or empty sample tube on the port.

Cause F: A gas inlet valve is open while the vacuum valve is open.

Action F: Enable manual control, then use the analyzer schematic to close the gas inlet valve. See *Enable Manual Control on page 11 - 19*.

Vacuum gauge shows reading above 20 mmHg, even after extended pumping through unrestricted valve with analysis or degas ports closed.

Cause A: **Oil-based Pump Only.** Vacuum pump oil is low, causing ineffective evacuation.

Action A: Add or change vacuum pump oil. Add oil to proper level according to oil level window.

Cause B: **Oil-based Pump Only.** No power to the vacuum pump.

Action B: Check the pump power plug, power switch, and line circuit breaker.

Cause C: **Oil-based Pump Only.** Filter in port being used is dirty.

Action C: Replace filter in port. See *Replace the Analysis Port Filter on page 11 - 23*.

Cause D: **Oil-based Pump Only.** The alumina in the oil vapor trap is holding moisture.

Action D: Replace or dry the alumina. Log in to your customer portal to access the Vacuum Pump Guide for information on replacing and drying alumina.

Cause E: **Oil Free Pump Only.** Filter in port being used is dirty.

Action E: Replace filter in port. See *Replace Port Filters on page 11 - 23*.

Cause F: **Oil Free Pump Only.** High vacuum pump may have timed out.

Action F: Power OFF the high vacuum pump, then power if back ON.

Cause G: **Oil Free Pump Only.** No power to the vacuum pump.

Action G: Check the pump power plug, power switch, and line circuit breaker.

Cause H: **Oil Free Pump Only.** The diaphragm(s) in the pump is worn or damaged.

Action H: Contact your Micromeritics Service Representative.

GUIDELINES FOR CONNECTING GASES

- Place gas cylinders within 6 feet (2 m) of the gas inlets of the analyzer. Using gas line extenders on gas cylinders located in remote areas may degrade gas quality and reduce pressure. Gas lines are typically five to six feet long. Place the cylinders close enough to allow for proper connection at the analyzer inlet.
- Use a retaining strap (or other appropriate tether) to secure the gas cylinder.
- Always use the gas lines provided with the analyzer. It is very important that proper gas lines are used with the analyzer.

    - **Do not use** polymer tubing for the gas line.
    - **Do not use** flexible gas lines. Some flexible lines may appear to be appropriate, such as those with a herringbone covering, but the line may be coated internally with a polymer.

- Long gas lines, such as those used with gas cylinders placed in remote areas, must be evacuated for an extended period of time to remove ambient gases. When possible, avoid placing gas cylinders in remote locations. It is always best to have gas cylinders located near the analyzer.
- Carefully route the gas lines from the cylinder to the analyzer avoiding overlapping or entangling gas lines. This will help avoid confusion when maintenance is required.
- Label the gas line at the analyzer inlet for proper identification and maintenance.
- Replace gas cylinders before gas is depleted. It is best to replace a gas cylinder when the pressure reads approximately 500 psi (3500 kPa) on the high pressure gauge. Contaminants absorbed to the walls of the cylinder will desorb as the pressure decreases.
- Ensure the gas cylinder is closed before connecting to the analyzer.

CLEAN AND VERIFY THE GAS LINE

> **CAUTION**
>
> Only evacuate the gas lines back to the gas cylinder if the gas lines are within 12 feet of the analyzer. Do not perform this procedure if the gas lines connected to the analyzer are longer than the 6 ft lines shipped with the analyzer.

There are two methods for cleaning and verifying gas lines:

- use the diagnostic test. See *Software Diagnostic Test to Clean and Verify Gas Lines below*, or
- perform the test manually. This method is less time consuming but should only be used by experienced service personnel. See *Manual Method to Clean and Verify Gas Lines on page 11 - 8*.

SOFTWARE DIAGNOSTIC TEST TO CLEAN AND VERIFY GAS LINES

Unit [n] > Diagnostics > Start Diagnostic Test

Always clean the gas lines and verify there are no leaks at the connections after a gas cylinder is connected. This test examines the gas line from the analyzer to the gas cylinder, then from the analyzer to the regulator shutoff valve. A report is generated at the completion of the test to verify that it has passed or failed. Causes and corrective action for a failure are provided.

Before beginning, confirm that the state for valves and the low-pressure gauge are as follows:
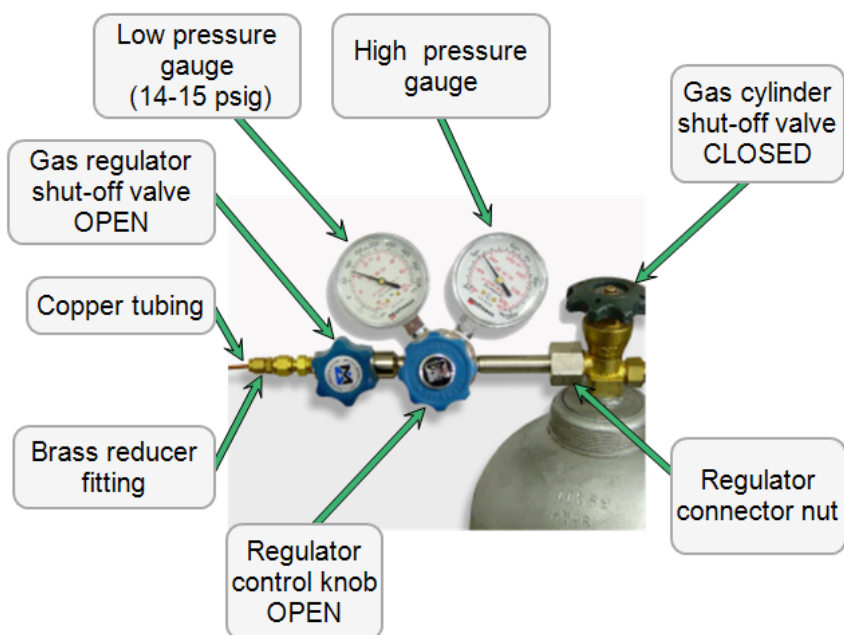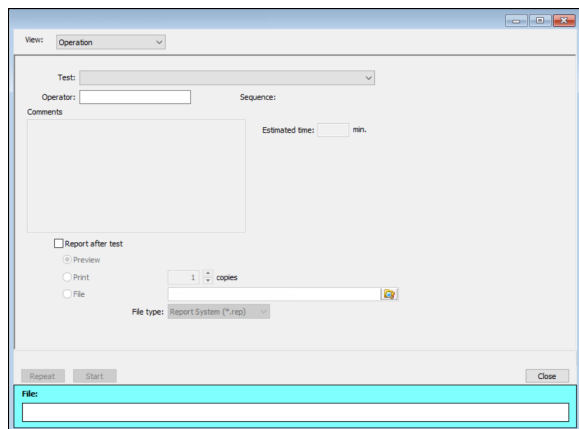
1. Select *Clean and Verify Analysis Gas Line [n] Test Rev [n]* in the *Test* field. The length of time a test will run is also indicated on the window. The *Sequence* field indicates the file created as a result of this test.

2. In the *Operator* field, enter either the name or initials of the operator performing this test.

3. Resize the window (if necessary) to display the *Report after test* option, then select *Preview* as the destination. Click **Start**.

4. From the *View* drop-down list, select either *Operation*, *Instrument Log*, or *Instrument Schematic*.

5. The following series of prompts display on the window requiring operator response:

   a. This is the gas line clean and leak check test for inlet port [*n*]. Inlet ports being tested must be connected to a gas cylinder according to the user manual. A Nupro isolation valve should be installed on the line between the analyzer and the regulator.

   b. The test starts with a manual leak check (requires Snoop or equivalent, and IPA), then the line and regulator are evacuated for 20 minutes for cleaning. Next, the leak rate of the gas line is determined.

   c. With the regulator set to 15 psig, open the cylinder, regulator shutoff valve, and isolation valve. Check each joint for bubbles with Snoop or equivalent. If a joint is leaking, attempt tightening (without over-tightening) or replace ferrules.

   d. When there are no leaking joints, use IPA to remove water from each joint, then wipe dry.

   e. Close the gas cylinder valve. Leave the regulator shutoff and isolation valves open.

   f. User will be needed in 30 minutes to close the isolation valve. Click **OK** to begin automated testing.

6. A popup window indicates the test is complete. Click **OK**. The reports display.
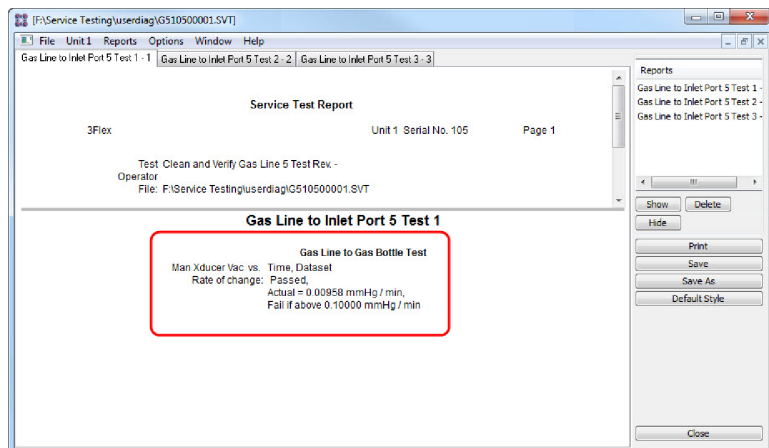
7. Click each tab across the top of the window and look for a reading of *Passed*. A *Passed* reading indicates all valves are in a proper state for operation. If any test shows a *Failed* reading, refer to the following table to help determine the location of the gas leak.

Tab	Test	If *Failed* status, then...
Gas Line to Inlet Port [*n*] Test 1	Gas Line to Gas Bottle Test	This test will show a reading of *Failed* if any of the other tabs has a *Failed* reading. Correct the failed connection and rerun the test.
Gas Line to Inlet Port [*n*] Test 2	Gas Line to Isolation Valve Test	Check for a leak between the gas line and the isolation valve. Correct the problem and rerun the test.
Gas Line to Inlet Port [*n*] Test 3	Isolation Valve To Bottle Leak Rate	Check for a leak between the isolation valve and the gas cylinder. Correct the problem and rerun the test.

If the *Fail if above* field indicates *Failed*, one or more valves is not in the proper position. Set the valves, then ensure the appropriate pressure is displayed on the low-pressure gauge.

If re-running the test, close the gas cylinder valve before starting the test.

1. Click **Close** to close the test report. Click **Close** again to close the test.
2. Repeat steps 1 through 8 for each gas line attached to the analyzer.

MANUAL METHOD TO CLEAN AND VERIFY GAS LINES

Master module
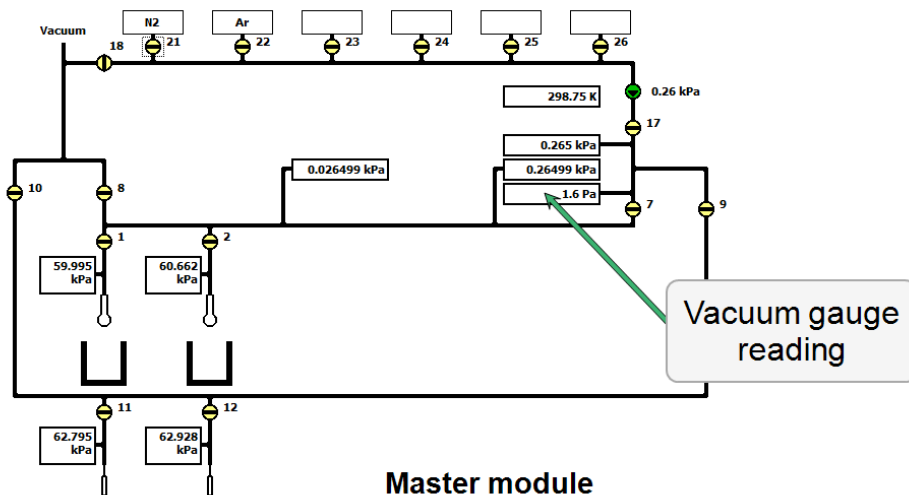
1. Open valves 7, 8, 9, 10, 17, 18, and the servo valve.
2. Open the port valve corresponding to the first gas line attached.
3. Wait for the vacuum gauge reading on the schematic to come down to below 10 µmHg, then wait an additional 15 minutes.
4. Close the port valve.
5. Perform steps 2 through 4 for any additional gas lines.
6. Close the open valves.

REPLACE A GAS CYLINDER

> ⚠️ These instructions apply to working with inert gases only. When working with hazardous gases, follow the safety procedures established by your lab.
>
> A power failure or loss of cryogen can result in dangerous pressures in the sample chamber. When using toxic or flammable gases, additional venting of the cabinet may be required.

Low pressure gauge

High pressure gauge

Gas cylinder shut-off valve

Gas regulator shut-off valve

Copper tubing

Brass reducer fitting

Regulator connector nut

Regulator control knob
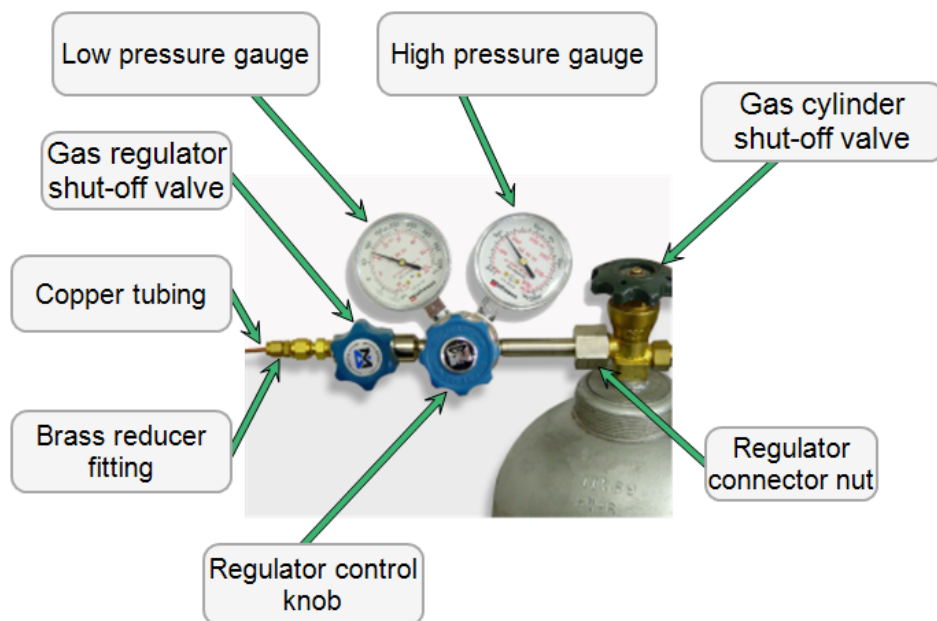
DISCONNECT THE DEPLETED GAS CYLINDER

1. Close the regulator shut-off valve and gas cylinder shut-off valve by turning the knobs clockwise.
2. Disconnect the gas line from the regulator. Gas will be vented from the line. It is not necessary to disconnect the gas line from the analyzer inlet if the cylinder will be replaced immediately with one of the same type.
3. Open the gas regulator shut-off valve by turning the knob counter-clockwise. Gas will be vented from the regulator.
4. Turn the regulator control knob clockwise to open and vent any remaining gas. Both gauges should read at or near zero. If not, make sure the gas regulator shut-off valve is open.
5. Close the regulator by turning the control knob counter-clockwise.
6. Use an appropriate wrench to loosen the nut at the regulator connector nut then remove the regulator from the cylinder.
7. Replace the protective cap on the depleted cylinder. Disconnect the retaining strap and move the cylinder to an appropriate location.

CONNECT A GAS CYLINDER

Regulator Pressure Settings

Analyzer Series	Gauge should indicate
3Flex	15 psig (103 kPag)
AccuPyc	25 psig (172 kPag)
ASAP	15 psig (103 kPag)
AutoChem	15-18 psig (103 - 124 kPag)
AutoPore	50-60psig (345 - 404 kPag)
Gemini	15-18 psig (103 - 124 kPag)
TriStar	15-18 psig (103 - 124 kPag)

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

> **CAUTION**   Over-tightening the fitting may cause a leak.

3. Check for leaks at the high-pressure side of the regulator and in the connector.
   a. Turn the regulator control knob fully counter-clockwise.
   b. Slowly open the gas cylinder shut-off valve, then quickly close it.
   c. Observe the pressure on the high-pressure gauge for approximately one minute:

   - If the pressure is stable, proceed with the next step.
   - If the pressure decreases, tighten the regulator connector nut until it becomes stable. If the pressure does not remain stable, remove the regulator and clean all contacts at the regulator connection, then reinstall the regulator.

4. Purge the air from the lines.

> Purge the regulator before proceeding to prevent contamination of the analysis gas supply.

   a. Open the gas cylinder valve to pressurize the regulator, then close the valve.
   b. Adjust the *Pressure Control* knob to approximately 5 psi.

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    c. Turn the regulator *Shut-off* valve counter-clockwise to open. Allow gas to flow until both gauges read approximately zero.

    d. Close the regulator *Shut-off* valve to stop gas flow.

    e. Reconnect the gas line to the regulator.

    f. Use two 7/16 in. (11 mm) wrenches to tighten the gas line connection. Hold one wrench fitting steady and the other to tighten the connector nut.

5. Set the analyzer pressure.

    a. Turn the *Regulator Control* knob clockwise until the low pressure gauge indicates the appropriate pressure. See *Regulator Pressure Settings on the previous page*.

    b. Open the regulator *Shut-off* valve.

    c. Open the gas cylinder *Shut-off* valve and flow gas for 10 to 30 seconds.

    d. Close the gas cylinder *Shut-off* valve.

    e. Close the gas cylinder valve.

6. If the gas line to the instrument inlet was previously disconnected, reconnect it now.

ENABLE MANUAL CONTROL

Unit [n] > Enable Manual Control

> **See also:**
>
> *Show Instrument Schematic on page 2 - 23*

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

OIL BASED VACUUM PUMP

Log in to your customer portal to access the Vacuum Pump Guide.

PREVENTIVE MAINTENANCE

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

Maintenance Required	Frequency
Clean the analyzer	As required or every 6 months
Lubricate elevator screw	As required or every 6 months. Use a light coat of lithium grease.
Check analysis port dewar	Weekly
Replace sample tube O-ring	As required or every 3 months
Replace port filters	Every 30 days
Replace vacuum pump exhaust filter*	Annually (heavy use may require more frequent maintenance)
Inspect and change vacuum pump fluid*	As required or every 3 months
Replace alumina in oil vapor traps* (if installed)	As required or every 3 months
Replace diaphragm(s) in vacuum pump (oil free pump only)**	Every 12 months
Calibrate manifold temperature sensor	Every 12 months
Calibrate system volume	Every 12 months
Check analyzer outgassing rate	Every 6 months
Test analyzer for leaks	As required or every 12 months
Perform reference material analysis	As required or every 3 months

* Oil-based vacuum pumps only.

** After about 12 to 18 months of operation, the diaphragm(s) in the pump will wear out and become completely inoperable. To prevent any instrument downtime due to an inoperable pump, it is recommended that you have the diaphragm(s) replaced by a Micromeritics Service Representative every 12 months.

CHECK ANALYSIS MANIFOLD OUTGAS RATE

1. Close the supply valves (Nupro valves) on the gas inlet lines.

2. Insert leak tight plugs in unused gas inlet ports.
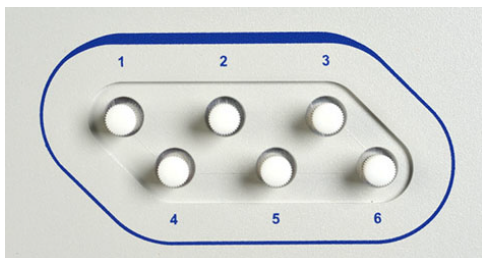
3. Go to **Unit [n] > Enable Manual Control**. Ensure a checkmark displays to the left of the menu item. If the analyzer schematic does not display, go to **Unit [n] > Show Instrument Schematic.**
4. Open valves 18, 8, 17, and 7.
5. Set the servo valve to dose to 1000 mmHg to ensure that it stays open.
6. Evacuate the inlet ports by opening valves 21 to 26.
7. Evacuate a minimum of 20 minutes. Overnight evacuation is preferable.
8. Close valves 8 and 18.
9. Record the pressure as the *Initial Reading* for *Inlet Ports* in the following table:

Test	Initial Reading	3 Min. Reading	Difference	Limits	OK?
Inlet Ports				30 µ (0.3 mmHg)	
Valve 21				21 µ (0.0021 mmHg)	
Valve 22				21 µ (0.0021 mmHg)	
Valve 23				21 µ (0.0021 mmHg)	
Valve 24				21 µ (0.0021 mmHg)	
Valve 25				21 µ (0.0021 mmHg)	
Valve 26				21 µ (0.0021 mmHg)	

10. Wait 3 minutes, then record the pressure as the *3 Minute Reading* in the table.

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11. Subtract the first reading from the second reading and record in the *Difference* column.

12. If the value in the *Difference* column is at or below the value in the *Limits* column, enter *Yes* in *OK? Column*. If the *Difference* value is not below the *Limits* value, a gas inlet valve, inlet plug, or gas line is leaking from atmosphere.

13. Close all gas inlet manifold valves (21 through 26).

14. Record the pressure as the *Initial Reading*, then begin timing as soon as the next step is completed.

15. Gas inlet valves 21 through 26 must remain closed during this procedure. Pressurize the inlet to valve 21 by opening the supply valve or removing the port plug. This allows gas or air to pressurize the inlet valve above the seat.

16. After 3 minutes, record the pressure as the *3 Minute Reading*. Subtract the first reading from the second and record in the *Difference* column.

17. Repeat steps 14 through 16 for the inlet valves 22 through 26.

CHECK AND CLEAN THE DEWAR

⚠️ When handling dewars, follow the precautions outlined in **_Dewar Precautions on page 6 - 1_**.

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

Ice and suspended frost particles may accumulate in the bottom of the analysis port dewar. Particles or deposits exceeding 1/4 in. in depth may jam between the bottom of the sample tube and the bottom of the dewar, causing the dewar not to raise fully. Accumulations of fine particles impede liquid nitrogen circulation around the bottom of the sample tube. This causes the sample temperature to be slightly higher which, in turn, can cause pore volume measurement errors in those samples exhibiting high isotherm slope above 0.97 relative pressure. Accumulated ice is likely to melt and form a pool of water in the dewar if all liquid nitrogen evaporates. The water must be removed, otherwise it will solidify when liquid nitrogen is added and could press on the bottom of the sample tube causing breakage.

To ensure problems do not develop due to ice accumulation, check the dewar after each use. Clean on a weekly basis.

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

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

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

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CLEAN THE ANALYZER

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

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

ENABLE MANUAL CONTROL

Unit [n] > Enable Manual Control

> **See also:**
>
> See *Show Instrument Schematic on page 2 - 23*.

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

Master module and two auxiliary modules
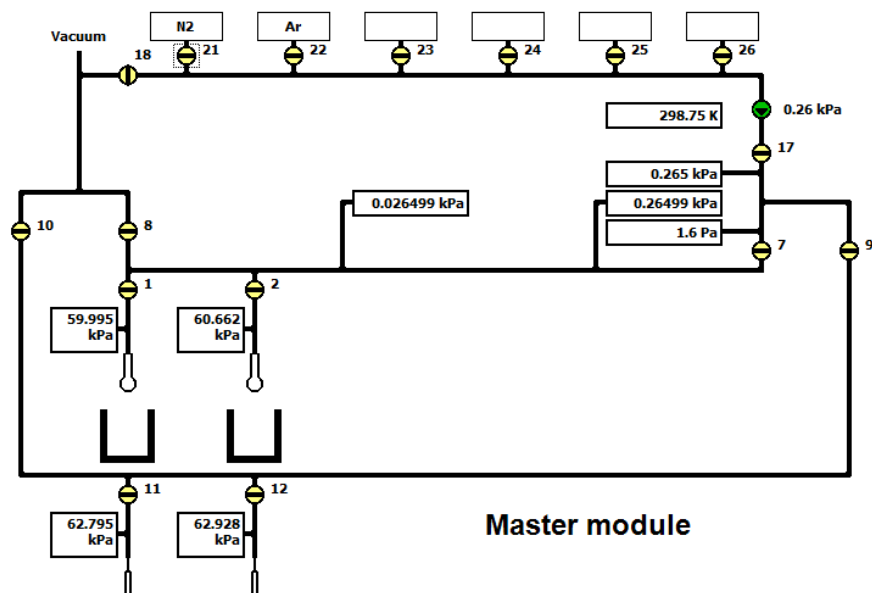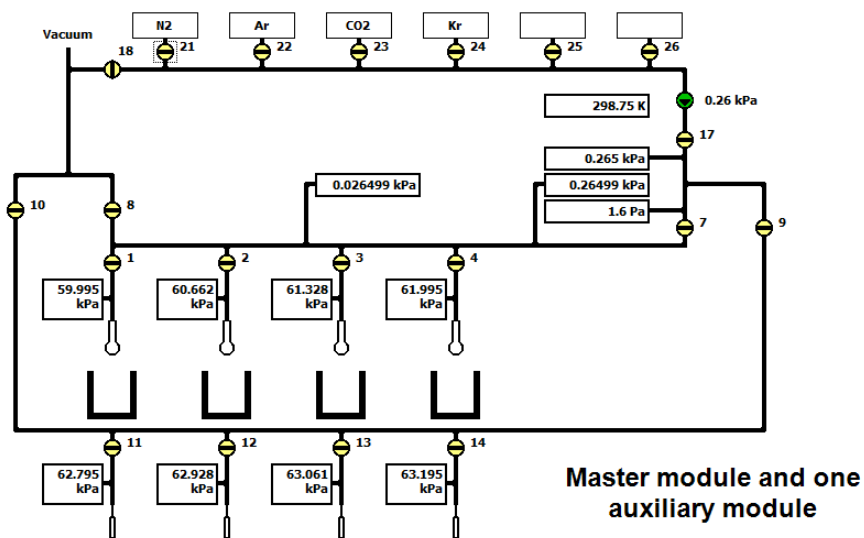
Master module and one auxiliary module

Master module

Analyzer Schematic Icon Table

Icon or Symbol	Description
	Open Valve. Green indicates an open valve.
	Closed Valve. Yellow indicates a closed valve. When manual control is disabled, closed valves appear white.

Analyzer Schematic Icon Table (continued)

Icon or Symbol	Description
●	**Servo Valve**. Closed.
◉	**Servo Valve**. Open.
⎣⎦	**Elevator.**
(dewar icons with arrows)	The arrow inside the dewar icon indicates the direction of dewar movement.
(elevator icons)	The elevator icon indicates the position of the dewar.
(sample tube icon)	**Sample Tube.** Cannot be manually controlled.
(tube icon)	P_0 **(Psat) tube.**
Transducers	Each sample port and P_0 port contains a 1000 mmHg transducer. The transducer readings display next to the ports. 298.75 K — Displays the temperature, the 1000 mmHg, 10 mmHg transducer readings, and vacuum gauge pressure. 0.265 kPa 0.26499 kPa 1.6 Pa — The 10 mmHg transducer (for krypton or Micropore installations). 0.026499 kPa — Displays the micropore transducer reading. This transducer is optional and is shown only if installed.

LUBRICATE THE ELEVATOR DRIVE ASSEMBLY

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

PORT FILTERS

Replace Port Filters

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

Replace the Analysis Port Filter

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

> Before replacing a port filter, ensure that the port valve is closed. Observe the analysis system schematic to verify valve status.

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

1. Use a wrench to remove the sample tube fitting from the analyzer.
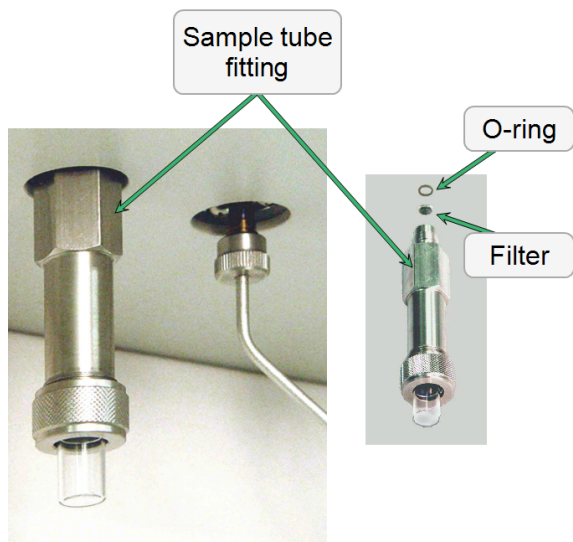
2. Remove and replace the filter and O-ring.
3. Reinstall the analysis port fitting. Securely tighten with a wrench to prevent leaks during evacuation.

Replace Degas Port Filter

> To avoid degassing problems, the new filter and O-ring must be clean. Do not touch them with bare hands.

1. Use a wrench to remove the degas port fitting, filter, and O-ring.
2. Replace the filter and the O-ring.
3. Carefully reassemble the sample tube fitting, filter, O-ring, and manifold connector. Hand tighten, then tighten with a wrench.

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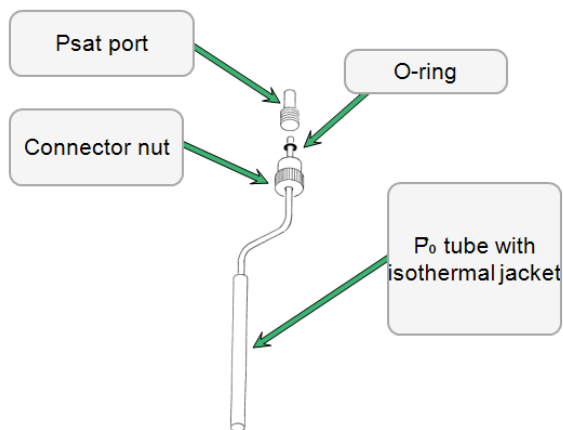
1. Turn the connector nut counter-clockwise to loosen.
2. Pull the connector nut downward.
3. Gently pull the Psat tube downward to remove it from the Psat port.
4. Remove the O-ring from the Psat tube and replace with a new one.
5. Insert the Psat tube into the Psat port.
6. Slide the connector nut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

REPLACE THE SAMPLE TUBE O-RING

It is important to maintain a vacuum-tight seal near the top of the sample tube stem. If an O-ring becomes worn or cracked, it does not provide a good seal and will need to be replaced. This procedure applies to both degas and analysis ports.
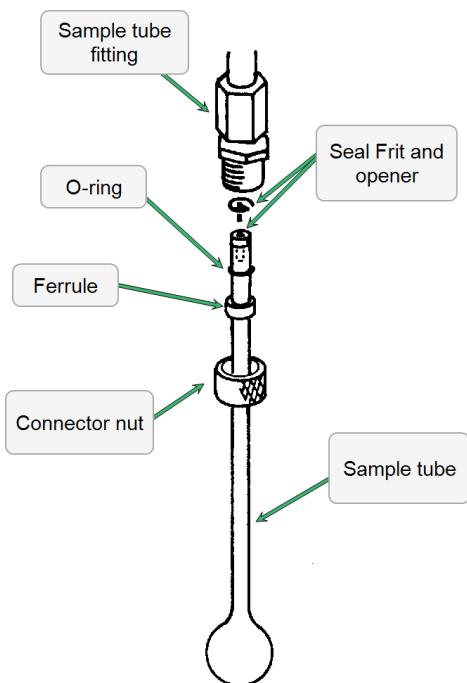
Before removing (or installing) a sample tube, ensure that the port valve is closed. Observe the analyzer schematic to verify valve status.

1. Carefully remove the dewar from the elevator. Take care not to bump the sample tube bulbs with the dewar during this process. Place the dewar aside.
2. Hold the sample tube firmly with one hand and loosen the sample tube connector nut by turning counter-clockwise.

CAUTION Do not allow the sample tube connector nut to drop onto the sample tube bulb as it may break the tube.

3. Carefully pull the sample tube down until it is free from the port. It may be necessary to grasp the sample tube with both hands.
4. Remove the O-ring from the top of the sample tube and replace it with a new one.

![Exploded view of sample tube connector showing O-ring, Ferrule, and Connector nut components]
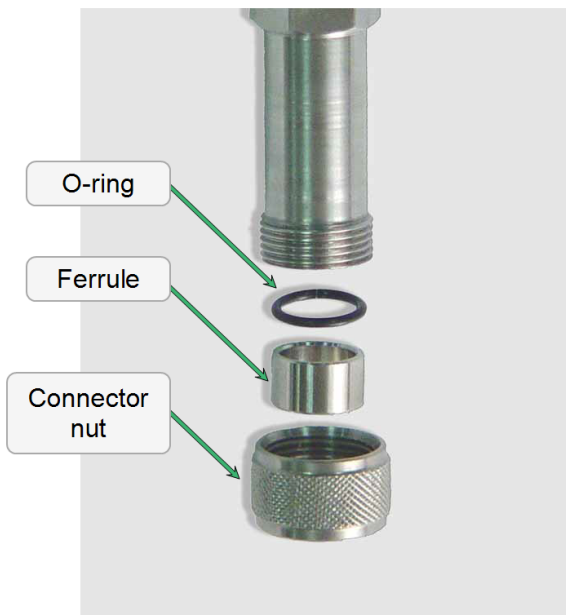
> ✏️ If the O-ring remains inside the sample port, use a pair of tweezers or needle-nose pliers to remove it.

5. While holding the connector nut in place, slide a new O-ring over the sample tube, about 1/4 in. from the top of the tube.

O-ring
(1/4 in. from top)

Ferrule

Connector nut

Isothermal
jacket
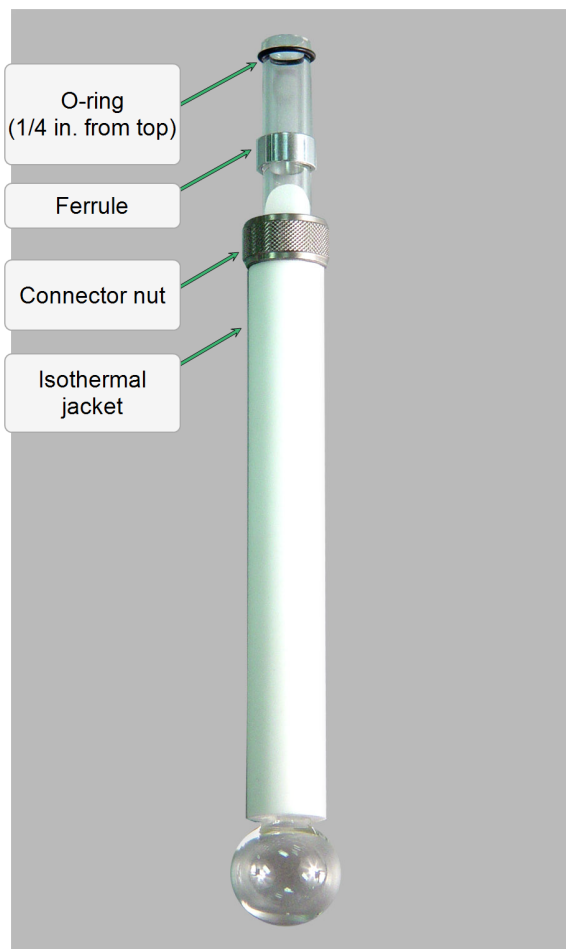
6. After the new O-ring is in place, insert the sample tube back into the sample port until it is fully seated.

7. Slide the sample tube connector nut up the tube until it comes in contact with the port fitting (the ferrule and O-ring will move along with the connector nut). Then, turning clockwise, hand tighten the connector nut to the sample connector.

POWER ANALYZER ON AND OFF

> ⚠ **CAUTION** DO NOT connect or disconnect cables when the analyzer is powered ON.

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

Power ON the equipment in the following order:

1. Computer, monitor, and printer
2. Analyzer
3. External vacuum pump (the pump must warm approximately two hours before performing analyses)
4. Degasser

Power OFF the equipment in the following order:

1. Exit the analysis program. Failure to do so could result in loss of data. If an analysis is in progress when closing the application, the following message is displayed:

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

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

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

2. Computer, monitor, and printer
3. Analyzer
4. External pump
5. Smart VacPrep
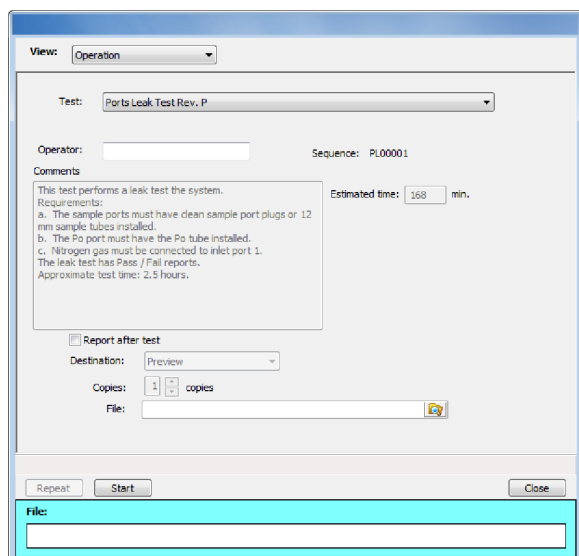
PERFORM A LEAK TEST

Unit [n] > Diagnostics > Start Diagnostic Test

A Micromeritics Service Representative may request that a leak test be performed to determine if there is a system leak and may also require a copy of the report generated by this test.

The test provides:

- Prompts on preparing the analyzer for the test
- Approximate time period of the test
- Prompts in which an operator response is required

1. Select the test to run.
2. Select *Report After Analysis* and choose *Preview* as the destination.
3. Click **Start**.
4. Verify all tests have a *Passed* status by selecting the tabs and looking for the *Passed* status for each test run.
5. Click **Save As** to save the test file results.

Suspend   Play   Skip   Report   Live Graph
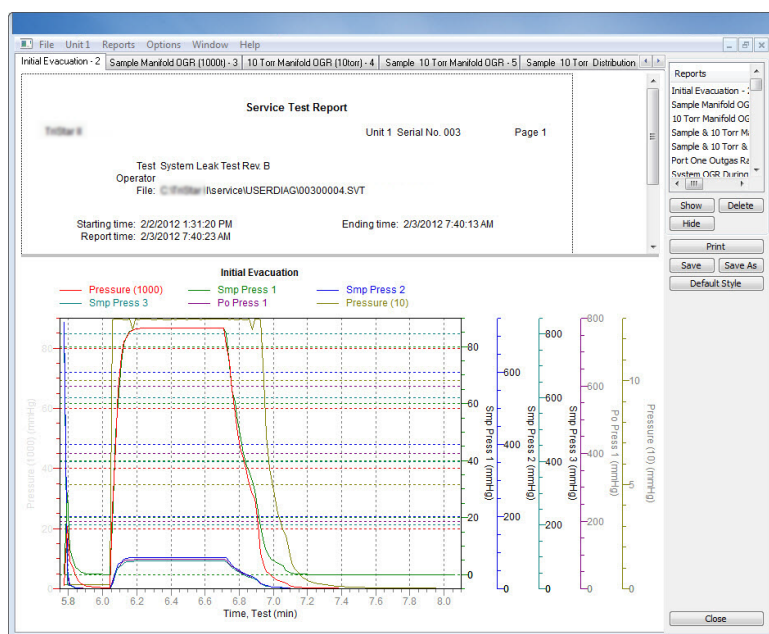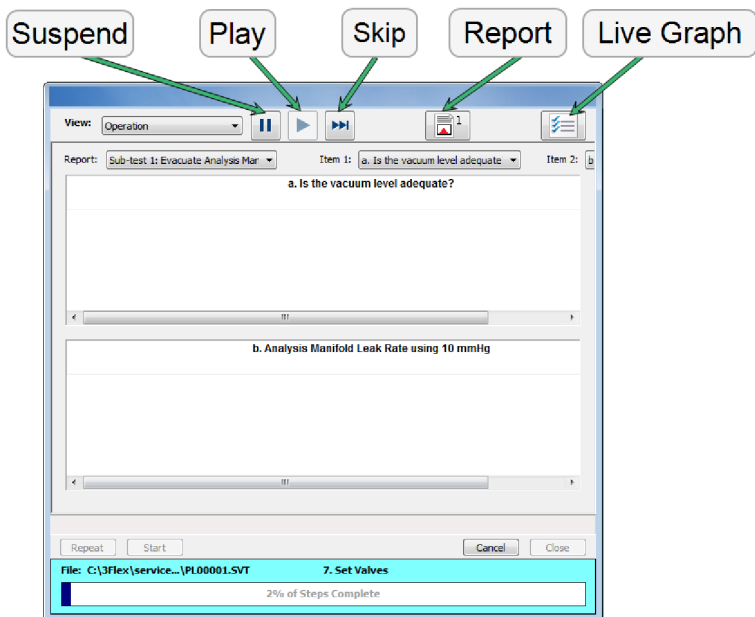
RECOVER FROM A POWER FAILURE

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

SERVICE TEST MODE

Options > Service Test Mode

Service Test Mode is a password protected option used to perform certain service tests with the assistance of a trained Micromeritics Service Representative. This password is supplied by your Micromeritics Service Representative.

If a menu item is grayed out, it is usually an indication that Service Test Mode is required.
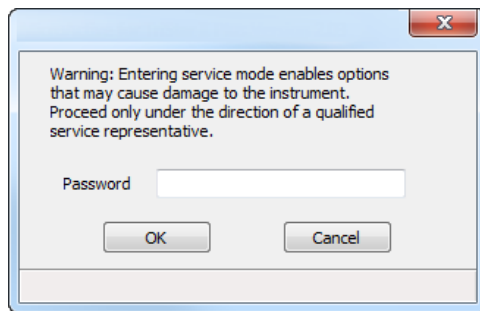
To exit Service Test Mode, go to **Options > Service Test Mode** and deselect the *Service Test Mode* option or close the application.

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A ADVANCED REPORTS - PYTHON MODULE

CFR Note In a 21CFR11 environment, the Advanced reports feature is applicable to members of the Developer group only.

> **See also:**
>
> *Mic Module Python Calls on page A - 15*

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

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

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

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

ADVANCED REPORT OPTIONS

Up to five Advanced reports, each with up to 10 summary reports, 10 tabular reports, and 10 graphical reports can be created. To use this feature, a file containing a Python script that imports a "mic" Python module must be created. See *Mic Module Python Calls on page A - 15* for an example of Python script and functions for the "mic" Python module.

1. Create the Python script and save it in the *Scripts* directory.
2. Open a sample file with a *Completed* status.
3. Select *Advanced* in the drop-down list at the bottom of the window to return to the tabbed view.
4. On the *Report Options* tab, select *Advanced* (or *User-Defined*) in the *Selected Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add** in the *Available Scripts* group box to locate and select the Python script. Repeat for each script to be added.
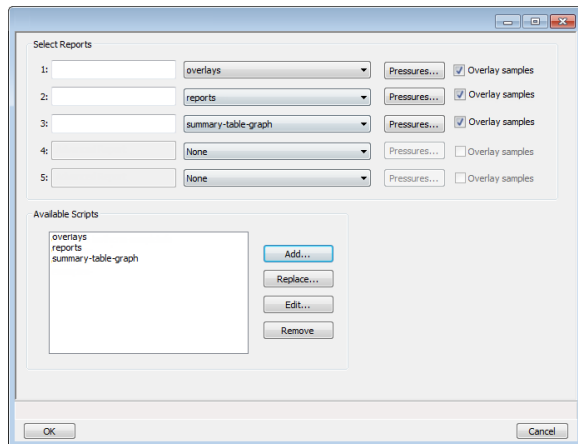
6. In the *Selected Reports* group box, click the drop-down arrows to select up to five Python scripts previously added in the *Available Scripts* box.
7. Click **Pressures** to add pressure points to the report. Click **OK** to return to the *Report Options* tab.
8. Select the *Overlay samples* checkbox to enable the overlay sample feature.
9. On the *Report Options* tab, click **Preview**. The Python Reports will be included on the tabs across the top portion of the *Reports* window.

Advanced Reports

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

> **For fields and buttons not listed in this table, see *Common Fields and Buttons on page 2 - 4*.**

SCRIPTS

RUN A SCRIPT

1. Open a sample file with a *Complete* file status.
2. Select *Advanced* in the drop-down list at the bottom of the window.
3. Select the *Report Options* tab.
4. Highlight *Advanced* in the *Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add**. Locate and select one or more python scripts then click **Select**. The selected scripts become a part of the drop-down list in the *Available Scripts* section of the *Advanced Report Options* window.
6. In the *Selected Reports* section, select up to five Advanced reports in the drop-down lists. Use the **Pressures** button to include or exclude available pressures in the report.
7. Click **OK**.
8. Click **Preview** on the *Report Options* tab to view all reports selected in the previous window.

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EDIT A SCRIPT

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

Field or Button	Description
Add *[button]*	Adds one or more scripts to the *Available Scripts* box. The added scripts then become available as options in the *Selected Reports* section.
Edit *[button]*	Edits the script stored within the application but does not affect the original .py text file.
Overlay samples *[check box]*	Select to enable the overlay sample files process.
Pressures *[button]*	Select to include or exclude pressures from the report. • **Calculation pressure range.** Enter the minimum and maximum pressures to be used in the pressure table. • **Cancel**. Discards any changes or cancels the current process. • **Exclude All.** Select to exclude all pressure points in the table. • **Include All.** Select to include all pressure points in the table. • **OK.** Saves and closes the active window.
Remove *[button]*	Removes the script from the *Available Scripts* box but does not affect original .py text file.
Replace *[button]*	Replaces the contents of the selected script however, the script name remains the same.

REMOVE A SCRIPT

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

SUMMARY REPORT

This script produces a summary report with two summaries:

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

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


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

The result is:

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


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

TABULAR REPORT

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

```
import mic
import numpy as np

mic.table("My Tables")

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

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

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


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

The result is:

My set A

x	y
1.0	0.5
2.0	1.0
3.0	1.5

My set B

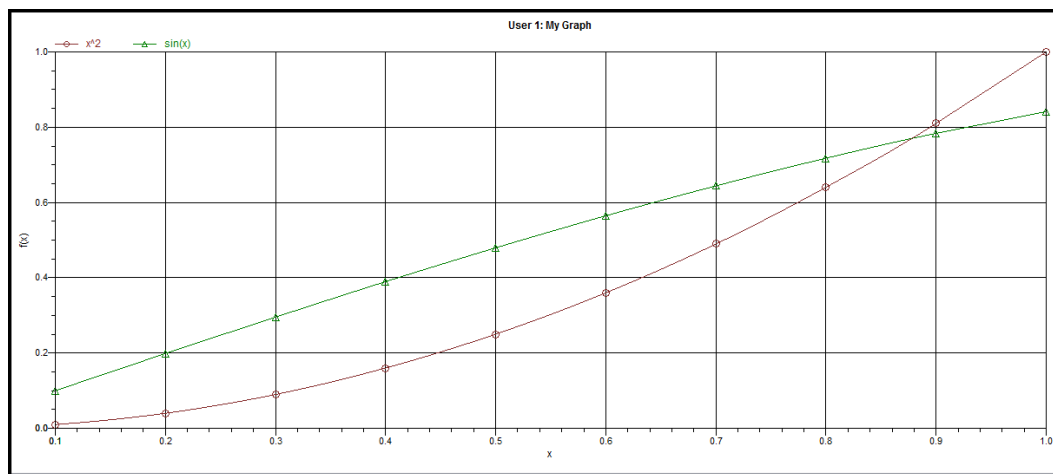| x	sin(x)	cos(x)
0.200	0.199	0.980
0.500	0.479	0.878
1.571	1.000	0.000

GRAPHIC REPORT

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

```
import mic
import numpy as np

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

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

The results are:

ACQUIRE BASIC INFORMATION

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

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

```
import mic

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

pabs, qads, n_ads, ambient_fs, analysis_fs, mass, desc = mic.isotherm('abs')

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

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

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

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

mic.summary.add( "Adsorptive Data",
                 [ "Cross Sectional Area",
                   "Hard Sphere Diameter",
                   "Density Conversion Factor",
```

```
                    "Molecular Weight",
                    "Analysis gas"],
              [ "%8.3f" % csa,
                "%8.3f" % hsd,
                "%8.3f" % dcf,
                "%8.3f" % mol_weight,
                analysis_gas  ]  )
```

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

ACQUIRE REPORT RESULTS

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

```
import mic

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

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

mic.summary.add( "Report Results",
                 [ "bet surface area",
                   "bet constant",
                   "bety 6" ,
                    "tplot external surface area",
                    "tplot micropore volume"],
                 [ "%10.5f" % sa,
                   "%10.5f" % c,
                   "%10.5f" % vm,
                   "%10.5f" % esa,
                   "%10.5f" % vol ] )
```

The result is:

Report Results

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

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

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

The result is:
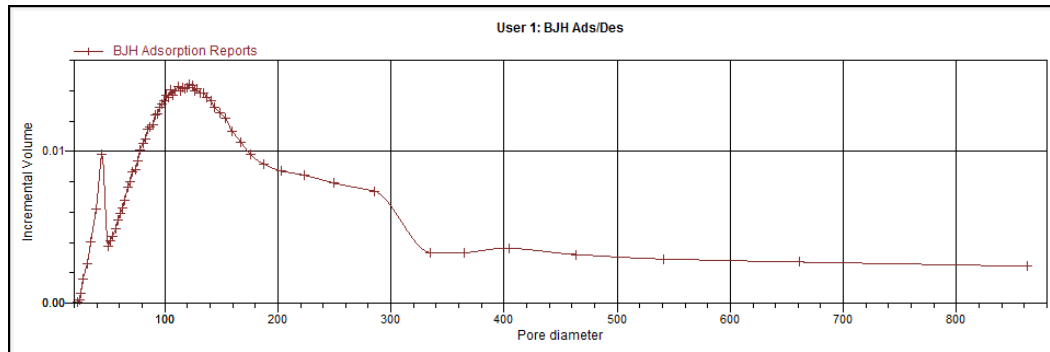
See the *Mic Module Python Calls* section for a more complete description of the usage and scope of the *mic.report* call.

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ACQUIRE OVERLAY SAMPLE DATA

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

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

```
import mic

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

mic.graph( 'Three Sample Isotherms',
           'Rel. Press',
           'Quantity Adsorbed (cm^3/g)' )

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

mic.summary( "A summary report" )

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

The results are:

User 1: Three Sample Isotherms

Legend: Primary Isotherm — Overlay Isotherm 1 — Overlay Isotherm 2

Y-axis: Quantity Adsorbed (cm^3/g)
X-axis: Rel. Press
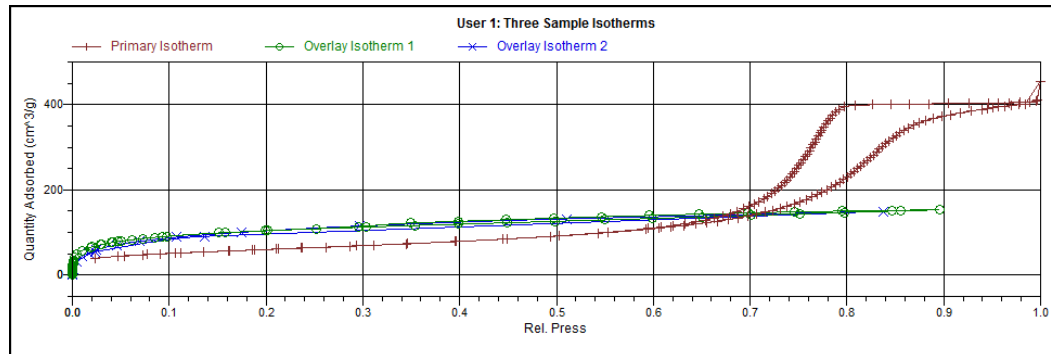
Two samples
Primary Sample: 12 mm Tube N2 Silica-Alumina ADS-DES with FS
Overlay Sample 1: Activated Carbon Hexane Dosed from Port 3 - 2
Overlay Sample 2: Activated Carbon Tube C4 Butane Port 3

To enable the use of overlay data in the Advanced reports, the following two actions must be taken prior to running the script:

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

ENABLE THE USE OF OVERLAY DATA

1. On the *Report Options* tab, click **Overlays**.
2. On the *Plot Overlay Sample Selection* window, to move a file from the *Available Files* list box to the *Selected Files* list box, either double click a file name in the *Available Files* list box or click one or more files in the *Available Files* list box then click **Add**.
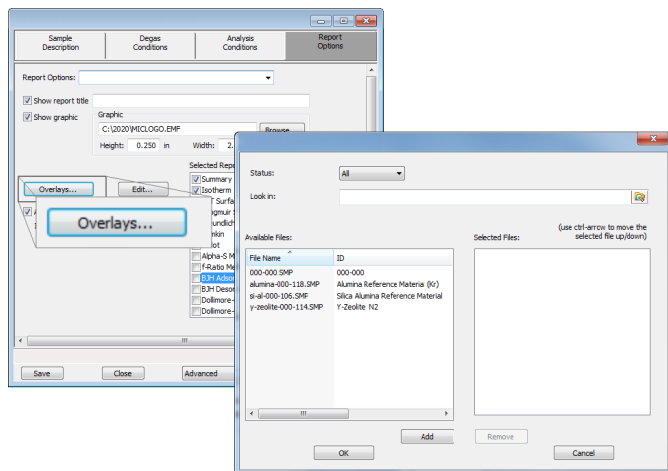
3. Click **OK**.
4. On the *Report Options* tab, highlight *Advanced* in the *Selected Reports* list box.
5. Click **Edit** to the left of the *Selected Reports* list box.
6. Select the *Overlay samples* checkbox to the right of the selected report.
7. Click **OK**.
8. Run the script using the instructions found in *Scripts on page A - 3*.
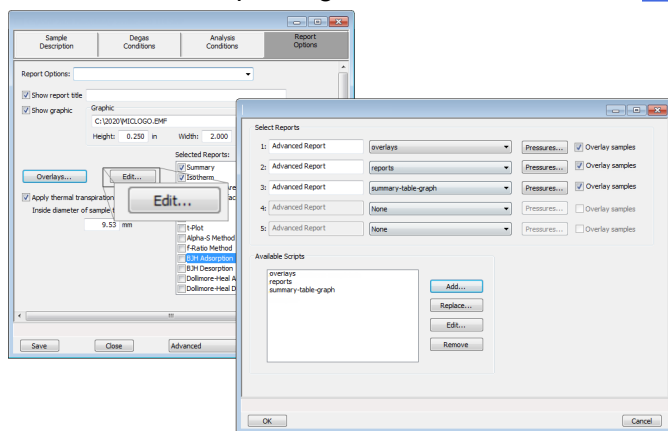
MIC MODULE PYTHON CALLS

TABLES

Available Mic Python calls for tables:

- Create a new tabular report
- Add a column
- Add a table

ADD A TABLE

This script adds a table to the last created tabular report:

```
mic.table.addtable( name )

Keyword arguments:

   name --- the table name
```

ADD A COLUMN

This script adds a column to the last created table:

```
mic.table.addcolumn( header, values )

Keyword arguments:

   header --- column header; must be a string (or convertible)
   values --- column values; must be a list of strings (or convertible)
```

CREATE A NEW TABULAR REPORT

```
mic.table( title='User Table' )

Keyword arguments:

  title --- the tabular report title (default = 'User Table')
```

SUMMARY REPORTS

Available Mic Python calls for summary reports:

- Add a summary section to the last created summary report
- Create a new summary report

ADD A SUMMARY SECTION

This script adds a summary section to the last created summary report:

```
mic.summary.add( name, labels, values )

Keyword arguments:

   name   --- summary section name
   labels --- column of labels; must be a list of strings
              (or convertible) and the same length as values
   values --- column of values; must be a list of strings
              (or convertible) and the same length as labels
```

CREATE A NEW SUMMARY REPORT

```
mic.summary( title='User Summary' )

Keyword arguments:

   title --- the summary title
```

GRAPHIC REPORTS

Available Mic Python calls for graphic reports:

- Add a curve
- Add a curve using the second y-axis
- Create a new graphic report

ADD A CURVE

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

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

Keyword arguments:

   name      --- the curve name
   x         --- list of x values; must be a list of floats
                 (or convertible) and the same length as y
   y         --- list of y values; must be a list of floats
                 (or convertible) and the same length as x
   yyaxis    --- place this curve on the yy-axis if True
                 otherwise place on the  y-axis (default = False)
   color     --- RGB color as an HTML hex string (e.g., '#4169e1')
                 or a three-element list or tuple (e.g., [65,105,225]);
                 if None, color is automatically selected (default = None)
   linestyle --- line style; (default = '-')
                     '-'        : solid
                     '--'       : dash
                     '.'        : dot
                     '-.'       : dash dot
                     '-..'      : dash dot dot
marker    --- marker shape; (default = 'a')
                 '+'        : plus
                 'o' or '0' : circle
                 'x'        : cross
                 '^'        : up triangle
                 'v'        : down triangle
                 's'        : square
                 'd'        : diamond
                 '8'        : hourglass
                 '~'        : horizontal hourglass
                 '' or None : no marker
                 'a'        : automatically selected
```

```
graphtype --- graph type; (default = 'both')
                'curve'  or 'c' : curve
                'points' or 'p' : points
                'both'   or 'b' : curve-and-points
                'hist'   or 'h' : histogram
```

ADD A CURVE USING THE SECOND Y-AXIS

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

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

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

CREATE A NEW GRAPHICAL REPORT

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

Keyword arguments:

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

GET PRIMARY ISOTHERM DATA

```
mic.overlay( overlay_number = 1, press_type='rel' )

Keyword arguments:

  overlay_number --- the overlay number (1 through 8; default = 1)
  press_type     --- the pressure basis; use 'rel' for relative pressure,
                     'abs' for absolute (default = 'rel')

Usage:
```

```
 p, qads, num_ads, ambient_fs, analysis_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
 ambient_fs  --- ambient free-space; 0.0 if overlay is unavailable
 analysis_fs --- analysis free-space; 0.0 if overlay is unavailable
 mass        --- sample mass; 0.0 if overlay is unavailable
 desc        --- sample description; empty-string if
                 overlay is unavailable
```

GET OVERLAY ISOTHERM DATA

```
mic.overlay( overlay_number = 1, press_type='rel' )

Keyword arguments:

 overlay_number --- the overlay number (1 through 8; default = 1)
 press_type     --- the pressure basis; use 'rel' for relative pressure,
                    'abs' for absolute (default = 'rel')

Usage:

 p, qads, num_ads, ambient_fs, analysis_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
 ambient_fs  --- ambient free-space; 0.0 if overlay is unavailable
 analysis_fs --- analysis free-space; 0.0 if overlay is unavailable
 mass        --- sample mass; 0.0 if overlay is unavailable
 desc        --- sample description; empty-string if
                 overlay is unavailable
```

GET ADSORPTIVE DATA FOR EACH SAMPLE

```
mic.adsorptive_data( sample_number = 0 )

Keyword arguments:

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

Usage:

csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data()
csa, hsd, dcf, mol_weight, analysis_gas = mic.adsorptive_data(0)

  csa          --- cross sectional area (nm^2)
  hsd          --- hard sphere diameter (angstroms)
  dcf          --- density conversion factor (dimensionless)
  mol_weight   --- molecular weight
  analysis_gas --- mnemonic for the analysis gas species
                   (e.g., 'CO', 'H2')
```

GET SAMPLE INFORMATION ITEM

```
mic.sample_information( item, sample_number = 0 )

Keyword arguments:

  item          --- string identifying the item to be returned.
                    Accepted identifiers are

                    'sample mass'
                    'sample description'
                    'analysis temperature'  (degrees Kelvin)
                    'sample density'        ( g/cm^3 )

  sample_number --- Sample to retrieve (default = 0).
                    0           : the current sample file
                    1 through 8 : the corresponding overlay sample file

Usage:

  mass = sample_information('sample mass')
  mass = sample_information('sample mass',0)
```

GET REPORT RESULTS

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

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



Keyword arguments:

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

Usage:

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

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

Report keyword  Result keyword               Description
--------------  --------------               -----------
   bet          surface area                 Surface area ( m^2/g )
   bet          bet constant                 BET constant ( dimensionless )
   bet          monolayer capacity           Monolayer capacity ( cm^3/g )
   tplot        external surface area        External surface area (m^2/g)
   tplot        micropore volume             Micropore volume (cm^3/g)
   bjhads       incremental distribution     Incremental Distribution
   bjhdes       incremental distribution     Incremental Distribution
   dhads        incremental distribution     Incremental Distribution
   hk           incremental distribution     Incremental Distribution
   dft          incremental distribution     Incremental Distribution
   nldft        incremental distribution     Incremental Distribution

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

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

GET IMPORTED PORE DATA

```
mic.imported_pore_data( import_number = 1 )

Keyword arguments:

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

Usage:

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


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

B DFT MODELS

Theories are developed by scientists in an attempt to explain a class of observed behavior. In the experimental physical sciences, theories are often expressed in terms of a model that can be visualized and described mathematically. Early models of physical adsorption were quite simple, both conceptually and mathematically, for very practical reasons — hand computations were required. Today we can explore complex models that describe adsorption systems on the atomic scale of size and sub-picosecond time frame. This is not because scientists are smarter, but because of available tools. The DFT models are created by classical approaches to adsorption as well as models based on modern statistical thermodynamics.

MODELS BASED ON STATISTICAL THERMODYNAMICS

Included in this group are methods that model the adsorption system in terms of forces acting between individual molecules.

THEORETICAL BACKGROUND

Traditional adsorption theories attempt to describe experimental adsorption isotherms with an isotherm equation containing a small number of parameters. At a minimum, these parameters include the extent of the surface, such as the monolayer capacity (V_m), and the molar intensity of the gas-surface interaction, such as the Langmuir "K" constant or the BET "C" constant. In some equations, additional parameters take into account the lateral interaction of adsorbed molecules with each other. Other theories, such as the Dubinin-Astakhov approach, also include parameters for the effect of adsorbent porosity.

Instead of this classical kinetic or phenomenological approach, we can use a molecular-based statistical thermodynamic theory that allows us to relate the adsorption isotherm to the microscopic properties of the system: the fluid-fluid and fluid-solid interaction energy parameters, the pore size, the pore geometry, and the temperature.

The following example is given so that you may understand how such a theory is constructed:

> A clean sample of a solid material containing slit-shaped pores of a single width is placed in an evacuated space. It is kept at a fixed temperature as a known quantity of pure argon gas is admitted into the space surrounding the sample. The pressure within the space is recorded over time. In this situation, the pressure falls rapidly from its initial value and gradually approaches a steady reading, called the equilibrium pressure. The amount adsorbed corresponds to the quantity of gas effectively removed from the gas phase by the solid surface. A graph that plots amount adsorbed versus equilibrium pressure is called an adsorption isotherm.

Under such conditions, the argon atoms that randomly enter the pore space feel the presence of the solid surface as the action of an external attractive force (the dispersion forces or Van der Waal's forces) and spend more time near the surface. As a result, the space near the surface acquires a greater average density of argon atoms than regions farther removed.

If the equilibrium distribution of the gas atoms near the surface could be described as a function of pressure and the molecular properties of the components of the system, then a model could be constructed for the adsorption isotherm for the system. Modern physical chemistry provides several ways to calculate this distribution. All these methods are based on the fundamental thermodynamic law that such a system adopts a configuration of minimum free energy at equilibrium. Also needed is a description of the pairwise interaction energy between atoms, U(s), commonly given by a Lennard-Jones potential:

$$U(s) = 4\epsilon\left(\frac{\sigma}{s}\right)^{12} - \left(\frac{\sigma}{s}\right)^{6}$$

where

ε = a characteristic energy of the adsorptive,
σ = the diameter of the adsorptive molecule, and
s = the separation distance.

MOLECULAR SIMULATION METHODS

Two simulation techniques are commonly used to determine the distribution of gas molecules in a system in equilibrium: the molecular dynamics method and the Monte Carlo method. Both of these are used as reference methods because their results are considered exact.

MOLECULAR DYNAMICS METHOD

In the molecular dynamics method, the position and velocity of individual gas particles are calculated over time at very short intervals. This method takes into account both the forces acting between the gas particles themselves and those acting between the gas particles and the atoms of the simulated surface. As the simulated particles collide with each other and with the surface, the average concentration of particles in the space near the surface is calculated; this calculation yields the amount of gas adsorbed.

This method can be thought of as a way to determine the chronological record of the movement of each particle in the system using time steps of 10-14 seconds. Although the mathematics are simple, the number of calculations required for a system of even a few hundred particles is astronomical and challenges even the fastest computers.

MONTE CARLO METHOD

In the Monte Carlo method, determination of the system equilibrium distribution begins with an assumption (which may be only approximate) about the initial configuration of particles in the system. The system is "equilibrated" through a process of randomly selecting one particle and conditionally moving it a random distance in a random direction.

If the move results in a configuration of *lower total energy*, then the move is completed and another particle is randomly selected to be moved.

If the move results in a configuration of *higher energy*, a probability for that event is calculated, and a random number between zero and one is generated. If the generated number is smaller than the probability of the event, then the move is accepted; otherwise, another particle is selected and the process is repeated. This process continues until the average total energy of the system no longer decreases; at this point, average configuration data are accumulated to yield the mean density distribution of particles in the system.

Monte Carlo simulations require considerably less computation time than molecular dynamic simulations and can yield the same results; however, neither method provides a really practical way to calculate complete isotherms.

DENSITY FUNCTIONAL FORMULATION

Density functional theory offers a practical alternative to both molecular dynamic and Monte Carlo simulations. When compared to reference methods based on molecular simulation, this theory provides an accurate method of describing inhomogeneous systems yet requires fewer calculations. Because the density functional theory provides accuracy and a reduced number of calculations, it is the basis embodied in the DFT models.

The system being modeled consists of a single pore represented by two parallel walls separated by a distance H. The pore is open and immersed in a single component fluid (adsorptive) at a fixed temperature and pressure. Under such conditions, the fluid responds to the walls and reaches an equilibrium distribution. In this condition (by the definition of equilibrium), the chemical potential at every point equals the chemical potential of the bulk fluid. The bulk fluid is a homogenous system of constant density; its chemical potential[1] is determined by the pressure of the system using well-known equations. The fluid near the walls is not of constant density; its chemical potential is composed of several position-dependent contributions that must total at every point to the same value as the chemical potential of the bulk fluid.

---

[1] Chemical potential may be thought of as the energy change felt by a probe particle when it is inserted into the system from a reference point outside the system. It can also be defined as the partial derivative of the grand potential energy with respect to density (or concentration).

As noted previously, at equilibrium, the whole system has a minimum (Helmholtz) free energy, known thermodynamically as the grand potential energy (GPE). Density functional theory describes the thermodynamic grand potential as a functional of the single-particle density distribution; therefore, calculating the density profile that minimizes the GPE yields the equilibrium density profile. The calculation method requires the solution of a system of complex integral equations that are implicit functions of the density vector. Since analytic solutions are not possible, the problem must be solved using iterative numerical methods. Although calculations using these methods still require supercomputing speed, the calculation of many isotherm pressure points for a wide range of pore sizes is a feasible task. The complete details of the theory and the mathematics can be found in the papers listed under *DFT Model References on page B - 17*.

The following graphs and accompanying text illustrate the results of using density functional theory to predict the behavior of a model system.

Figure 1 shows the density profile for argon at a carbon surface as calculated by density functional theory for a temperature of 87.3 K and a relative pressure of about 0.5.
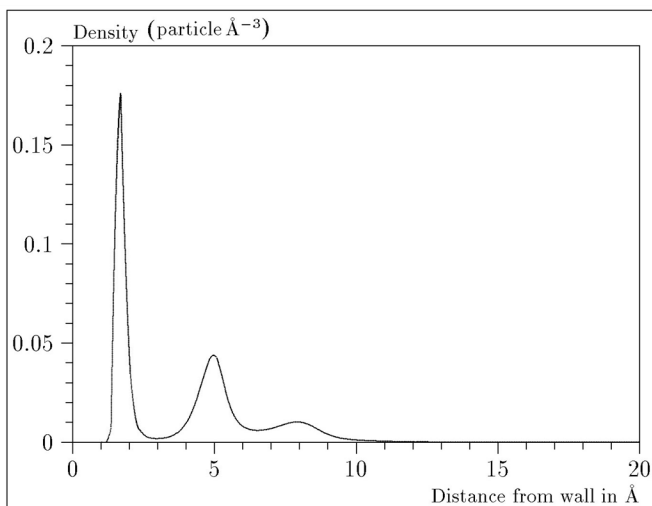

Figure 1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5

This figure represents a cross-section of the region near the surface. Note the layerwise distribution of adsorbate; the first monolayer is sharply defined and a third layer can be distinguished. The area under the profile curve represents the amount adsorbed per unit area at this pressure. The positions of the maxima are separated by a distance determined by the size of the adsorptive atom.

Given the density profile, the amount adsorbed at the stated pressure can be easily calculated as the integral over the profile. Repeating this calculation over a range of pressures yields the adsorption isotherm for the model. If the value of H is very large, the isotherm obtained corresponds to that of an external, or *free*, surface. If H is smaller, a range of pressures is reached where two minima exist for the grand potential, showing the presence of two metastable phases having different density distributions but the same chemical potential. The phase with the lower GPE is the stable one. As the pressure is increased, a point is reached where the other phase becomes the stable one. This phase transition reflects condensation of adsorbate in the pore; the pressure at which it occurs is called the *critical pore-filling pressure*. This pressure is analogous to the condensation pressure predicted by the Kelvin equation in the classical model of pore filling.

Figure 2 shows how the profiles change with pressure for a model pore with H = 40 angstroms. The inset shows the density profiles for the corresponding points of the isotherm.
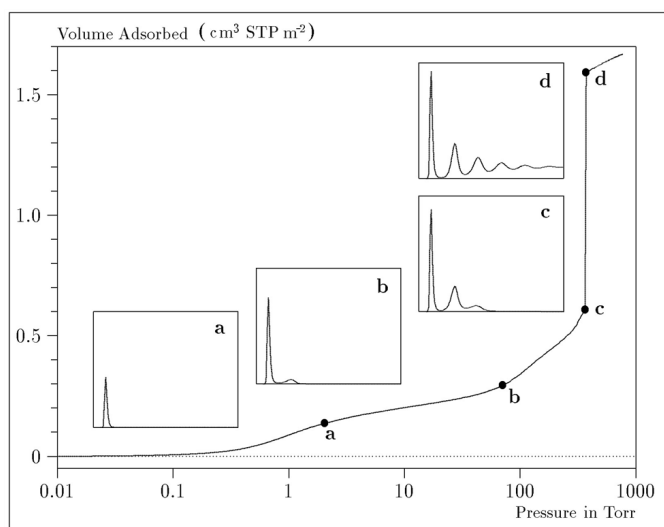
Figure 2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate

The profiles show the density distribution from one wall to the center of the slit; the other half of the distribution is a mirror image of the profile shown.

As the pressure is first increased from zero, almost all the adsorbed atoms occupy a position close to the surface.

- Inset *a* shows the profile corresponding to point a on the isotherm where the surface is about half covered.
- At point *b*, the first layer is so full that it is more favorable for atoms to start a new layer.
- At point *c*, a third layer is forming. Point *c*, for this size slit, is the critical pore-filling pressure. In inset *c*, the profile shows the density decreasing to near zero (actually the bulk gas density) at 4 or 5 molecular diameters from the surface.
- Inset *d* shows the profile converging on a density similar to that of bulk liquid argon in the center of the pore, indicating a phase transition.

![micromeritics logo]

Note that the adsorption isotherms for pores larger than the one shown in the previous graph is identical up to point **c**. The lower branch of the isotherm simply continues to a higher pressure for larger pores. This trend is illustrated in the Figure 3, where isotherms for some larger size pores are shown. It is clear that pore size is uniquely characterized by a corresponding critical pore-filling pressure. At large pore sizes, density functional theory produces results for the critical filling pressures that are in good agreement with those produced by the Kelvin equation.
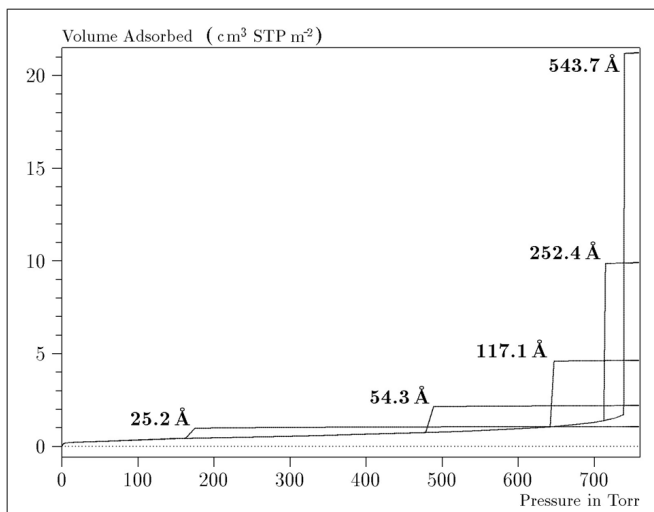
Figure 3. Model Isotherms for Some Larger Pore Widths Argon on Carbon at 87.3 K

Figure 4 shows model isotherms for pores in the micropore size range. Note the logarithmic scale for pressure.
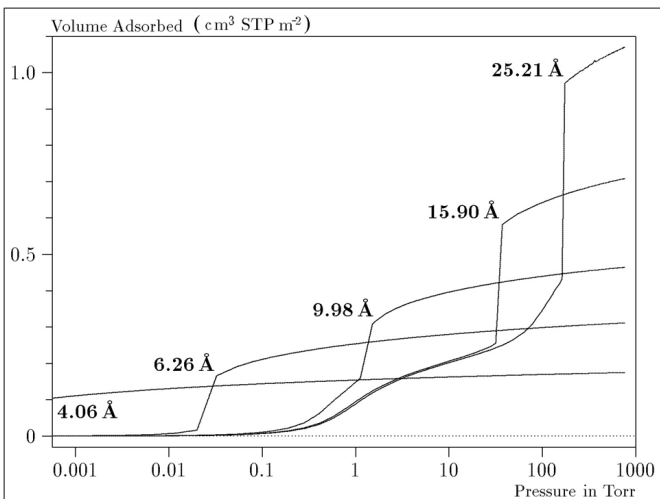
Figure 4. Model Isotherms in the Micropore Size Range of Pore Width Argon on Carbon at 87.3 K

Pores of 4 Å width, barely larger than the argon atom (3.38 Å), fill at pressures below 1 millitorr. Pores below 15 Å fill before a monolayer is completed on the surface of the larger pores. In the micropore size range, the pore volume fills more gradually with pressure and the total shape of the isotherm is important in characterizing the pore size.

Models Included

Non-Local Density Functional Theory with Density-Independent Weights

N2 - DFT Model
AR - DFT Model

Geometry:	Slit
Substrate:	Carbon (graphite)
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Using the methods of non-local density functional theory, two sets of isotherms have been calculated to serve as kernel functions for the characterization of porous solids from adsorption data. The model isotherms are stored in binary format files. These models assume a *slit-like pore geometry*. The pore size range from 4.0 to 4000 Å is covered in 91 classes in a geometric progression. The class intervals are rounded to the nearest 0.02 molecular diameters. A model for the free or external surface is included to account for unfilled pores. Each of the 92 model isotherms has been calculated at 181 pressure points from near 1×10^{-6} to near 1.00 relative pressure.

These models are identical to those supplied with the original DOS version of DFT software. Some slight difference from the DOS results may be noted when they are applied to the same data due to improvements in the deconvolution algorithm and better regularization of the current software.

Non-Local Density Functional Theory with Density-Dependent Weights

N2 - Modified Density Functional

Geometry:	Free surface
Substrate:	Surface energy
Method:	Nitrogen at 77K

Using the modified Tarazona prescription described by Olivier (see *DFT Model References on page B - 17* [items 3 and 4]), model isotherms were calculated for a wide range of adsorptive energies to a relative pressure of 0.6. The model makes no provision for pore filling in the micropore region. If the sample solid contains small mesopores, the isotherm data should be truncated (using the *Select Data Points* window) to a suitably low relative pressure to avoid trying to fit this region; mesopore filling reports as a large area of low energy in the calculated distribution of adsorptive potential.

The surface energy is reported in terms of the effective Lennard-Jones interaction parameter, i.e., for the adsorptive / adsorbent pair divided by Boltzmann constant. The units are therefore Kelvin.

N2 - Cylindrical Pores - Oxide Surface
AR - Cylindrical Pores - Oxide Surface

Geometry:	Cylinder
Substrate:	Oxide
Category:	Porosity
Method:	Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using a combination of statistical mechanical calculations and experimental observations for macroporous silicas and MCM-41 mesoporous silicas as well as zeolites. The pore-filling pressures were determined as a function of the pore size from adsorption isotherms on MCM-41 materials characterized by X-ray and other techniques. The variation of the pore fluid density with pressure and pore size has been accounted for by density functional theory calculations. The N2 model reports pore sizes ranging from 3.8 to 387 Å and the AR model from 3.8 to over 500 angstroms.

References: M. Jaroniec, M. Kruk, J.P. Olivier, and S. Koch, "A New Method for the Accurate Pore Size Analysis of MCM-41 and Other Silica-Based Mesoporous Materials," Proceedings of COPS-V, Heidelberg, Germany (1999).

N2 – Cylindrical Pores – Pillared Clay Surface (Montmorillionite)

Geometry:	Cylinder
Substrate:	Crystalline Silicate
Category:	Porosity
Method:	Nitrogen at 77K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFT) calculations and experimental isotherms for reference samples of montmorillionite. The construction method for the hybrid models was analogous to that described in the first reference below (Jaroniec et al,1999). The additional references add additional theoretical details as well as examples of the application of the model to pillared clay catalysts. This model reports pore widths from 3.8 to 387 angstroms.

References: Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V* , Unger, et al, Eds, Elsevier, Amsterdam, 2000.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of a Pillared Interlayered Clay (PILC) from a Hybrid Density Functional Theory

(DFT) Method," *The Journal of Physical Chemistry B*; 2001, 105(3), 623-629.

M. L. Occelli, J. P. Olivier, J. A. Perdigon-Melon, and A. Auroux, "Surface Area, Pore Volume Distribution, and Acidity in Mesoporous Expanded Clay Catalysts from Hybrid Density Functional Theory (DFT) and Adsorption Microcalorimetry Methods," *Langmuir* 2002, 18, 9816-9823.9b.

James P. Olivier, "The Importance of Surface Heterogeneity in Developing Characterization Methods." *6th International Symposium on the Characterization of Porous Solids*, Studies in Surface Science and Catalysis 144, Elsevier, 2002.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of Pillared Rectorite Catalysts from a Hybrid Density Functional Theory Method," *Microporous and Mesoporous Materials* 2003, 57, 291-296.

C02 - DFT Model

Geometry:	Slit
Substrate:	Carbon
Category:	Porosity
Method:	Carbon dioxide at 273 K

Model isotherms were calculated using the non-local prescription of Tarazona, employing molecular parameters derived from the known bulk properties of carbon dioxide.

AR - Modified Density Functional Model

Geometry:	Free surface
Substrate:	Any
Category:	Surface energy
Method:	Argon at 87K

This model was produced in the same manner as the N2 Modified Density Functional model listed earlier, except applicable to argon adsorbed at 87.3 K.

N2 - Tarazona NLDFT, Esf = 30.0K

Geometry:	Cylinder
Substrate:	Oxide
Category:	Porosity
Method:	Nitrogen at 77K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a cylindrical pore geometry. The wall potential used is k = 30 K, typical for a silica or alumina surface.

This model file is particularly useful for sizing zeolites or zeolite containing materials that have substantial micropore volume. The reported pore size range is 3.8 to 387 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Slit Pores by NLDFT
Ar - Carbon Slit Pores by NLDFT

Geometry:	Slit
Substrate:	Carbon
Category:	Porosity
Method:	Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and a slit-like pore geometry. These models are slightly different from N2-DFT and Ar-DFT models that were calculated using NLDFT with density independent weighting functions.

The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Finite Pores, As=6, 2D-NLDFT
Ar - Carbon Finite Pores, As=6, 2D-NLDFT

Geometry:	Finite Slit
Substrate:	Carbon
Category:	Porosity
Method:	Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions assuming 2D model of finite slit pores having a diameter-to-width aspect ratio of 6.

This model is particularly useful for microporous carbon materials. The reported pore size range is from 3.5 to 250 angstroms.

References: Jacek Jagiello and James P. Olivier. "A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.," The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N2 - Carbon Finite Pores, As=12, 2D-NLDFT
Ar - Carbon Finite Pores, As=12, 2D-NLDFT

Geometry: Finite Slit

Substrate: Carbon

Category: Porosity

Method: Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using the same methods and assumptions that were used in the model above except in this model, the aspect ratio is equal to 12.

These two finite pore models may be used as a research tool in conjunction with independent analytical techniques such as high-resolution transmission electron microscopy (HRTEM) and/or X-ray diffraction (XRD) to obtain comprehensive information about the structure of studied carbon material.

References: Jacek Jagiello and James P. Olivier. "A simple two-dimensional NLDFT model of gas adsorption in finite carbon pores. Application to pore structure analysis.," The Journal of Physical Chemistry C, 113(45):19382-19385, 2009.

N2 - Carbon Cylinder, single-wall nanotube by NLDFT
Ar - Argon Cylinder, single-wall nanotube by NLDFT

Geometry: Cylinder

Substrate: Carbon

Category: Porosity

Method: Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the graphitic surface of an infinitely long cylinder.

This model is particularly useful for characterizing carbon single-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References: P. Tarazona, Phys. Rev. A 31: 2672 (1985).
Idem, Phys. Rev. A 32: 3148 (1985).
P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987).

N2 - Carbon Cylinder, multi-wall nanotube by NLDFT
Ar - Argon Cylinder, multi-wall nanotube by NLDFT

Geometry:	Cylinder
Substrate:	Carbon
Category:	Porosity
Method:	Nitrogen at 77 K; Argon at 87 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and multiple concentric graphitic surfaces of infinitely long cylinders.

This model is particularly useful for characterizing carbon multi-wall nanotubes. The reported pore size range is from 3.5 to 1000 angstroms.

References:	P. Tarazona, Phys. Rev. A 31: 2672 (1985).
	Idem, Phys. Rev. A 32: 3148 (1985).
	P. Tarazona, U. M. B. Marconi, and R. Evans, Mol. Phys. 60: 573 (1987)

Ar - Zeolites H-Form by NLDFT

Geometry:	Cylinder
Substrate:	Zeolite
Category:	Porosity
Method:	Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is particularly useful for characterizing oxides and H+ and (NH4)+ exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

Ar - Zeolites Me-Form by NLDFT

Geometry:	Cylinder
Substrate:	Zeolite
Category:	Porosity
Method:	Argon at 77 K

Model isotherms were calculated using the prescriptions of Tarazona for density dependent weighting functions and cylindrical pore geometry. The pore wall potential is described by the Lennard-Jones potential of interaction between a gas molecule and the oxide surface of an infinitely long cylinder.

This model is similar to the model above, but it more appropriate is for characterizing alkali metal exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

MODELS BASED ON CLASSICAL THEORIES

Both surface energy distribution and pore size distribution may be evaluated using classical approaches to model kernel functions for use with equation (1) of the DFT Theory. (Log in to your customer portal to access the Calculations document.) Be aware that the deconvolution method only provides a fitting mechanism; it does not overcome any inherent shortcomings in the underlying theory.

SURFACE ENERGY

The use of classical theories to extract adsorptive potential distribution is mostly of historical interest. At a minimum, the equation must contain a parameter dependent on adsorption energy and another dependent on monolayer capacity or surface area. This is sufficient to permit the calculation of the set of model isotherms that is used to create a library model. The Langmuir equation has been used in the past, as have the Hill-de Boer equation and the Fowler-Guggenheim equation. All of these suffer from the fact that they only describe monolayer adsorption, whereas the data may include contributions from multilayer formation.

PORE SIZE

It is well established that the pore space of a mesoporous solid fills with condensed adsorbate at pressures somewhat below the prevailing saturated vapor pressure of the adsorptive. When combined with a correlating function that relates pore size with a critical condensation pressure, this knowledge can be used to characterize the mesopore size distribution of the adsorbent. The correlating function most commonly used is the Kelvin equation. Refinements make allowance for the reduction of the physical pore size by the thickness of the adsorbed film existing at the critical condensation pressure. Still further refinements adjust the film thickness for the curvature of the pore wall.

The commonly used practical methods of extracting mesopore distribution from isotherm data using Kelvin-based theories, such as the BJH method, were for the most part developed decades ago and were designed for hand computation using relatively few experimental points. In general, these methods visualize the incremental decomposition of an experimental isotherm, starting at the highest relative pressure or pore size. At each step, the quantity of adsorptive involved is divided between pore emptying and film thinning processes and exactly is accounted for. This computational algorithm frequently leads to inconsistencies when carried to small mesopore sizes. If the thickness curve used is too steep, it finally will predict a larger increment of adsorptive for a given pressure increment than is actually observed; since a negative pore volume is non-physical, the algorithm must stop. Conversely, if the thickness curve used underestimates film thinning, accumulated error results in the calculation of an overly large volume of (possibly nonexistent) small pores.

The use of equation (1) represents an improvement over the traditional algorithm. Kernel functions corresponding to various classical Kelvin-based methods have been calculated for differing geometries and included in the list of models.

MODELS INCLUDED

Kelvin Equation with Halsey Thickness Curve

N2 - Halsey Thickness Curve

Geometry:	Slit
Substrate:	Average
Category:	Porosity
Method:	Nitrogen 77 K

The kernel function is calculated using the Halsey equation with standard parameters:

$$t = 3.54\left(\frac{-5.00}{ln(P/P_0)}\right)^{1/3}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension =	8.88 dynes cm^{-1}
Molar density =	0.02887 g cm^{-3}

N2 - Halsey Thickness Curve

Geometry:	Cylinder
Substrate:	Average
Category:	Porosity
Method:	Nitrogen 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

Reference: G. Halsey, J. Chem. Phys 16, 931 (1948).

Kelvin Equation with Harkins and Jura Thickness Curve

N2 - Harkins and Jura Thickness Curve

Geometry: Slit
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

The kernel function is calculated using the Harkins and Jura equation with standard parameters:

$$t = 3.54\left(\frac{13.99}{0.034-log(P/P_0)}\right)^{1/2}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}
Molar density = 0.02887 g cm^{-3}

N2 - Harkins and Jura Thickness Curve

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

References: W. D. Harkins and G. Jura, J.A.C.S. 66, 1366 (1944).
J. H. DeBoer et al., J. Colloid and Interface Sci. 21, 405 (1966).

Kelvin Equation with Broekhoff-de Boer Thickness Curve

N2 - Broekhoff-de Boer Model

Geometry: Cylinder
Substrate: Average
Category: Porosity
Method: Nitrogen 77 K

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The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

$$\log\left(p/p^0\right) = \frac{-16.11}{t^2} + 0.1682^{-0.1137t}$$

The nitrogen properties used in the Kelvin equation are:

Surface tension = 8.88 dynes cm^{-1}

Molar density = 0.02887g cm^{-3}

N2 - Broekhoff-de Boer Model

Geometry: Cylinder

Substrate: Average

Category: Porosity

Method: Nitrogen 77 K

The calculation is similar to the above except that cylindrical geometry is assumed, and the film thickness depends on pore size (see reference).

References: Specifically, equations 20 and 21 in: J.C.P. Broekhoff and J.H. de Boer, "The Surface Area in Intermediate Pores," Proceedings of the International Symposium on Surface Area Determination, D.H. Everett, R.H. Ottwill, eds., U.K. (1969).

DFT Model References

The papers listed below provide additional information on DFT models:

1. "Determination of Pore Size Distribution from Density Functional Theoretic Models of Adsorption and Condensation within Porous Solids," J.P. Olivier and W.B. Conklin, Micromeritics Instrument Corp; presented at the International Symposium on the Effects of Surface Heterogeneity in Adsorption and Catalysts on Solids, Kazimierz Dolny, Poland (July 1992).
2. "Classification of Adsorption Behavior: Simple Fluids in Pores of Slit-shaped Geometry," Perla B. Balbuena and Keith E. Gubbins, *Fluid Phase Equilibria*, 76, 21-35, Elsevier Science Publishers, B.V., Amsterdam (1992).
3. "Modeling Physical Adsorption on Porous and Nonporous solids Using Density Functional Theory," J.P. Olivier, *Journal of Porous Materials*, 3, 9-17 (1995).
4. "The Determination of Surface Energetic Heterogeneity Using Model Isotherms Calculated by Density Functional Theory," J.P. Olivier; presented at the Fifth International Conference on the Fundamentals of Adsorption, Pacific Grove, CA (1995).

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C EXPORTED DATA EXAMPLE

```
Sample Information

                    Sample Information Parameters

              Method:    Default
              Sample:    Activated Carbon c1003 Carbon Dioxide Analysis
            Operator:    wendy
           Submitter:    s/n 103
           Mass type:    Entered
                Mass:    0.1281 g
             Density:    1.000 g/cm³
        Type of data:    Automatically collected
     Instrument type:    2460
Original instrument type: 2460
            Comments:


Sample Tube

              Sample Tube Options

          Sample Tube:    w1
      Warm free space:    1.0000 cm³
      Cold free space:    1.0000 cm³
   Non-ideality factor:   0.0000620
 Use isothermal jacket:   No
        Use filler rod:   No
     Vacuum seal type:    None


Degas Conditions

        Degas Conditions Options

   Description:    Degas Conditions

              Evacuation Phase

   Temperature Ramp Rate:    10.0 K/min
       Target Temperature:   303 K
          Evacuation Rate:   0.67 kPa/s
  Unrest. Evacuation from:   0.67 kPa
         Vacuum Setpoint:    1.333224e-003 kPa
          Evacuation Time:   10 min

              Heating Phase

   Sample                   Ramp
   Prep:      Temperature    Rate        Time
   Stage      (K)  (K/min)   (min)
   ----
       1          303    10.0       10

Evacuation and Heating Phases

   Hold Pressure:    13.3 kPa
```

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Backfill Sample Tube: Automatic

Analysis Conditions

Analysis Conditions Options

Description: Charcoal, CO2, @ 273.15 K
Isotherm Collection: Target Pressure
Absolute Pressure Dosing: No

Pressure Table

Relative Pressure (p/p°)	Rel Pressure Increment (p/p°)
0.000010000	
0.000518305	
0.001026610	
0.001534915	
0.002043220	
0.002551525	
0.003059831	
0.003568136	
0.004076441	
0.004584746	
0.005093051	
0.005601356	
0.006109661	
0.006617966	
0.007126271	
0.007634576	
0.008142881	
0.008651186	
0.009159492	
0.009667797	
0.010176102	
0.010684407	
0.011192712	
0.011701017	
0.012209322	
0.012717627	
0.013225932	
0.013734237	
0.014242542	
0.014750847	
0.015259153	
0.015767458	
0.016275763	
0.016784068	
0.017292373	
0.017800678	
0.018308983	
0.018817288	
0.019325593	
0.019833898	
0.020342203	
0.020850508	

```
0.020850508
0.021358814
0.021867119
0.022375424
0.022883729
0.023392034
0.023900339
0.024408644
0.024916949
0.025425254
0.025933559
0.026441864
0.026950169
0.027458475
0.027966780
0.028475085
0.028983390
0.029491695
0.030000000
```

Preparation

Fast evacuation:	No
Evacuation rate:	0.67 kPa/s
Unrestricted evacuation from:	4.00 kPa
Vacuum setpoint:	1.3 Pa
Evacuation time:	0.50 h

Leak test:	No
Use TranSeal:	No

Free Space

Entered

| Warm free space: | 16.0000 cm³ |
| Cold free space: | 45.0000 cm³ |

p° and Temperature

p° type:	Calculated from analysis temperature
Temperature type:	Entered
Temperature:	273.150 K

Dosing

Use first pressure fixed dose:	No
Use maximum volume increment:	No
Target tolerance:	5.0% or 0.6666 kPa
Minimum equilibration delay at p° >= 0.995:	600 s
Low pressure dosing:	No

Equilibration

| Equilibration time (p/p° = 1.000000000): | 20 s |
| Minimum equilibration delay at p/p° >= 0.995: | 600 s |

```
            Sample Backfill

   Backfill at start of analysis:     Yes
     Backfill at end of analysis:     Yes
                    Backfill gas:     N2
```

Adsorptive Properties

```
              Adsorptive Properties Options

                     Adsorptive:    Carbon Dioxide @ 273.15 K  (CO2)
         Non-condensing adsorptive:  No
         Maximum manifold pressure:  123.323 kPa
   Therm. tran. hard-sphere diameter: 3.6040 Å
     Molecular cross-sectional area:  0.170 nm²
              Non-ideality factor:   0.0000000
          Density conversion factor: 0.0018306
         Adsorbate molecular weight: 2.00
                  Dosing method:     Normal
```

Psat vs. Temperature Table

	Saturation Pressure (kPa)	Temperature (K)
1	17188.920	258.150
2	19871.720	263.150
3	22849.400	268.150
4	26141.720	273.150
5	29770.720	278.150
6	33763.000	283.150
7	38145.920	288.150
8	42959.000	293.150
9	48250.120	298.150
10	54086.160	303.150

Report Options

```
                 Report Options

                  Description:    Report Options
           Show report title:    Yes
                Report title:
                Show graphic:    Yes
                Graphic file:    miclogo.emf
               Graphic width:    2.000 inches
              Graphic height:    0.250 inches
  Apply thermal transpiration correction:    No
```

        Summary: No

        Isotherm: Yes

```
                Isotherm Reports

                   Elapsed time:     Yes
            Time between points:     No
          Plot adsorption branch:    Yes
          Plot desorption branch:    Yes
               Quantity adsorbed:    Per Gram

          Tabular report selected:   Yes
             Linear plot selected:   Yes
        Logarithmic plot selected:   Yes
      Linear absolute plot selected: Yes
  Pressure composition plot selected: Yes

Isotherm Linear Plot axis data

          Plot curve:     Yes
          Plot points:    Yes
      Overlay samples:    No
     Autoscale X axis:    Yes
     Autoscale Y axis:    Yes

Isotherm Log Plot axis data

          Plot curve:     Yes
          Plot points:    Yes
      Overlay samples:    No
     Autoscale X axis:    Yes
     Autoscale Y axis:    Yes

Isotherm Linear Absolute Plot axis data

          Plot curve:     Yes
          Plot points:    Yes
      Overlay samples:    No
     Autoscale X axis:    Yes
     Autoscale Y axis:    Yes

Isotherm Log Absolute Plot axis data

          Plot curve:     Yes
          Plot points:    Yes
      Overlay samples:    No
     Autoscale X axis:    Yes
     Autoscale Y axis:    Yes

Isotherm Pressure Composition axis data

          Plot curve:     Yes
          Plot points:    Yes
      Overlay samples:    No
     Autoscale X axis:    Yes
     Autoscale Y axis:    Yes


        BET: No


        Langmuir: No
```

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```
Freundlich: No

Temkin: No

t-Plot: No

Alpha-S Method: No

f-Ratio Method: No

BJH Adsorption: No

BJH Desorption: No

Dollimore-Heal Adsorption: No

Dollimore-Heal Desorption: No

Horvath-Kawazoe: No

NLDFT Advanced PSD: Yes

                    NLDFT Advanced PSD Reports

         Type:    DFT
     Geometry:    Slit
        Model:    CO2@273-Carbon, NLDFT
  Dual Sample:    ...\Activated Carbon c1003 Nitrogen Tube w1 Port 1.SMP
   Dual Model:    N2 @ 77 on Carbon Slit Pores by NLDFT
Regularization:   Non-negative Regularization:  0.01000

              Reports

        Tabular Report:      Yes
        Isotherm Table:      Yes
   Cumulative Area Graph:    Yes
  Incremental Area Graph:    Yes
        dA/dw Area Graph:    Yes
   dA/dlog(w) Area Graph:    Yes
 Cumulative Volume Graph:    Yes
Incremental Volume Graph:    Yes
        dv/dw Volume Graph:  Yes
   dv/dlog(w) Volume Graph:  Yes
 Log Goodness of Fit Graph:  Yes
    Goodness of Fit Graph:   Yes
```

DFT Pore Size: No

DFT Surface Energy: No

Dubinin: No

MP-Method: No

User-Defined: No

Options: No

Sample Log: No

Validation: No

Manufacturing: No

Collected Data

```
          Collected Data Options

           Start time:    7/12/2013 9:32:00 AM
             End time:    7/12/2013 4:22:47 PM
    Type of instrument:   I
          Unit number:    2
        Serial number:    103
Analysis Conditions modified:   No

              p° and Temperature

     p° type:    Calculated from analysis temperature
          p°:    3,485.3701 kPa
Temperature type:   Entered
    Temperature:    273.150 K

        Free Space

              Entered
Warm free space:    27.5646 cm³
Cold free space:    29.3717 cm³
```

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```
                 Isotherm Data Table

    Absolute      Relative     Quantity    Quantity    Run
    Pressure      Pressure      Dosed      Adsorbed    Time
     (kPa)         (p/p°)       (mmol)     (mmol/g)    (min)
  -----
      0.0413701   0.000011870   0.00110     0.00437     186
      1.9603866   0.000562462   0.06236     0.28892     194
      3.8850574   0.001114676   0.11400     0.49768     200
      5.2017264   0.001492446   0.14684     0.62114     240
      7.0840621   0.002032514   0.19129     0.77807     244
      8.8650414   0.002543501   0.23161     0.91301     248
     10.6392632   0.003052549   0.27035     1.03635     253
     12.4107274   0.003560806   0.30802     1.15159     257
     14.1810860   0.004068746   0.34479     1.25987     261
     15.9413807   0.004573799   0.38063     1.36196     265
     17.7055345   0.005079958   0.41590     1.45919     269
     21.4229054   0.006146522   0.48840     1.64981     274
     24.9877600   0.007169328   0.55630     1.81996     278
     28.5408927   0.008188770   0.62268     1.97941     282
     32.1173308   0.009214898   0.68828     2.13046     287
     35.6914253   0.010240354   0.75281     2.27339     291
     39.2576571   0.011263555   0.81627     2.40875     295
     42.8103727   0.012282877   0.87870     2.53743     299
     46.3579903   0.013300737   0.94038     2.66074     303
     49.9232578   0.014323660   1.00174     2.77978     308
     53.4732190   0.015342193   1.06228     2.89401     312
     57.0251087   0.016361278   1.12246     3.00520     316
     60.5796187   0.017381115   1.18218     3.11259     320
     64.1341327   0.018400953   1.24145     3.21635     325
     67.6873976   0.019420433   1.30034     3.31738     329
     71.2350193   0.020438294   1.35873     3.41505     333
     74.7973899   0.021460387   1.41709     3.51095     338
     78.3370085   0.022475951   1.47475     3.60373     342
     81.8930319   0.023496223   1.53225     3.69359     347
     85.4406495   0.024514082   1.58942     3.78168     351
     88.9965428   0.025534316   1.64640     3.86754     355
     92.5500844   0.026553876   1.70310     3.95140     360
     96.0968720   0.027571497   1.75948     4.03340     364
     99.6515203   0.028591374   1.81568     4.11332     368
    103.1916922   0.029607097   1.87141     4.19093     373
    106.7503441   0.030628123   1.92728     4.26776     377
```

Sample log

```
  Date   Time   Log Message

  7/12/2013    9:32:00 AM    Starting a sample analysis for C:\MicroActive
  7/12/2013    10:09:25 AM   Standard data collection started.
  7/12/2013    3:21:56 PM    New analysis conditions downloaded.
  7/12/2013    3:49:25 PM    Termination started.
  7/12/2013    4:22:47 PM    Finished a sample analysis for C:\MicroActive
```

D FREE SPACE CORRECTION

Free space is that volume of the sample tube which is unoccupied by the sample. The quantity of gas dosed into the sample tube is calculated from the difference in pressures in the manifold before and after the dose is delivered. The quantity of gas adsorbed by the sample is calculated by subtracting the quantity of gas remaining in the free space of the sample tube after equilibrium is established from the quantity of gas originally dosed into the sample tube. Free space must be determined accurately to obtain a precise value for quantity adsorbed.

Static-volumetric systems consist basically of a gas manifold joined to a sample tube by an isolation valve. The manifold section has connections for an absolute pressure transducer, a temperature gauge, and a vacuum system. It also has inlets for the adsorptive gas and helium. A dewar flask containing a cryogenic liquid (usually 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 analysis free space).

The analyzer provides the following methods for free space determination:

- Measure
- Calculate
- Enter

MEASURE

Generally, this method, although requiring a little more time (approximately 10 minutes), is the most preferred one for determining free space. It is simple, automatic, requires very little information, and essentially is error-proof. With this method, the instrument first evacuates the manifold and sample tube (containing sample), then isolates the sample tube by closing the valve. Then the manifold is charged with helium, the pressure measured, and the valve opened allowing the helium to expand into the sample tube at ambient temperature. Again the pressure is measured.

The dewar is raised and the sample tube is cooled to cryogenic temperature. Again pressure drops; when pressure has equilibrated, the value is recorded. Ambient and analysis 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 analysis free space at cryogenic temperature which requires correction for nonideality can be determined.

This method may be undesirable if:

- Helium is unavailable; free space determination by the analyzer requires the use of helium.
- Analysis speed is a major factor; a helium free space measurement of 10 to 15 minutes is required.
- The sample tends to absorb and retain helium for a prolonged period of time or if it adsorbs helium.

CALCULATE

This method is the most rapid and efficient way of compensating for free space. Ensure the following is accomplished:

- Perform a blank analysis on the sample tube.
- Load the blank analysis file data into the sample tube file.
- Enter the analysis bath temperature (found on the *p° and Temperature* window).
- Enter the sample mass and density (found on the *Sample Description* tab).

ENTER

This method allows for entering predetermined values for the ambient and analysis 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 MAINTAIN HIGH PURITY GASES

The analysis system was designed to accurately measure the surface area of all types of materials. It is important that the gases (especially krypton) used for these measurements be of highest purity, especially when analyzing low surface area samples. Three ways to ensure high purity gases are to always maintain:

- thoroughly purged gas pressure regulators
- non-permeable gas lines
- leak-free connections

Impure gas is strongly indicated, for example, if a series of measurements on a low surface area material yields decreasing specific surface areas with decreasing quantities of sample. The analyzer uses very small amounts of helium; therefore any residual air in the regulator can distort results of subsequent analyses for quite some time.

Micromeritics offers the following suggestions to assist in maintaining high purity gases (particularly helium):

- Use metal gas lines only
- Remove trapped air from the regulator and gas lines

USE METAL GAS LINES

Always use metal gas lines which have been carefully cleaned of any oils and greases used in the manufacturing process. Do not use plastic or rubber gas lines. When these types of permeable, nonmetallic gas lines are used with helium, contaminants accumulate at a much faster rate. This causes errors in analysis results and can also contaminate a clean sample.

REMOVE TRAPPED AIR

When connecting the regulator to the gas cylinder, air is unavoidably trapped on the high- and low-pressure sides of the regulator, as well as in the gas lines. Remove as much of this air as is possible *before* opening the gas cylinder valve. If this air is allowed to remain in the regulator, it will mix with the helium and cause inaccurate results in subsequent analyses. Or if the valve is open for any length of time, the air trapped on the high pressure side may diffuse back into the gas cylinder and contaminate its entire contents.

There are two methods for removing trapped air from the regulator lines: the Purge Method and the Evacuation Method.

PURGE METHOD

This is the preferred method for removing trapped air.

1. Go to **Unit [n] > Enable Manual Control**. Ensure a checkmark displays to the left of the menu item. If the analyzer schematic does not display, go to **Unit [n] > Show Instrument Schematic.**

> ✏️ If multiple instruments are installed, choose the correct *Unit* menu.

2. Close all valves by right clicking each valve, then click **Close**.
3. Open the regulator shutoff valve.
4. Open the gas cylinder valve **briefly** and allow the regulator to be charged with gas until the high-pressure gauge reads just over half the tank pressure, then quickly close the valve.

Low pressure gauge   High pressure gauge   Gas cylinder shut-off valve
Gas regulator shut-off valve   Copper tubing   Brass reducer fitting   Regulator connector nut   Regulator control knob
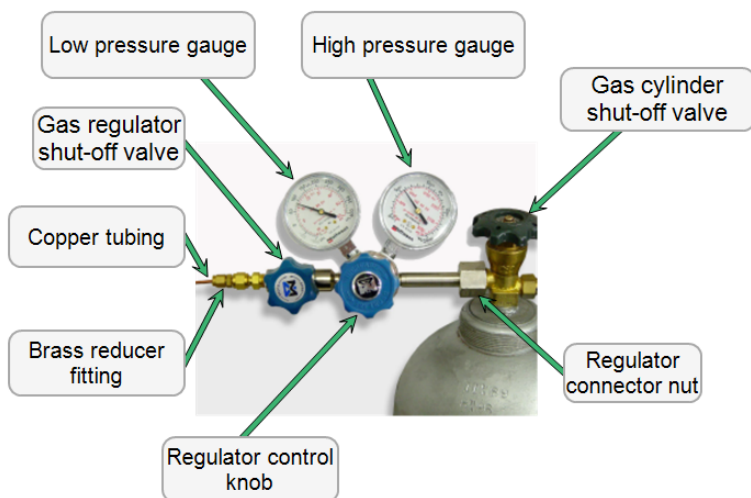
5. Use the Pressure Control knob to set the output pressure (gas cylinder pressure gauge) to 15 psig.
6. Loosen the fitting at the instrument helium inlet until the low pressure side drops to approximately 3 psig (0.02 MPa), then tighten the fitting.
7. Repeat steps 4, 5, and 6 three times.
8. Briefly open the gas cylinder valve, then use the Pressure Control knob to reset the regulator output pressure to 15 psig.
9. After the pressure has stabilized (indicating there are no leaks), open the gas cylinder valve.

EVACUATION METHOD

> To use this method, the gas tank must be within 10 feet of the instrument.

1. Do one of the following:

If...	Then...
The regulator has not been filled with gas and the gas line is attached to the instrument:	Close the gas cylinder valve.
	Open the regulator shutoff valve.
The regulator is filled with gas:	Close the gas cylinder valve.
	Open the regulator shutoff valve.
	Loosen the helium inlet fitting (or nut) on the instrument.
	Allow all of the gas in the regulator to expel from the line (pressure reading will be zero).
	Retighten the helium inlet fitting (or nut).

2. Go to **Unit > Enable manual control** (if the instrument schematic is not displayed, go to **Unit > Show instrument schematic**).

> If multiple instruments are installed, ensure the correct *Unit* menu is selected.

3. Close all valves, then open valves 6, 7, and 10.
4. Allow evacuation to continue for 20 minutes. This pulls a vacuum on the helium line to the gas cylinder. The manifold pressure transducer should fall close to zero.

> Allow evacuation for a full 20 minutes. If evacuation time is too short, trapped air may remain in the lines.

5. Close valves 6, 7, and 10.

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F WORKSHEETS

Worksheets in this section may be copied as needed.

Sample Data Worksheet for Gas Adsorption on the next page

mi micromeritics®

SAMPLE DATA WORKSHEET FOR GAS ADSORPTION

Sample tube identification: _____

Sample Mass (g)				
		Before Degas	After Degas	After Analysis
1.	Mass of empty sample tube set	g		
2.	Mass of sample tube set plus sample	g	g	g
3.	Mass of sample (step 2 minus step 1)	g	g	g

Degas Information	
Degas apparatus	
Temperature (°C)	
Time (hours)	
Actual time started	
Actual time finished	

Degas Notes:

_____

_____

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INDEX