

**3FLEX™**

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*SURFACE AND CATALYST CHARACTERIZATION ANALYZER*



***OPERATOR MANUAL***

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Sep 2016  
(Rev M)

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## ***CONTACT US***

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[Customer Support Portal](#)

<http://techsupport.micromeritics.com/portal>

## ABOUT THIS MANUAL



All references to 3500, 3Flex, or 3Flex 3500 in this document encompass the 3Flex and the MicroActive Share for 3Flex unless otherwise noted.

The following icons may be found in this document:

### Common Icons

Icon	Refers to
	Chemical Adsorption and Dynamic Analysis
	Physical Adsorption only
	Dynamic Analysis
	Chemical Adsorption only
	MicroActive Share only



**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.

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## **3FLEX LINKS**



For Calculations, Error Messages, Report Tutorials, and Parts and Accessories, click the links below or enter [www.micromeritics.com/3Flex\\_Links](http://www.micromeritics.com/3Flex_Links) in the address box on a device with internet connection.

[Calculations for 3Flex Software Version 4.x](#)

[Error Messages for 3Flex Software Version 4.x](#)

[Parts and Accessories for Physisorption](#)

[Parts and Accessories for Chemisorption](#)

[MicroActive Report Tutorials](#)

[Smart VacPrep Operator Manual](#)

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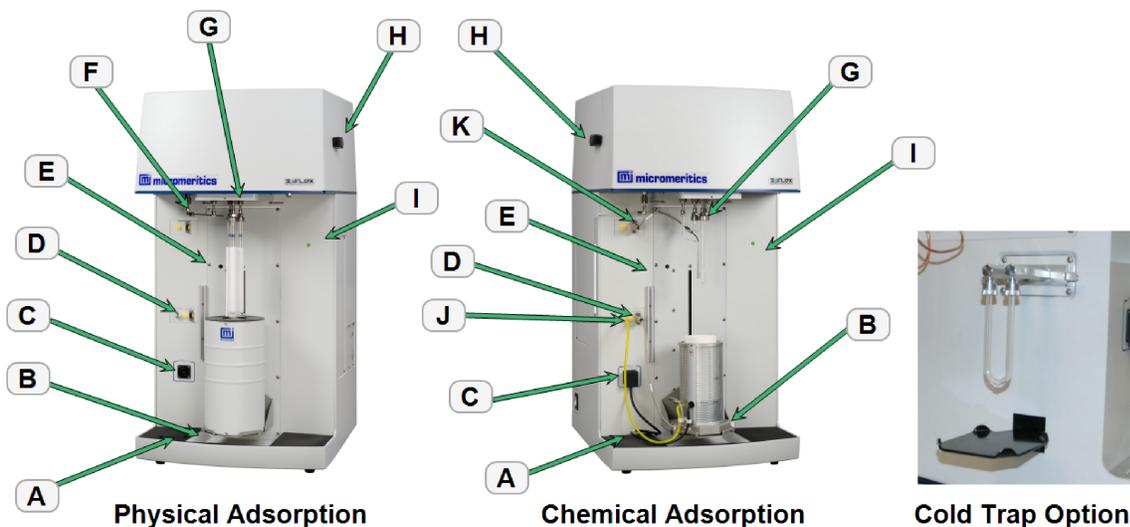
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## 1 ANALYZER COMPONENTS

### FRONT PANEL



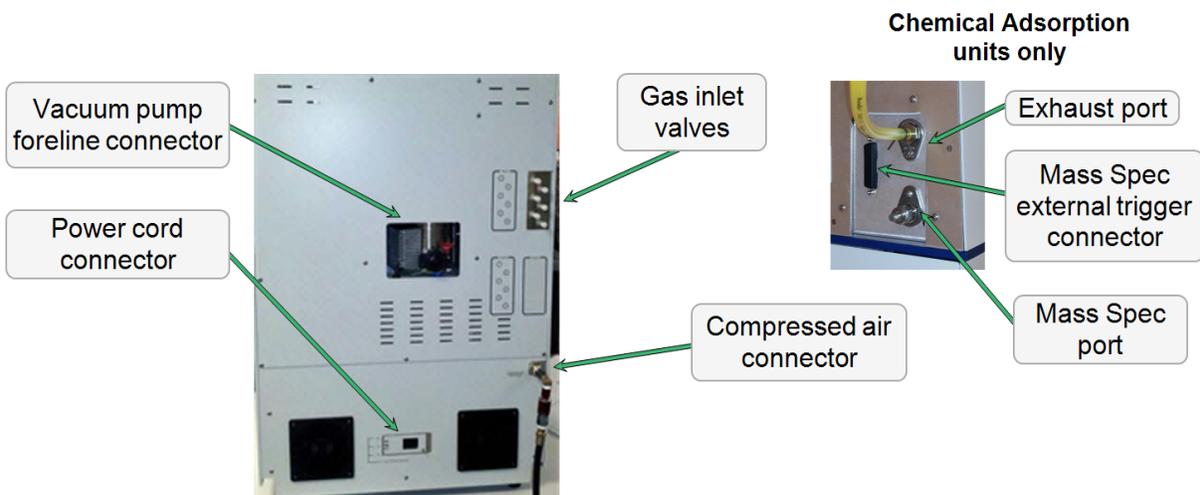
### Front Panel Components

Component	Description
<b>A</b>	Access panels to reset buttons Lift the pad and panel to access the reset buttons for the mantle, heater, and transformer.
<b>B</b>	Elevator The elevator raises and lowers automatically when the analysis is started and completed. During analysis, the elevator <i>optionally</i> lowers after the free-space measurement to allow evacuation, then raises and continues the analysis.
<b>C</b>	Mantle / Furnace power connector Power connector for the mantle or furnace.
<b>D</b>	Mantle / Furnace thermocouple connector Connector for the thermocouple.
<b>E</b>	Elevator reset button Resets the elevator in case of failure. The message <i>Elevator Circuit Breaker Open</i> on the analyzer schematic indicates this reset button should be pushed.
<b>F</b>	$P_0$ tube For measuring the saturation pressure.
<b>G</b>	Sample ports For installing up to three sample tubes.
<b>H</b>	Manifold compartment cover latch Holds the removable manifold compartment cover.
<b>I</b>	Power indicator light Blinks when power is applied to the analyzer; illuminates when analysis program is initiated and ready for operation.

**Front Panel Components (continued)**

<b>Component</b>		<b>Description</b>
<b>J</b>	Cooling gas port	Forces air into the furnace to cool it down.
<b>K</b>	Thermocouple	For chemical adsorption samples
	Cold Trap option	Attaches to the left side of the analyzer and removes water of reaction or other reaction products that might interfere with the operation of the thermal conductivity detector or to remove impure vapors in the analysis gas stream, especially water vapor.

## BACK AND SIDE PANELS



### Back and Side Panel Components

Component	Description
<b>Compressed air connector</b>	For compressed air supply for the pneumatically actuated, hard seal valves.
<b>Ethernet port</b> <i>(not shown)</i>	Located on the side panel. Port for an Ethernet cable allowing communication between the analyzer and the computer.
<b>Exhaust port</b>	The top port is to vent flowing chemical adsorption gas. The bottom port is used for the optional Mass Spectrometer.
<b>Gas inlet valves</b>	Inlet valves 1-6 for analysis gases.
<b>Mass Spec external trigger signal</b>	For connecting the external trigger signal for the optional Mass Spec.
<b>Mass Spec exhaust port</b>	For connecting the exhaust line for the optional Mass Spectrometer.
<b>Power switch</b> <i>(not shown)</i>	Located on the side panel. For powering the analyzer on and off.
<b>Power cord connector</b>	For setting the power voltage and connecting the analyzer to the power supply.
<b>RS-232 connector</b> <i>(not shown)</i>	Located on the side panel. Used to connect the Smart VacPrep.
<b>Vacuum pump foreline connector</b>	For attaching the dry diaphragm roughing vacuum pump hose.

## EQUIPMENT OPTIONS AND UPGRADES

Equipment Option	Description
<b>Chemical Adsorption</b>	The Chemical Adsorption option includes a Mass Flow Controller for precise flowing preparation of samples, a furnace to control sample temperature from ambient to 1100 °C, quartz flow-through sample tubes, an exhaust port for venting hazardous gases, a Mass Spec port and electronic trigger signal, and six additional gas inlets for a total of twelve. The single chemical adsorption port is port number 2. The Micropore Option is required for the chemical adsorption port.
<b>Chiller Dewar</b>	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.
<b>CryoStat</b>	A closed-cycle cryocooler based on the Gifford-McMahon (GM) refrigeration principle. It uses helium gas from a helium compressor to generate cryogenic temperatures. The cryostat eliminates the need for liquid nitrogen and can obtain temperatures below the 77 K of liquid nitrogen. The decibel rating of the CryoStat HC-4A Zephyr is 56 dBA at 1 meter.
<b>Micropore Option</b>	<p>Each port on the standard analyzer can be upgraded individually for high quality micropore analyses with 10 torr and 0.1 torr transducers on each micropore port. Any remaining ports continue to operate in standard mode.</p> <p>The micropore option is required to run krypton.</p>
<b>Thermal Conductivity Detector (TCD)</b>	<p>With the TCD option, dynamic analyses provide the ability to perform temperature programmed reduction (TPR), oxidation (TPO), desorption (TPD), and reactions (TPRx).</p> <p>Provides the capability to investigate temperature dependence of specific adsorption or desorption process profiles for catalyst and adsorbents, as well as pulse chemical adsorption.</p>
<b>Vacuum Pump</b>	The analyzer requires a dry roughing vacuum pump for sample analysis. Appropriate vacuum pumps are available from Micromeritics.
<b>Vapor Adsorption Option</b>	<p>Vapor adsorption allows analyses with hydrocarbon vapors or water vapor. The analyzer allows for dosing from one dedicated sample port to the other two sample ports.</p> <p>A vapor adsorption option provides for dosing from a reservoir attached to the Psat port to all three sample ports. The vapor option includes a stainless steel chamber with a hard seal, manual cutoff valve to be attached in place of the Psat tube, and a heating mantle to control the temperature of the chamber at an operator-specified temperature between ambient and 43 °C.</p>

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

### ***DEGASSER OPTIONS***

Degasser Option	Description
<b>FlowPrep 060</b>	<p>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.</p>
<b>Smart VacPrep</b>	<p>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. Convenient front panel buttons allow QuickStart operation with preprogrammed conditions.</p> <p>Up to three additional Smart VacPrep instruments can be connected to one computer permitting 24 preparation ports to be used. The Smart VacPrep is the recommended degassing unit.</p>
<b>SmartPrep</b>	<p>The SmartPrep passes flowing-gas over the sample at elevated temperatures. It contains six sample ports in which temperature, ramp rates, and soak times are individually controlled by the analyzer program so that all degas information is integrated into the sample data file for future reference. It contains 2 serial ports, one for connecting to the computer and the other for connection of up to 3 additional SmartPrep instruments permitting 24 preparation ports to be used.</p>
<b>VacPrep 061</b>	<p>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.</p>

## ***GAS REQUIREMENTS***

Compressed gases are required for analyses. Gas cylinders or an outlet from a central source should be located near the analyzer.

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. For parts and accessories, see [\*3Flex Links on page iv\*](#).

## SPECIFICATIONS FOR THE 3500 3FLEX

Specification	Description
<b>Cabinet Temperature</b>	Upper cabinet is maintained at 45 °C providing a stable environment for: <ul style="list-style-type: none"> <li>• Dosing volumes</li> <li>• Pressure transducers</li> <li>• Thermal conductivity detector (TCD option)</li> </ul>
<b>Control of cryogen level on sample tube</b>	Isothermal jacket (for use with physical adsorption tubes only)
<b>Degas</b>	3 in situ, 6 additional with optional Smart VacPrep
<b>Dewar</b>	3.2 L capacity <ul style="list-style-type: none"> <li>• &gt; 80 hrs (single tube, no isothermal jacket)</li> <li>• &gt; 70 hrs (3 sample tubes, isothermal jackets, P<sub>0</sub> tube)</li> </ul>
<b>Electrical</b>	<b>Voltage.</b> 100 / 115 / 230 VAC <b>Frequency.</b> 50 / 60 Hz <b>Power.</b> 1350 VA
<b>Furnace</b> 	<b>Range.</b> Ambient +5 °C to 1100 °C <b>Accuracy.</b> ±1% <b>Ramp Rates:</b> 1 to 100 °C/min up to 800 °C 1 to 50 °C/min from 800 to 1000 °C 1 to 25 °C/min from 1000 to 1100 °C
<b>Gas Flow Control</b> 	200 sccm mass flow controller: <ul style="list-style-type: none"> <li>• sample preparation in chemical adsorption analyses</li> <li>• carrier gas in dynamic analyses</li> </ul> <b>Accuracy.</b> ±1% of setpoint
<b>Gas Inlets</b>	<ul style="list-style-type: none"> <li>• 6 for physical adsorption</li> <li>• 12 for chemical adsorption (can be used for physical adsorption and chemical adsorption)</li> <li>• 4 additional for dynamic analysis loop</li> </ul>
<b>Heating Mantle</b>	Temperature to 450 °C
<b>Loop</b> 	For use with level 2 analyzers only. <b>Gas inlets.</b> 4 <b>Volume.</b> 0.5 cm <sup>3</sup> nominal (other volumes are available) <b>Control.</b> Automatic electric rotary valve
<b>Manifold Outgas Rate</b>	< 0.1 µm/min

Specification	Description
<b>Mass Spectrometer Port</b> 	Dedicated and heated
<b>Minimum Measurable Surface Area</b>	0.01 m <sup>2</sup> /g
<b>Physical</b>	<b>Height.</b> 112 cm (44 in.) <b>Width.</b> 60 cm (23.5 in.) (TCD option adds 15 cm (6 in.) for cold trap) <b>Depth.</b> 62 cm (24.5 in.) (additional 15 cm (6 in.) needed for rear connections) <b>Weight.</b> 103 - 106 kg (227 - 234 lb) depending on the configuration
<b>Pressure Transducers</b>	<b>1000 mmHg.</b> ±0.12% of reading accuracy display resolution 0.01 mmHg <b>10 mmHg.</b> ±0.12% of reading accuracy display resolution 0001 mmHg <b>0.1 mmHg.</b> ±0.15% of reading accuracy display resolution 0.000001 mmHg
<b>Sample Analysis Ports</b>	<ul style="list-style-type: none"> <li>•1, 2, or 3 micropore-capable ports</li> <li>•1 chemical adsorption-capable port</li> </ul>
<b>Sample Tubes</b>	<ul style="list-style-type: none"> <li>•12 mm and 9 mm flat bottom physical adsorption tubes</li> <li>•12 mm standard and extended capacity flow-through quartz chemical adsorption tubes</li> </ul>
<b>Vacuum System</b>	Turbo molecular drag pump in series with four-stage diaphragm pump <b>Pumping Speed.</b> 3 L/sec (hydrogen), 61 L/sec (nitrogen) <b>Ultimate Vacuum.</b> $3.75 \times 10^{-10}$ mmHg <b>Vacuum Gauge.</b> Dual cold cathode / MicroPirani™ gauge
Computer	
<ul style="list-style-type: none"> <li>• <b>Operating System.</b> Windows 7 Professional or higher operating system is recommended for the best user experience.</li> <li>• <b>Desktop Installation Required.</b> The application should not be installed on a network drive with shared access. Multiple users cannot operate the application at the same time.</li> <li>• <b>10 Base T Ethernet Port.</b> 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.</li> <li>• <b>Read/Write Permissions.</b> All users of the application will need Read / Write permission to all directories and subdirectories where the application is installed.</li> <li>• <b>Continuous Internet Connection.</b> MicroActive Share installations require access to the Share servers at all times.</li> <li>• <b>Drives.</b> CD-ROM drive and thumb drive.</li> </ul>	
<i>In keeping with a policy of ongoing product improvement, specifications are subject to change without notice.</i>	

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## 2 ABOUT THE SOFTWARE

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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.

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 cut-and-paste graphics, are easily generated. Customized reports can be generated to screen, paper, or exported for use in other programs. There are two report functions:

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

Report options can be specified when creating the sample 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.

### ***PEAK EDITOR FOR DYNAMIC ANALYSIS***

See [Peak Editor for Dynamic Analysis on page 7 - 11](#)

The TCD (Thermal Conductivity Detector) option features a Peak Editor which allows the evaluation of results, peak editing, and reports. Adjusting peak boundaries can be used to eliminate baseline noise or other undesirable effects. The Peak Editor also allows the separation of composite peaks.

## SOFTWARE SETUP



If a software update is required for the analyzer to support the Smart VacPrep, the update can be downloaded from the internet at this by going to [www.Micromeritics.com](http://www.Micromeritics.com) and logging in.



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.

## SOFTWARE UPDATES

To run SmartVacPrep, the application software must include support for this accessory. On the internet, go to [www.Micromeritics.com](http://www.Micromeritics.com) and log in to your customer portal to 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.

## UNINSTALL THE SOFTWARE

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

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

## MENU STRUCTURE

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

### Main Menu Bar Options

Option	Description
<b>File</b>	Use to manage files used by the application — such as sample files, analysis conditions files, report options files, etc.
<b>Unit [n]</b>	Use to perform analyses, calibrations, and other analyzer operations. <i>Unit [n]</i> displays on the menu bar for each analyzer attached to the computer.
<b>Smart VacPrep</b>	Use to access the <i>Unit [n]</i> menu for each Smart VacPrep for starting degas operations, calibrations, and other operations.  Also used to install or remove a Smart VacPrep degasser. The Setup program can also be used to install a Smart VacPrep. See <a href="#">Software Setup on the previous page</a> .
<b>Reports</b>	Use to run reports and view the results.
<b>Options</b>	Use to edit the default method, specify system configuration, specify units, and change presentation options.
<b>Window</b>	Use to manage open windows and display a list of open windows. A checkmark appears to the left of the active window.
<b>Help</b>	Provides access to the online operator manual, the Micromeritics web page, the analyzer web page, and information about the analyzer.

## COMMON FIELDS AND BUTTONS

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

Common Fields and Buttons Table

Field or Button	Description
<b>Add Log Entry</b>	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.
<b>Autoscale</b>	When enabled on report parameters windows, allows the x- and y-axes to be scaled automatically. <i>Autoscale</i> means that the x- and y-ranges will be set so that all the data is shown. If <i>Autoscale</i> is not selected, the entered range is used.
<b>Axis Range</b>	On report parameters windows, the <i>From / To</i> fields are enabled when <i>Autoscale</i> options are not selected. Enter the starting and ending values for the x- and/or y-axes.
<b>Bar Code</b>	Enter bar code reader information if a bar code reader is connected to the computer's USB port. If a bar code reader is not used, this alphanumeric field can be used to enter additional information about the sample, such as a sample lot number, sample ID, etc.
<b>Browse</b>	Searches for a file.
<b>Cancel</b>	Discards any changes or cancels the current process.
<b>Close</b>	Closes the active window.
<b>Close All</b>	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.
<b>Comments</b>	Enter comments about the sample or analysis. Comments display in the report header.
<b>Delete</b>	When working with report parameters, <b>Delete</b> removes the selected report. Deleted reports will have to be regenerated if deleted in error.
<b>Destination</b>	Select the report destination.  <b>Preview.</b> Previews the predefined report on the screen.  <b>Print.</b> Sends the report to the default printer.  <b>Copies.</b> Select the number of copies to print. This field is only enabled when <i>Print</i> is selected.

Common Fields and Buttons Table (continued)

Field or Button	Description
	<b>File.</b> Select the destination directory. Enter a new file name in the <i>File name</i> field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
<b>Edit</b>	When working with report parameters, highlight the item in the <i>Selected Reports</i> list box and click <b>Edit</b> to modify the report details.
<b>Exit</b>	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.
<b>Export</b>	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.
<b>File name</b>	Select a file from either the <i>Name</i> column or from the library.
<b>From / To</b>	When working with report parameters windows, enter the <i>From</i> and <i>To</i> range for x- and/or y-axes.
<b>List</b>	Provides the option to create a list of sample or report options file information, for example, file name, date / time the file was created or last edited, file identification and file status.
<b>Name column</b>	A list of files in the selected directory or library.
<b>Next</b>	Click to move to the next window or next step.
<b>OK</b>	Saves and closes the active window.
<b>Open</b>	Opens the selected file. Alternatively, double click the file name in the <i>Name</i> column to open the file.
<b>Prev</b>	Click to move to the previous window.
<b>Preview</b>	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.
<b>Print</b>	Sends the report to the selected destination (screen, printer or file).
<b>Remove</b>	Click to remove an item from the list.
<b>Replace</b>	Click to select another file where the values will replace the current file's values.
<b>Replace All</b>	Click to select another .SMP file where the values will replace all values for the active sample file. The original file will remain unchanged.

## Common Fields and Buttons Table (continued)

Field or Button	Description
<b>Report</b>	<p>Click to display a window to specify report output options.</p> <p><b>Start Date.</b> Displays a calendar to select the start date for the report.</p> <p><b>Preview.</b> Previews the predefined report on the screen.</p> <p><b>Print.</b> Sends the report to the default printer.</p> <p><b>Copies.</b> Select the number of copies to print. This field is only enabled when <i>Print</i> is selected.</p> <p><b>File.</b> Select the destination directory. Enter a new file name in the <i>File name</i> field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.</p>
<b>Save</b>	Saves changes to the active window.
<b>Save As</b>	Saves a file in the active window under a different file name.
<b>Start</b>	Starts the report, test, analysis, or operation.
<b>Table</b> buttons	<p>Use to modify the table contents.</p> <p><b>Insert.</b> Inserts one row above the selected row.</p> <p><b>Delete.</b> Deletes the selected row.</p> <p><b>Clear.</b> Clears all table entries and displays only one default value.</p> <p><b>Append.</b> Inserts one row at the end of the table.</p>
<b>View</b>	<p><b>Operation.</b> Use to display the current mode of operation.</p> <p><b>Instrument Log.</b> Use to display recent analyses, calibrations, errors or messages.</p> <p><b>Instrument Schematic.</b> Use to display a schematic of the analyzer system.</p>
<b>View Instrument Log</b>	For use by a Service Technician. Operators should use <b><i>Unit [n] &gt; Show Instrument Log.</i></b>

## FILE STATUS, DESCRIPTION, EXTENSION, AND LOCATION

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

### File Status and Description Table

File Status	Description
Analyzing	Sample 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 Table

File Type	File Name Extension	Default Location
Alpha-s curve	.ALS	Param Directory
Adsorptive properties	.ADP	Param Directory
Analysis conditions	.ANC	Param Directory
Calibration File	.CAL	
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

**File Type, Extension, and Location Table**

<b>File Type</b>	<b>File Extension</b>
<b>Portable document format</b>	.PDF
<b>Report</b>	.REP
<b>Spreadsheet</b>	.XLS
<b>Unicode</b>	.TXT

## KEYBOARD SHORTCUTS

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

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



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

### Keyboard Shortcut Table

Field or Button	Description
<b>Alt + [Unit n]</b>	Opens the <i>Unit [n]</i> menu.
<b>Alt + F</b>	Opens the <i>File</i> menu.
<b>Alt + F4</b>	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
<b>Alt + H</b>	Opens the <i>Help</i> menu.
<b>Alt + I</b>	Opens the <i>Options</i> menu.
<b>Alt + R</b>	Opens the <i>Reports</i> menu.
<b>Alt + V</b>	Opens the <i>Smart VacPrep</i> menu.
<b>Alt + W</b>	Opens the <i>Window</i> menu.
<b>Shift + F9</b>	Opens the shortcut menu of (1) selected component on analyzer schematic when manual control is enabled or (2) onscreen reports.
<b>Ctrl + N</b>	Opens a new sample file.
<b>Ctrl + O</b>	Opens the <i>File Selector</i> window.
<b>Ctrl + P</b>	Opens the <i>File Selector</i> to start a report from a selected .SMP file.
<b>Ctrl + S</b>	Saves the open file.
<b>F1</b>	Opens the online help operator manual.
<b>F2</b>	Opens the <i>File Selector</i> window.
<b>F3</b>	When in the <i>File Selector</i> window, opens the file search box.
<b>F4</b>	When in the <i>File Selector</i> window, opens the address bar.
<b>F6</b>	Cascades open windows.

**Keyboard Shortcut Table (continued)**

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

## OPTION PRESENTATION

### Options > Option Presentation

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

#### Option Presentation Display Table

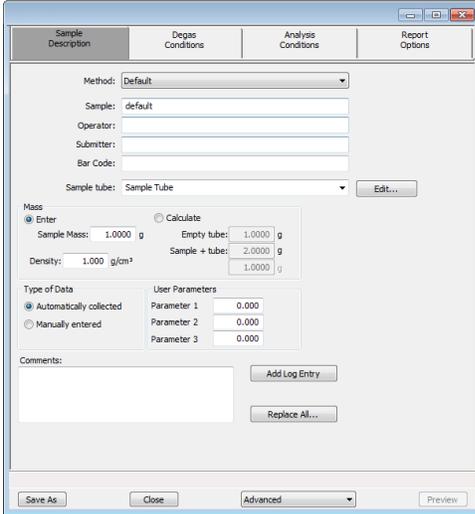
Presentation Display	Description
<b>Advanced</b>	Displays all parts of sample information and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
<b>Basic</b>	Displays sample information in a single window. This display option is used after the parameter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

- **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.
- **Check Shield.** When enabled, checks to ensure the shield is in place around the dewar or furnace prior to starting an analysis. If this option is selected and the dewar or furnace shield is not in place prior to starting an analysis, a warning message displays on the analyzer schematic window. An entry is made in the analyzer log regardless of operator choice.

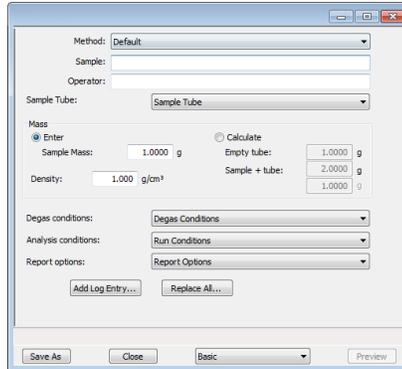


Specify or change the default option presentation by selecting **Options > Option Presentation**, or select *Basic* or *Advanced* from the drop-down list at the bottom of the window.

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



**Advanced presentation option**



**Basic / Restricted presentation option**



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.

## **LIBRARIES**

### **Options > Manage Libraries**



This feature is available only for 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 instant access at once to all of those files. Even though libraries do not store actual sample and parameter files, folders can be added or removed within each library.

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

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

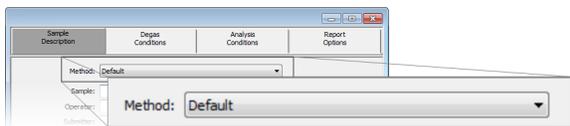
**File > New Method**

**Options > Default Method**

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

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 Files

Method Selected	Default File Modified
Physical Adsorption	3500.SMP
Chemical Adsorption	3500Chemi.SMP
Dynamic Analysis	3500DynamicChemi.SMP

## CONFIGURE THE ANALYZER

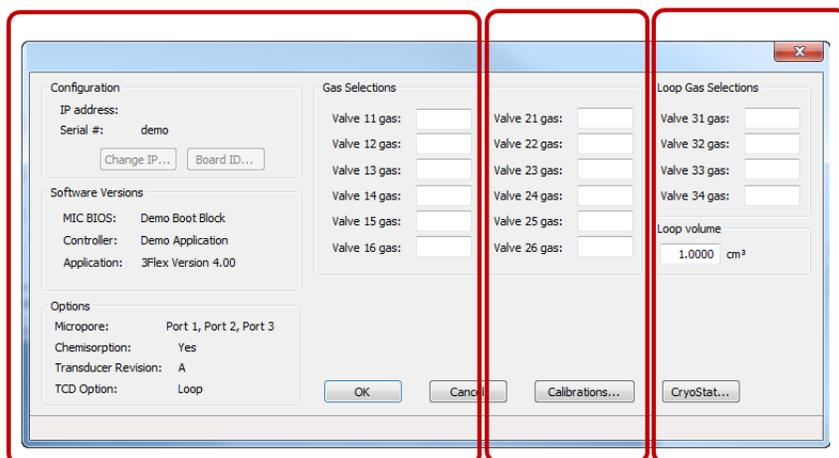
### SPECIFY GAS PORTS

#### Unit [n] > Unit Configuration

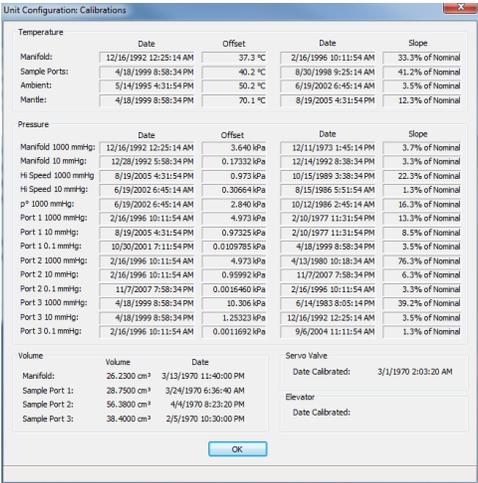
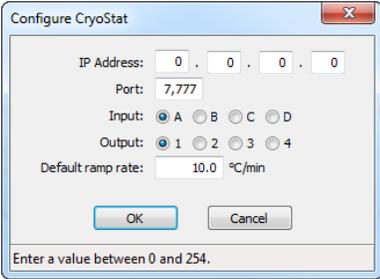
Use to display hardware/software configurations, calibrations, and gas selections of the connected analyzer.

The analyzer has gas inlets for up to six analysis gases (12 with the chemical adsorption option, plus four loop gas inlets with the TCD Loop option). The gases connected to the inlets must be specified in the analysis program. If the gas is changed on one of the inlets, the same change must be made on the *Unit Configuration* window. The analysis software must be kept informed of any changes in gases.

Enter the gas mnemonic in the gas fields to indicate the type of gas connected to that valve.



## Unit Configuration Fields and Buttons Table

Field or Button	Description
<b>Calibrations</b> [ <i>button</i> ]	<p>Displays calibration information for analyzer components.</p> 
<b>Configuration</b> [ <i>group box</i> ]	<p>Displays the IP address used by the analysis program and the serial number of the selected analyzer.</p> <p><b>Change IP.</b> Displays the <i>Unit IP Setup</i> window. The IP address and subnet mask assigned during installation display. Do not edit these fields unless instructed by a Micromeritics service representative.</p> <p><b>Board ID.</b> Displays information from the electronic circuit boards in the instrument. These parameters cannot be edited.</p>
<b>CryoStat</b> [ <i>button</i> ]	 <p><b>IP Address.</b> The IP address of the cryostat temperature controller.</p> <p><b>Port.</b> The computer port the cryostat temperature controller is attached to.</p> <p><b>Input.</b> Temperature sensor input channel.</p> <p><b>Output.</b> Heater output channel.</p>

## Unit Configuration Fields and Buttons Table (continued)

Field or Button	Description
	<b>Default ramp rate.</b> The rate at which the temperature will change after evacuation while advancing to the hold temperature.
<b>Gas Selections</b> [group box]	Enter the mnemonics for the analysis gases attached to inlet valves.  Valves 11-16. <b>PC</b>  Valves 21-26. <b>C</b> With chemical adsorption option.  Valves 31-24. <b>DA</b> With loop configuration
<b>Loop Volume</b> [text box] <b>DA</b>	Enter the loop volume for TCD.
<b>Options</b> [group box]	Displays options installed on the analyzer.
<b>Software Versions</b> [group box]	Displays the software versions of the MIC BIOS, controller, and analysis program.
	For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4.</a>

## ***SPECIFY THE TYPE OF ANALYZER***

***Unit [n] > Physical Adsorption***

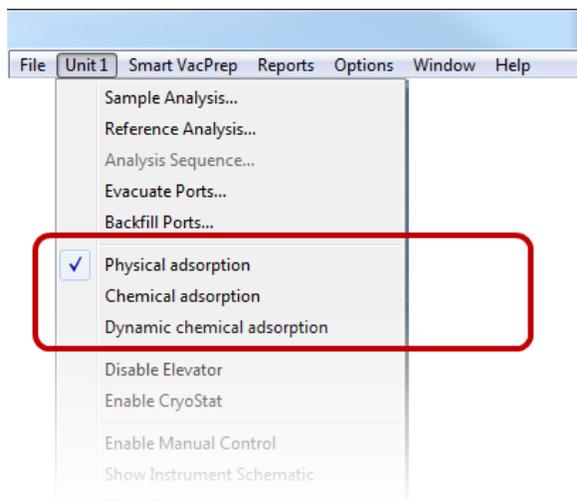
***Unit [n] > Chemical Adsorption***

***Unit [n] > Dynamic Analysis***



This topic does not apply if physical adsorption units only are installed.

Up to four analyzers can be connected, and each analyzer may be either physical adsorption or chemical adsorption. When multiple units are installed, the application's title bar reflects the number of analyzers installed and the type of analyzer.



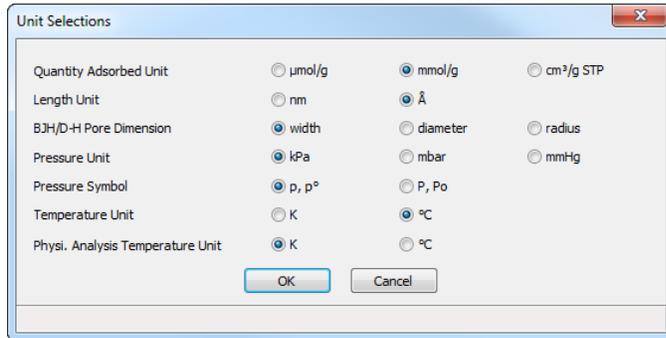
The analysis type affects the appearance of the analysis sample selection window, the analyzer schematic, and the status window. This menu selection is disabled when an analysis is being performed.

When application windows are opened, the title bar reflects both the serial number and the analysis type. The title bar changes when the analysis type changes.

## SPECIFY UNIT SELECTIONS

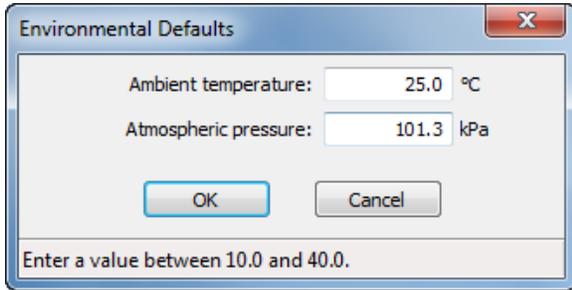
### Options > Units

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



**ENVIRONMENTAL DEFAULTS FOR TCD ANALYZERS**

**Options > Environmental Defaults**

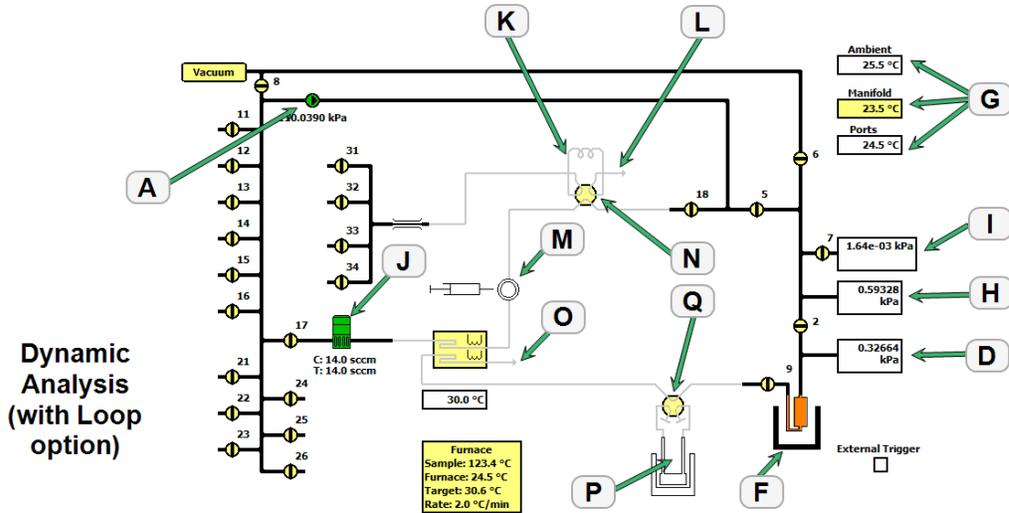


To ensure accurate loop calibration, enter current environmental conditions daily, or whenever there is a significant change in one or more of the environmental conditions. These values are used in some calculations to account for the effect of environmental conditions on the analysis. These parameters are used in the Dynamic Analysis reports.

**Environmental Defaults Fields and Buttons Table**

Field or Button	Description
<b>Ambient temperature</b> [text box]	The temperature of the room where the analyzer is located.
<b>Atmospheric pressure</b> [text box]	The atmospheric pressure of the room where the analyzer is located.
 <b>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</b>	

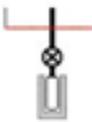




**Analyzer Schematic Icon Table**

Icon or Symbol	Description
	<b>Open Valve.</b> Green indicates an open valve.
	<b>Closed Valve.</b> Yellow indicates a closed valve. When manual control is disabled, closed valves appear white.
	<b>Servo Valve. Closed.</b>
	<b>Servo Valve. Open.</b>
	<b>Elevator.</b>
	<b>Sample Tube and Furnace Elevator.</b> The sample tube icon is white when the sample and furnace temperatures are 50 °C or lower. If either the sample or furnace temperature exceeds 50 °C, the sample tube icon turns orange. Temperature readings and ramp rate are displayed below and to the left of the icon. The furnace icon resides on the elevator.
	<b>Mass Flow Controller (MFC).</b> Controls the flow of gas into the sample port. The current (C) rate and the target (T) rate are shown to the right of the MFC icon. Applicable only for the gas used in the flowing operations. The mass flow controller constant is preset for gases provided with the application. See <a href="#">Chemical Adsorption Tasks on page 4 - 13</a> .

## Analyzer Schematic Icon Table (continued)

Icon or Symbol	Description
	<b>Sample Tube.</b> Cannot be manually controlled.
	<b>PISC</b> A warning that indicates the safety shield is not in place.
	<b>Vapor Source with Heating Mantle.</b> Also displays the current temperature, the target vapor source temperature, and rate of temperature increase.

## Analyzer Schematic Components Table

Schematic Components	Description
1-3	Sample ports
4	Po port
5	Servo isolation valve
6	Manifold vacuum
7	Vacuum gauge isolation valve
8	Inlet vacuum
9	Exhaust valve for chemical adsorption only
10	Reference volume (shown in Service Test mode only)
11-16 and 21-26	Inlet valves
31-34 *	<b>DA</b> Loop gas inlet valves
A	Servo valve
B	Po pressure
C	Port 1 pressure
D	Port 2 pressure
E	Port 3 pressure
F	Elevator
G	Temperature sensors
H	Manifold pressure
I	Vacuum gauge

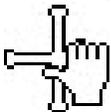
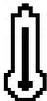
**Analyzer Schematic Components Table (continued)**

Schematic Components	Description
J	 Mass flow controller
K *	 Injection loop
L *	 Loop gas exhaust port
M	 Septum
N *	 Loop rotary valve
O	 Chemi exhaust port
P	Cold trap
Q	Cold trap rotary valve
* Displays only if the TCD Loop Valve option is installed	

### Instrument Schematic Shortcut Menus

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

#### Schematic Shortcuts Table

Schematic Shortcut Icon	Available Options:
<b>Valve options</b> 	<p><b>Automatic.</b> Automatically operates the servo valve during dosing or evacuation. Enter the target pressure.</p> <p><b>Close.</b> Closes the selected valve.</p> <p><b>Direct.</b> Used in Service Test mode only under the direction of a Micromeritics service representative.</p> <p><b>Open.</b> Opens the selected valve.</p> <p><b>Pulse.</b> Use to quickly turn the valve on and off allowing the operation to proceed in small increments.</p>
<b>Elevator options</b> 	<p><b>Raise.</b> Select <i>Raise</i> to raise the elevator. When it is moving, press the keyboard space bar to stop the movement (or right click and select <i>Stop</i> from the menu).</p> <p><b>Lower.</b> Select <i>Lower</i> and press the keyboard space bar to lower the elevator.</p> <p><b>Stop.</b> Stops the elevator from moving.</p>
<b>Temperature control options</b> 	<p><b>On.</b> Enables the temperature control.</p> <p><b>Off.</b> Disables the temperature control.</p> <p><b>Set.</b> Select to set the following:</p> <ul style="list-style-type: none"> <li>• Enable or disable temperature control</li> <li>• Control sample temperature</li> <li>• Control furnace temperature</li> <li>• Cool the sample to less than 50 °C</li> <li>• Set heater power percent</li> </ul>

## PRESSURE TRANSDUCERS

The schematic shows the pressure transducers present in the system.

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

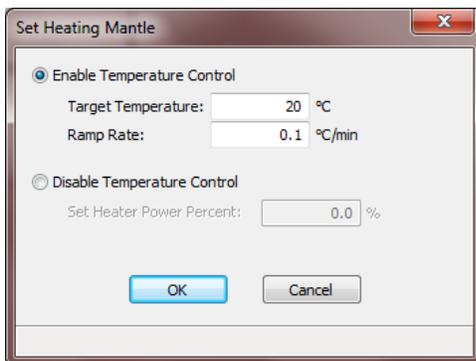
When multiple pressure transducers are present in a port or the manifold, the display automatically changes to show the pressure reading from the lowest range transducer currently on the scale.

## HEATING MANTLE

When samples are being degassed on the analysis ports, the following icons appear beneath the elevator. When the mantle temperature is above 50 °C, the sample tube icons (not shown below) turn orange. If using a GlasCol heating mantle, the sample temperature shows in the yellow *Degas Mantle* box.



Right click the *Degas Mantle* box to set the target temperature or the rate of temperature increase.



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

## SHOW STATUS

### Unit [n] > Show Status

Use to show the current status for each port.

1:	Preliminary	Analysis	Termination			
Sample:	Last Point	p (mmHg)	p/p'	Q (cm <sup>3</sup> /g STP)	p' (mmHg)	Run Time
Analysis	24 of 30	564.000000	0.917000000	37.0000	774.000	4:43
Details:						

2:	Preliminary	Analysis	Free Space	Termination		
Sample:	Last Point	p (mmHg)	p/p'	Q (cm <sup>3</sup> /g STP)	p' (mmHg)	Run Time
Analysis	24 of 30	564.000000	0.917000000	37.0000	774.000	4:43
Details:						

3:	Preliminary	Analysis	Termination			
Sample:	Last Point	p (mmHg)	p/p'	Q (cm <sup>3</sup> /g STP)	p' (mmHg)	Run Time
Analysis	29 of 30	563.000000	0.967000000	42.0000	779.000	5:33
Details:						

Physical Adsorption

Status		Preliminary	Analysis	Termination	
Sample:	Last Point	P (mmHg)	Est. Qty. Ads. (cm <sup>3</sup> /g STP)	Run Time	Manifold Gas
Details:					

Chemical Adsorption

Sample:	Temperature:
Description:	TCD Reading:
Step:	Recording Time:
Status:	Run Time:

Dynamic Analysis

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

## 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
- Manifold temperature statistics
- Nitrogen P<sub>0</sub> 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 and Descriptions Table

Field or Button	Description
<b>Analyses completed / started</b>	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.
<b>Days until roughing-pump service is due</b>	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.

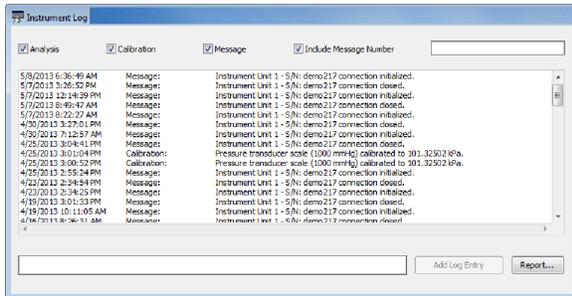
**Dashboard Gauges and Descriptions Table (continued)**

Field or Button	Description
<b>Manifold outgas rate</b>	<p>Provides the qualitative indication of the outgas rate in the dosing manifold. LED images constitute a bidirectional bar graph of the outgas rate.</p> <p>The gauge is updated when the <i>Analysis Manifold Test</i> is run. See <a href="#">Schedule Diagnostic Tests on page 9 - 3</a> and <a href="#">Start Diagnostic Test on page 9 - 1</a>.</p> <ul style="list-style-type: none"> <li>• Three green LEDs are lit if outgas rate is below 30% of outgas rate limit.</li> <li>• At 30%, the left LED turns off.</li> <li>• At 60%, the center LED turns off.</li> <li>• At 90%, three green LED lights turn off and the center yellow LED is turned on.</li> <li>• At 110% and above, only the red LED is lit and attention is required.</li> </ul>
<b>Manifold temperature</b>	<p>Displays the statistics of the manifold temperature reading. The mean, the value at two standard deviations, the minimum, and the maximum display.</p>
<b>Nitrogen Po</b>	<p>Displays statistics of the saturation pressures measured with nitrogen gas at liquid nitrogen temperatures. The mean, two-sigma, minimum, and maximum values display.</p> <p>The gauge is updated when a Po is logged with nitrogen as the adsorptive and a bath temperature of <math>77 \pm 2</math> K.</p>

## SHOW INSTRUMENT LOG

### Unit [n] > Show Instrument Log

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



### Instrument Log Fields and Buttons Table

Field	Description
<b>Analysis / Calibration / Message [selection]</b>	Select the logs to display.
<b>Filter by Message Number [text box]</b>	<p>Enter any of the following information in the message text box to generate a log report that includes the entered text:</p> <ul style="list-style-type: none"> <li>enter the message number in the text box to view all occurrences of the entered message,</li> <li>enter an asterisk in the message box to see all numbered messages, or</li> <li>enter several message numbers separated by commas to include only messages with those numbers.</li> </ul> <p>Numbered messages contain more detailed information about analyzer operation.</p>
<b>Report [button]</b>	Click to select print destination and report start date.
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

## EXPORT FILES

### File > Export

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

## 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.

File Listing					
No.	File Name	Date	Time	File Identification	Status
1	000-000.SMP	2/5/2014	11:02:22 AM	Nitrogen Reference Material Silica Alumina	Complete
2	000-002.SMP	2/5/2014	11:06:48 AM	000-002	No Analysis
3	000-003.SMP	2/5/2014	11:02:46 AM	000-002	Complete
4	000-004.SMP	2/5/2014	11:02:50 AM	Nitrogen Reference Material Silica Alumina	No Analysis
5	000-012.SMP	8/5/2015	1:55:42 PM	000-012	No Analysis
6	000-035.SMP	9/15/2015	12:52:40 PM	000-035	No Analysis
7	000-038.SMP	9/11/2015	8:17:01 AM	000-038	No Analysis
8	000-039.SMP	9/11/2015	8:33:43 AM	000-039	No Analysis
9	000-040.SMP	9/11/2015	9:50:59 AM	000-040	No Analysis
10	1-1.SMP	2/5/2014	11:56:54 AM	000-002	No Analysis

## ***ANALYSIS TYPES FOR TCD ANALYZERS***

The basic concept for all analyses is the same — the filament detects changes in the gas mixture flowing past it. The sample, gas selection, and analysis conditions determine what changes occur.

### ***TEMPERATURE PROGRAMMED DESORPTION ANALYSIS***

Temperature Programmed Desorption (TPD) analyses determine the number, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of analysis gas flows through the sample and adsorbs on the active sites. Programmed desorption begins by raising the temperature linearly with time while a steady stream of inert carrier gas flows through the sample.

At a certain temperature, the heat overcomes the activation energy; therefore, the bond between the adsorbate and adsorbent will break, and the adsorbed species desorb. If different active metals are present, they usually desorb the adsorbed species at different temperatures. These desorbed molecules enter the stream of inert carrier gas and are swept to the detector, which detects change in gas composition. The volume of desorbed species, combined with the stoichiometry factor and the temperature at which pre-adsorbed species desorb, yields the number and strength of active sites.

#### **Temperature Programmed Desorption**

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- Heated pretreatment
- Active sites to probe
- Active gas to probe sites (NH<sub>3</sub>, CO<sub>2</sub>, H<sub>2</sub>, etc.)
- Material (sample) does not change during temperature ramp

#### **Temperature Programmed Decomposition**

---

- No pretreatment
- No active sites to probe
- Only inert gas is used
- Material changes throughout temperature ramp

Observe changes of gas phase species during temperature ramp.

## TEMPERATURE PROGRAMMED REDUCTION ANALYSIS

Temperature Programmed Reduction (TPR) determines the number of reducible species present in the catalyst and reveals the temperatures at which the reduction occurs.

The TPR analysis begins by flowing analysis gas (typically hydrogen in an inert carrier gas such as nitrogen or argon) through the sample, usually at ambient temperature. While the gas is flowing, the temperature of the sample is increased linearly with time and the consumption of hydrogen by adsorption/reaction is monitored. Changes in the concentration of the gas mixture are determined. This information yields the hydrogen uptake volume.



When using any mixture of gases for TPR or TPO analyses, ensure the thermal conductivities of the two gases in the mixture are quite different for maximum sensitivity. See [Gas Charts on page E - 1](#).

## TEMPERATURE PROGRAMMED OXIDATION ANALYSIS

Temperature Programmed Oxidation (TPO) examines the extent to which a catalyst can be oxidized or was previously reduced.

Usually the sample is pretreated and the metal oxides are reduced to the base metal, typically with a gas mixture of hydrogen with either nitrogen or argon. Then the oxidizing gas, typically 2-5% oxygen with helium, is applied to the sample in pulses or, alternatively, as a steady stream.

The furnace heats the sample tube and sample according to the user-selected temperature program. The oxidation reaction occurs at a specific temperature. The analyzer measures the uptake of oxygen.



When using any mixture of gases for TPR or TPO analyses, ensure the thermal conductivities of the two gases in the mixture are quite different for maximum sensitivity. See [Gas Charts on page E - 1](#).

## ***PULSE CHEMISORPTION ANALYSIS***

A Pulse Chemisorption analysis determines the amount of gas irreversibly adsorbed and subsequently calculates active surface area, percent metal dispersion, and the average active particle size by applying measured doses of reactant gas to the sample.

The active gas interacts with each active site until all active sites are covered. Once the active sites have been completely covered, the discretely injected gas volumes elute from the sample tube unchanged. The amount chemisorbed is the difference between the total amount of active gas injected and the amount that eluted from the system. The quantity of each pulse of reactant gas is determined by the loop volume on an automatically controlled valve. A 0.5 cm<sup>3</sup> loop is provided with the analyzer and an optional 1cm<sup>3</sup> loop is available.

## ***ADDITIONAL USES OF THE TCD ANALYZER***

The analyzer may also be used for Temperature Programmed Reactions, Catalyst Pretreatment, and Isothermal Reactions. The tremendous flexibility of the analyzer allows the use of custom applications.

### **Temperature Programmed Reaction**

A Temperature Programmed Reaction monitors the products from the reaction between gases and a catalyst at a specified temperature. The analyzer can be programmed to raise the temperature of a catalyst bed at a constant ramping rate as the gases flow through the catalyst. At the optimal temperature, the gases react in the presence of the catalyst, creating products. The products of the reaction and the excess reactants can be diverted to a gas chromatograph or to a mass spectrometer to be analyzed.

### **Catalyst Pretreatment**

Catalyst Pretreatment usually consists of activating a catalyst prior to its use in a chemical reaction. For example, when a temperature programmed reaction is to be performed, the catalyst must be reduced under a flow of H<sub>2</sub> at a specific temperature. The reduction is necessary to create active sites, or reduced atoms of active metals, which are responsible for the activity of a catalyst.

### **Isothermal Reaction**

An Isothermal Reaction is similar to a temperature programmed reaction except that the catalyst is kept at a constant temperature (isothermal) to perform the catalytic reaction. Both the product of the reaction and the excess reactants can be diverted to a gas chromatograph or to a mass spectrometer to be analyzed.

## ***APPLICATIONS***

Catalytic processes that benefit from TPD/TPR analyses include:

- Polymerization Oxidation
- Hydrogenation Dehydrogenation
- Catalyst Cracking Hydrotreating
- Hydrocracking Alkylation Reforming
- Isomerization

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## 3 ABOUT SAMPLE FILES

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

A sample 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 software contains a default method. A method is a template for sample files that contains the parameters to be used for an analysis. When a new sample 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.

### SAMPLE FILES

**Options > Option Presentation > [Advanced / Basic / Restricted]**

**Options > Option Presentation > Show Degas Conditions**

**File > New Sample > [.SMP File]**

**File > Open > [.SMP File]**

See [Specify the Type of Analyzer on page 2 - 18](#)

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. See [About Parameter Files on page 4 - 1](#).

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. See [Option Presentation on page 2 - 11](#).

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. *Adsorptive Properties* are edited on the *Analysis Conditions* tab.



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

**Physical Adsorption**

**Chemical Adsorption or Dynamic Analysis**

**Advanced Option Presentation**

Physical Adsorption

Chemical Adsorption or Dynamic Analysis

## Basic Option Presentation

## Sample File Fields and Buttons Table

Field or Button	Description
<b>Active Metals</b> [button] 	Displays a list of active metals. See <a href="#">Active Metals for Chemical Adsorption on page 3 - 10</a> .
<b>Add Log Entry</b> [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.
<b>Comments</b> [text box]	Enter comments about the sample or analysis. Comments display in the report header.
<b>Mass</b> [group box]	<p>If mass = 1, the reported surface area equals the total surface area but it is always shown as m<sup>2</sup>/g. If the actual mass is entered, the surface area is reported as m<sup>2</sup>/g. Choose whether to enter mass manually or have the system automatically calculate mass. Enter a value for sample mass. Mass can be changed any time before, during, or after analysis.</p> <p><b>Enter.</b> Enables the <i>Sample Mass</i> field. Enter a value for the sample mass.</p> <p><b>Calculate.</b> Enables the <i>Empty tube</i> and <i>Sample + tube</i> fields. Enter the values necessary to calculate the sample mass. Equation used to calculate sample mass:</p> $Mass_{sample} = Mass_{sample+tube} - Mass_{tube}$ <p><b>Density.</b>  Value is used for the calculated free space method only.</p>

## Sample File Fields and Buttons Table (continued)

Field or Button	Description
	Use 0.000 for a blank analysis.
<b>Method</b> [ <i>drop-down box</i> ]	Select a method from the drop-down list. See <a href="#">Methods on page 2 - 14</a> .
<b>Operator</b> [ <i>text box</i> ]	Enter operator identification information. This field label may have been renamed or may not display if modified in <b>Options &gt; Default Methods</b> .
<b>Sample</b> [ <i>text box</i> ]	Enter a sample description.
<b>Sample Tube</b> [ <i>drop-down box</i> ] <b>P</b>	Select a sample tube file from the drop-down list, or click <b>Edit</b> to modify or create a new Sample Tube file. See <a href="#">Sample Tube for Gas Adsorption on page 4 - 34</a> .
<b>Submitter</b> [ <i>text box</i> ]	Enter submitter identification information. This text box may have been renamed or may not display if modified in <b>Options &gt; Default Methods</b> .
<b>Type of Data</b> [ <i>group box</i> ]	<p><b>Automatically collected.</b> Select if the type of data will be automatically collected by the system while an analysis is running.</p> <p><b>Manually entered.</b> Use to enter data manually that was collected from another source. If <i>Manually entered</i> is selected, the Isotherm Report becomes available in the <i>Basic/Advanced</i> drop-down list for pasting or importing data into the file.</p> <p>See <a href="#">Manually Enter Data on page 3 - 6</a>.</p>
<b>User Parameters</b> [ <i>group box</i> ] <b>P</b>	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 <i>Summary Report</i> . Some fields may not display (or may have a different field label) if modified in the method from which the sample file was created, either through <b>Options &gt; Default Method</b> or <b>File &gt; Open &gt; Method</b> .
	For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .

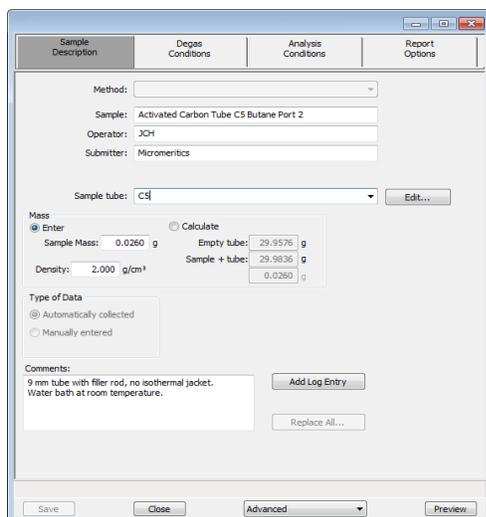
## OPEN A SAMPLE FILE

**File > Open > [.SMP File]**

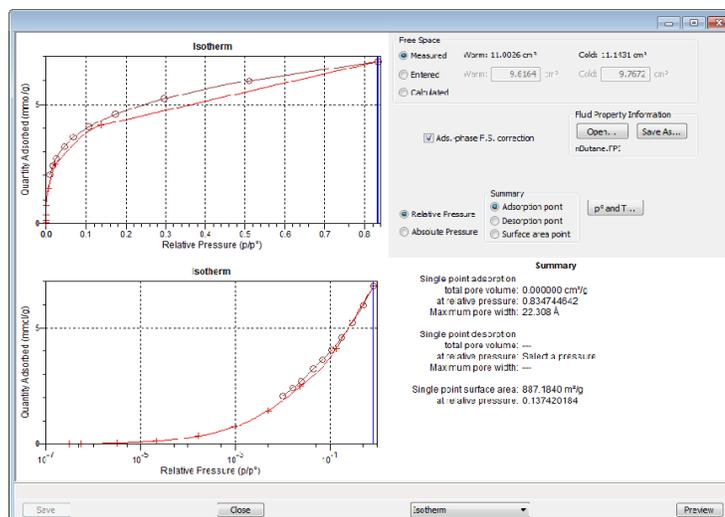


When working with an existing sample file, consider making a copy of the sample file to maintain the original configuration options.

File Type	File Status	Displays
Physical Adsorption or Chemical Adsorption	Preparing Prepared No Analysis	Tabbed file editor
	Complete Analyzing Entered	MicroActive report window



Example of tabbed file editor



Example of MicroActive report

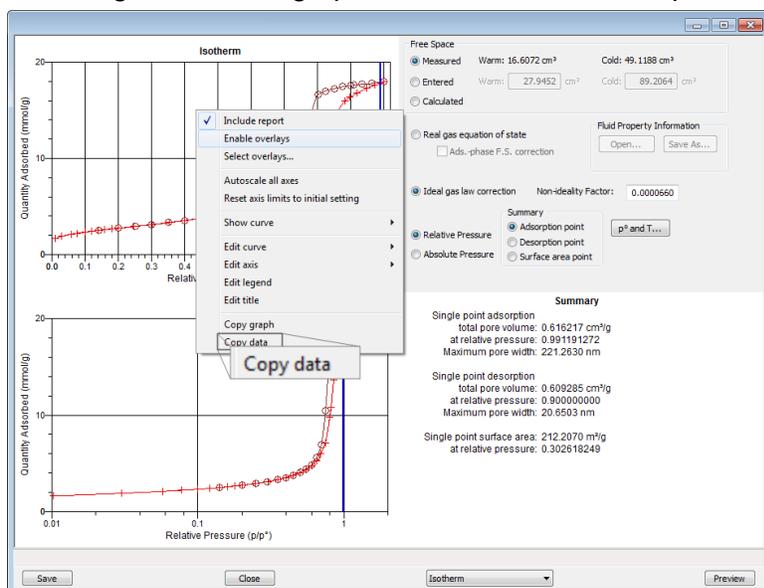
## MANUALLY ENTER DATA

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:

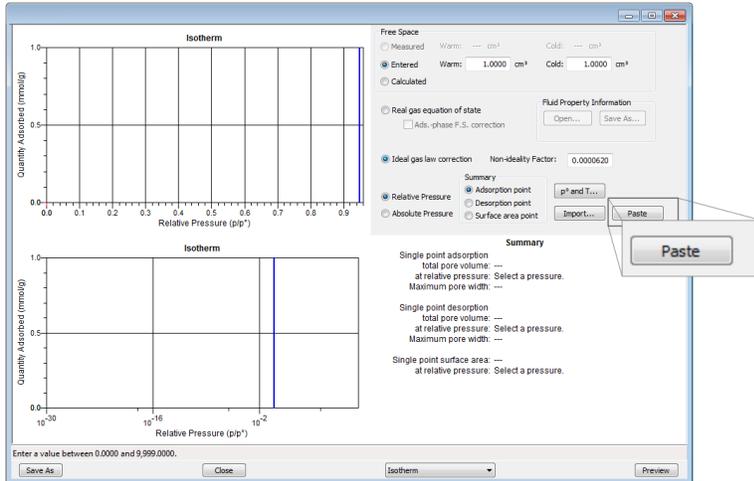
- Copy and paste onto the graph area of the interactive window
- Import data into the interactive window

## 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 the **Paste** button.

## 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:

### For Physical Adsorption or Chemical Adsorption:

- Relative Pressure
- Absolute Pressure (mmHg)
- Absolute Pressure (kPa)
- Absolute Pressure (mBar)
- Quantity Adsorbed (mmol/g)
- Quantity Adsorbed (cm<sup>3</sup>/g STP)
- Quantity Adsorbed (cm<sup>3</sup>/g STP)

### Sample Physical Adsorption ASCII Text File

Silica Alumina : Adsorption

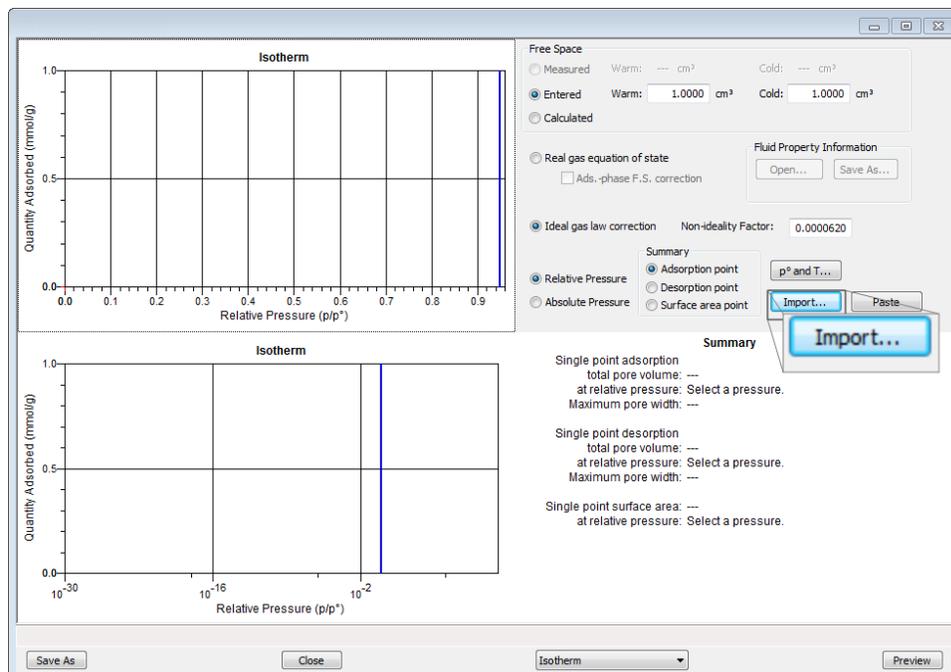
Relative Pressure	Quantity Adsorbed (cm <sup>3</sup> /g STP)
0.108629	50.6657
0.22288	60.7813
0.339909	71.3095
0.459512	84.4172
0.577447	102.672
0.654583	121.707
0.760074	179.096
0.855713	334.565
0.958511	394.675
0.996251	403.793

Silica Alumina : Desorption

Relative Pressure	Quantity Adsorbed (cm <sup>3</sup> /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.

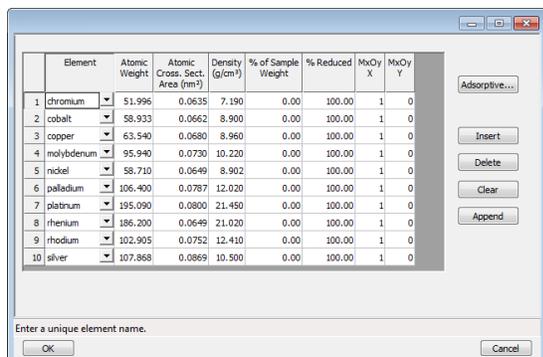
## ACTIVE METALS FOR CHEMICAL ADSORPTION

### Options > Active Metals Defaults

(or click [Active Metals](#) on the *Sample Description* tab when in Advanced presentation option)

See [Atomic Weights and Cross Sectional Areas on page A - 1](#)

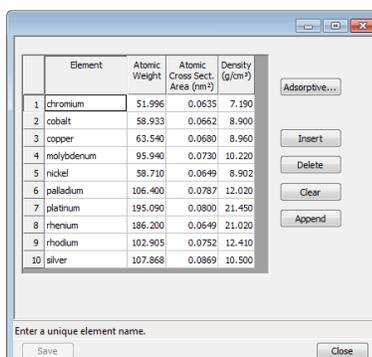
Up to 20 elements can be specified. At least one element must have a non-zero % of sample weight.



	Element	Atomic Weight	Atomic Cross. Sect. Area (nm <sup>2</sup> )	Density (g/cm <sup>3</sup> )	% of Sample Weight	% Reduced	MxOy X	MxOy Y
1	chromium	51.996	0.0635	7.190	0.00	100.00	1	0
2	cobalt	58.933	0.0662	8.900	0.00	100.00	1	0
3	copper	63.540	0.0680	8.960	0.00	100.00	1	0
4	molybdenum	95.940	0.0730	10.220	0.00	100.00	1	0
5	nickel	58.710	0.0649	8.902	0.00	100.00	1	0
6	palladium	106.400	0.0787	12.020	0.00	100.00	1	0
7	platinum	195.090	0.0800	21.450	0.00	100.00	1	0
8	rhenium	186.200	0.0649	21.020	0.00	100.00	1	0
9	rhodium	102.905	0.0752	12.410	0.00	100.00	1	0
10	silver	107.868	0.0869	10.500	0.00	100.00	1	0

Buttons: Adsorptive..., Insert, Delete, Clear, Append

Enter a unique element name.

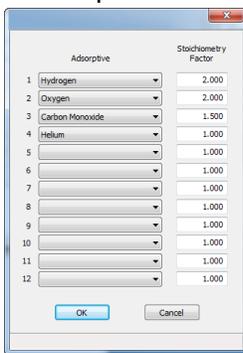


	Element	Atomic Weight	Atomic Cross. Sect. Area (nm <sup>2</sup> )	Density (g/cm <sup>3</sup> )
1	chromium	51.996	0.0635	7.190
2	cobalt	58.933	0.0662	8.900
3	copper	63.540	0.0680	8.960
4	molybdenum	95.940	0.0730	10.220
5	nickel	58.710	0.0649	8.902
6	palladium	106.400	0.0787	12.020
7	platinum	195.090	0.0800	21.450
8	rhenium	186.200	0.0649	21.020
9	rhodium	102.905	0.0752	12.410
10	silver	107.868	0.0869	10.500

Buttons: Adsorptive..., Insert, Delete, Clear, Append

Enter a unique element name.

### Active Metals Fields and Buttons Table

Field or Button	Description																										
<b>% of Sample Weight *</b>	Percentage, by weight, of each element contained in the sample.																										
<b>% Reduced *</b>	The percent of metal reduced during preparation.																										
<b>Adsorptive [button]</b>	Click to display and modify both the adsorptives and their associated stoichiometry factor. Stoichiometry factors for each gas are metal-specific. <div data-bbox="591 1257 834 1610" data-label="Form">  <table border="1"> <thead> <tr> <th>Adsorptive</th> <th>Stoichiometry Factor</th> </tr> </thead> <tbody> <tr><td>1 Hydrogen</td><td>2.000</td></tr> <tr><td>2 Oxygen</td><td>2.000</td></tr> <tr><td>3 Carbon Monoxide</td><td>1.500</td></tr> <tr><td>4 Helium</td><td>1.000</td></tr> <tr><td>5</td><td>1.000</td></tr> <tr><td>6</td><td>1.000</td></tr> <tr><td>7</td><td>1.000</td></tr> <tr><td>8</td><td>1.000</td></tr> <tr><td>9</td><td>1.000</td></tr> <tr><td>10</td><td>1.000</td></tr> <tr><td>11</td><td>1.000</td></tr> <tr><td>12</td><td>1.000</td></tr> </tbody> </table> <p>Buttons: OK, Cancel</p> </div>	Adsorptive	Stoichiometry Factor	1 Hydrogen	2.000	2 Oxygen	2.000	3 Carbon Monoxide	1.500	4 Helium	1.000	5	1.000	6	1.000	7	1.000	8	1.000	9	1.000	10	1.000	11	1.000	12	1.000
Adsorptive	Stoichiometry Factor																										
1 Hydrogen	2.000																										
2 Oxygen	2.000																										
3 Carbon Monoxide	1.500																										
4 Helium	1.000																										
5	1.000																										
6	1.000																										
7	1.000																										
8	1.000																										
9	1.000																										
10	1.000																										
11	1.000																										
12	1.000																										
	<b>Stoichiometry Factor.</b> The stoichiometry factor is defined as the moles of metal species covered per mole of adsorptive.																										

## Active Metals Fields and Buttons Table (continued)

Field or Button	Description
Atomic Cross Sect. Area (nm <sup>2</sup> )	Atomic cross-sectional area of the element.
Atomic Weight	Atomic weight of the element.
Density g/cm <sup>3</sup>	Density of the element.
Element [ <i>drop-down box</i> ]	Select or enter the active metal.
MxOy, X * MxOy, Y *	X and Y values specify the empirical formula for metal and oxygen, respectively, in a metal oxide.
* Options are shown only when using the <b>Active Metals</b> button on the <i>Sample Description</i> tab.	
 For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4.</a>	

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## 4 ABOUT PARAMETER FILES

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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.

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

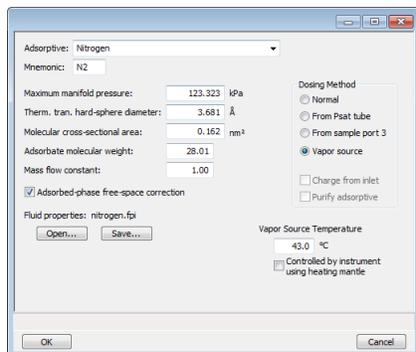
File Type	File Extension
Adsorptive Properties	.ADP
Analysis Conditions	.ANC
Degas Conditions <b>P</b>	.DEG
Method	.MTH
Report Options	.RPO
Sample Tube <b>P</b>	.STB

## ADSORPTIVE PROPERTIES

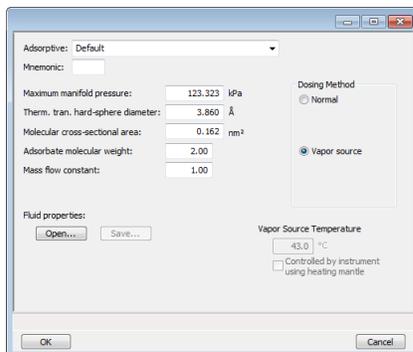
### File > Open > [.ADP File]

(or, for physical adsorption and chemical adsorption, click **Edit** next to the *Adsorptive* selection on the *Analysis Conditions* tab when in *Advanced* option presentation. For a dynamic analysis, there is no *Adsorptive Properties* tab; use **File > Open > [.ADP file]**.)

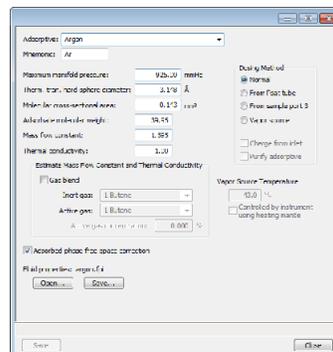
Adsorptive properties provide the adsorptive (analysis gas) characteristics for the analysis.



Physical Adsorption



Chemical Adsorption



Dynamic Analysis

### Adsorptive Properties Fields and Buttons Table

Field or Button	Description
<b>Adsorbate molecular weight</b> [ <i>text box</i> ]	The molecular mass is used for the weight % column of the isotherm tabular report and for the pressure composition isotherm plot.
<b>Adsorbed phase free-space correction</b> [ <i>check box</i> ]	Adsorbed molecules occupy volume in the sample tube, reducing the cold free space. Select <i>Adsorbed-phase free-space correction</i> to adjust the reported quantity adsorbed to correct for this effect. This option is appropriate for all sample analyses that use the real gas equation of state
<b>Adsorptive</b> [ <i>drop-down box</i> ]	Name of the adsorptive gas whose properties are being defined.
<b>Dosing Method</b> [ <i>group box</i> ]	<p><b>Normal.</b> Dose from a pressurized tank of gas attached to a gas inlet port.</p> <p><b>From Psat tube.</b> <b>P</b> Select if the Psat tube is to be filled with condensed adsorptive and dosed from the Psat tube. Select this option if using Krypton.</p> <p><b>From sample port 3.</b> <b>P</b> Select if the tube attached to sample port 3 is to be filled with condensed adsorptive and dosed from port 3.</p>

## Adsorptive Properties Fields and Buttons Table (continued)

Field or Button	Description
	<p><b>Vapor source.</b> <b>P SC</b> Select if a container of condensed vapor is to be attached to the Psat port in place of the Psat tube and is dosed from the Psat port.</p> <p><b>Charge from inlet.</b> <b>P</b> Use to have the tube automatically charged with condensate from a gas inlet port after the dewar is raised.</p> <p><b>Purify adsorptive.</b> <b>P</b> Use to have the condensate in the tube purified after charging by evacuating the gas over the condensate. If <i>Charge from inlet</i> is selected, select <i>Purify adsorptive</i> to have noncondensing contaminants automatically removed from the dosing tube prior to analysis. After the adsorptive has condensed in the selected Psat tube or port 3, the remaining gas in the tube will be evacuated to remove noncondensing contaminants. A small amount of the purified adsorptive condensate will then return to gas phase to restore equilibrium pressure in the tube.</p>
<b>Fluid properties</b> [ <i>button</i> ]	Use to import parameters from a Fluid Properties (.FPI) file. Changing fluid properties should only be necessary if an adsorptive is to be used for which no adsorptive properties are provided. Contact Micromeritics Scientific Services if new fluid properties are required. See <a href="#">Contact Us on page ii</a> .
<b>Gas Blend</b> [ <i>checkbox</i> ] <b>DA</b>	Select to specify a preblended mixture of a chemically inert gas and a chemically active gas. The <i>Mass Flow Constant</i> and <i>Thermal Conductivity</i> for the blend are automatically calculated from those for the two gases in the blend.
<b>Mass flow constant</b> [ <i>text box</i> ] <b>C</b>	Scaling factor for the Mass Flow Controller measured flow rate. Applicable only for the gas used in the flow prep tasks. The default is preset for gases provided with the application.
<b>Maximum manifold pressure</b> [ <i>text box</i> ]	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.
<b>Mnemonic</b> [ <i>text box</i> ]	Enter the mnemonic name for the adsorptive. If this gas is connected to a gas inlet port, this mnemonic must be entered in the <i>Unit Configuration Gas Selection</i> for the inlet port.. See <a href="#">Specify Gas Ports on page 2 - 15</a> .

**Adsorptive Properties Fields and Buttons Table (continued)**

Field or Button	Description
<b>Molecular cross-sectional area</b> [ <i>text box</i> ]	The area that a single adsorbed molecule occupies on the surface of the sample. It is used in surface area calculations.
<b>Thermal conductivity</b> 	See <a href="#">Gas Charts on page E - 1</a> .
<b>Therm. tran. hard-sphere diameter</b> [ <i>text box</i> ]	An estimate of molecular size used in calculating the thermal transpiration correction.
<b>Vapor Source Temperature</b> [ <i>group box</i> ]	Select if the vapor source temperature is to be controlled by the analyzer. This field is enabled only if <i>Vapor Source</i> is selected.
 <b>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</b>	

## ANALYSIS CONDITIONS FOR PHYSICAL ADSORPTION AND CHEMICAL ADSORPTION

**File > Open > [.ANC File]**

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

Analysis conditions specify the data used to guide an analysis.

See [Chemical Adsorption Tasks on page 4 - 13](#)

See [Evacuation Rules for Chemical Adsorption on page 4 - 16](#)

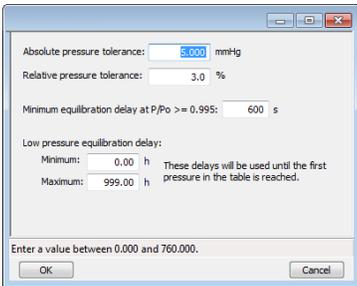
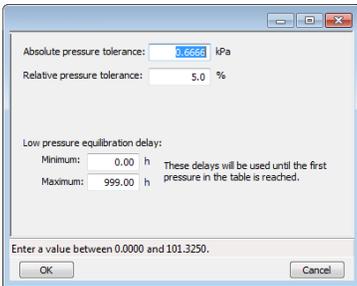
See [Analysis Conditions for Dynamic Analysis on page 4 - 17](#)

**Physical Adsorption**

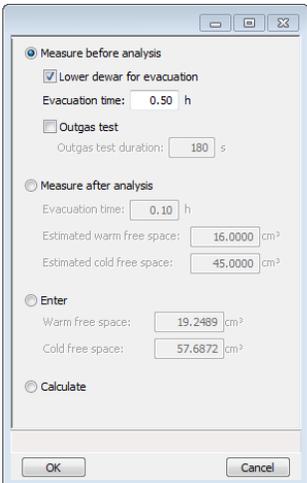
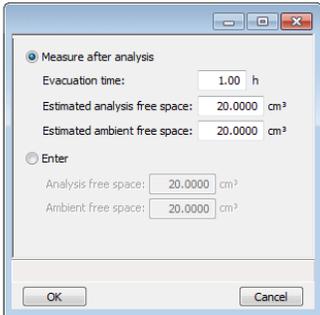
**Chemical Adsorption**

The Pressures tables have a *Starting Pressure* and an *Ending Pressure* column. Each table row represents a pressure interval. The *Starting Pressure* column is for information only and cannot be edited.

## Analysis Conditions Fields and Buttons Table

Field or Button	Description
<b>Absolute pressure dosing</b> [selection] 	Specifies pressure targets in mmHg, mbar, or kPa instead of relative pressure. If this option is selected, the <i>Relative Pressure</i> labels and entries change to <i>Absolute Pressure</i> in the selected pressure units.
<b>Adsorptive</b> [drop-down box]	Select an <i>Adsorptive Properties</i> file from the of defined gases to be used for analysis. After selection, click <b>Edit</b> to modify adsorptive properties.
<b>Analysis Conditions</b> [drop-down box]	Use to browse for an <i>Analysis Conditions</i> file that contains analysis condition parameters to be used in the analysis.
<b>Dosing</b> [button]	Options for dosing tolerance, low pressure dosing, and dosing near saturation pressure. <div style="display: flex; justify-content: space-around; align-items: flex-start; margin-top: 10px;"> <div style="text-align: center;">  <p><b>Physical Adsorption</b></p> </div> <div style="text-align: center;">  <p><b>Chemical Adsorption</b></p> </div> </div> <p><b>Absolute / Relative pressure tolerance.</b> Values used to determine how close the actual pressure must be to each target pressure from the pressure table. At lower pressures, the relative tolerance value is lower. At higher pressures, the absolute tolerance value is lower. For example:</p> <p><b>Experiment 1.</b> There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 40 mmHg; 5% of 40 mmHg is 2 mmHg. Since 2 mmHg (relative tolerance) is lower than 5 mmHg (absolute tolerance), 2 mmHg is used. Therefore a minimum pressure of 38 mmHg (40 - 2) must be attained to collect data for a target pressure of 40 mmHg.</p> <p><b>Experiment 2.</b> There might be an absolute tolerance of 5 mmHg, a relative tolerance of 5%, and a target pressure of 200 mmHg; 5% of 200 mmHg is 10 mmHg. Since 5 mmHg (absolute tolerance) is lower than 10 mmHg (relative tolerance), 5 mmHg is used. Therefore a minimum pressure of 195 mmHg (200 - 5) must be attained to collect data for a target pressure of 200 mmHg.</p> <p>Normally, surface area measurement points are widely spaced and the resulting measurement is not very sensitive to the precise</p>

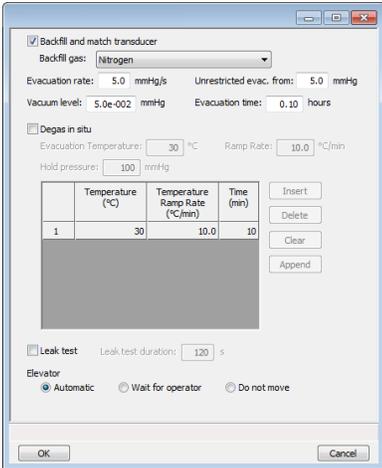
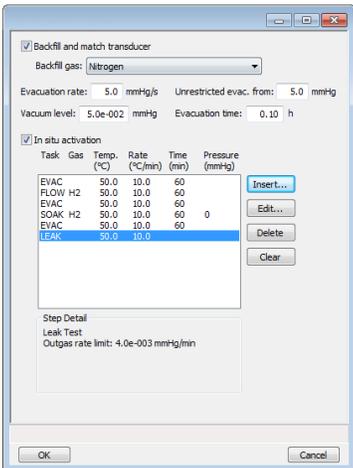
## Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	<p>location of points so wider tolerances may be used. Unnecessarily tight tolerances lengthen the analysis.</p> <p><b>Minimum equilibration delay at <math>p/p_0 \geq 0.995</math>.</b> <b>P</b> The minimum number of seconds required before equilibration can occur for a relative pressure greater than or equal to 0.995. This field is not available if <i>Absolute pressure dosing</i> is selected on the <i>Analysis Conditions</i> tab.</p> <p><b>Low pressure equilibration delay.</b> Delays to be used until the first pressure in the table is reached.</p>
<b>Free Space [button]</b>	<p>How the free space is to be measured.</p> <div style="display: flex; justify-content: space-around;"> <div style="text-align: center;">  <p><b>Physical Adsorption</b></p> </div> <div style="text-align: center;">  <p><b>Chemical Adsorption</b></p> </div> </div> <p><b>Calculate.</b> <b>P</b> Use to have the free space measurement calculated using the sample and tube parameters.</p> <p><b>Enter.</b> Measures free space after analysis ends. Enter the estimated warm free space measurement and the estimated cold free space measurement.</p> <p><b>Measure after analysis.</b> Measures free space after analysis ends. Enter the estimated warm free space measurement and the estimated cold free space measurement.</p> <p><b>Measure before analysis.</b> <b>P</b> Select if the free space is to be measured before the analysis begins.</p>

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	<ul style="list-style-type: none"> <li>• <b>Lower dewar for evacuation.</b> If the dewar is to be lowered for evacuation, select this option and enter the length of time for evacuation after the free-space measurement in the <i>Evacuation time</i> field. If using a cryostat, the operator must manually move the cryostat assembly when prompted.</li> <li>• <b>Outgas test.</b> Checks for system leaks or sample outgassing. After free space is measured, the dewar is lowered and the sample evacuated for the specified amount of time. The leak test is performed after evacuation. If a leak is found, the leak test repeats nine times, with 30 minutes evacuation between tests. If the 10th leak check fails, the 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.</li> </ul>
<p><b>p° and Temperature Options [button] P</b></p>	<p>How the saturation pressure (<math>P_0</math>) is to be measured or calculated and the analysis bath temperature.</p> <p><b>Analysis Temperature Options.</b> Select an option to enter analysis temperature manually, or choose to have it automatically calculated from <math>p^\circ</math> or <math>P_0</math>.</p> <p><b><math>p^\circ</math> Options.</b> Select one option indicating how <math>P_0</math> is to be measured or calculated. If using a cryostat, <i>Calculate <math>p_0</math> from the analysis temperature</i> should be selected.</p> <p><b>Psat Gas.</b> If choosing to measure the <math>P_0</math> for each isotherm point using a gas other than the adsorptive, select the <math>P_0</math> gas from the drop-down list,</p>

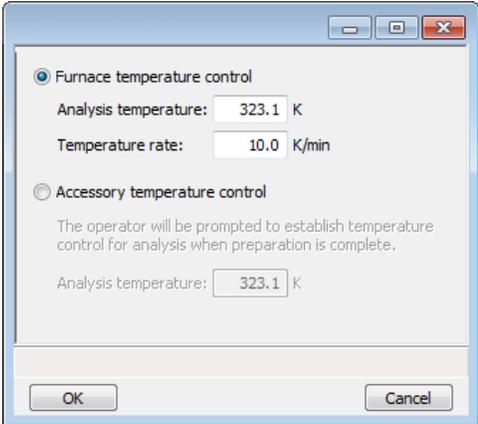
## Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	then click <b>Edit</b> to modify the $P_0$ adsorptive properties. Refer to the <i>Adsorptive</i> drop-down list earlier in this table for details on editing this window.
<b>Preparation [button]</b>	<p>Evacuation rate / time level, leak test and time values, elevator prompts, and in situ degassing or activation.</p> <hr/> <div style="border: 1px solid green; padding: 5px; display: flex; align-items: center;"> <p>To insert chemical adsorption tasks, see <a href="#">Chemical Adsorption Tasks on page 4 - 13</a>.</p> </div> <hr/> <div style="display: flex; justify-content: space-around;"> <div style="text-align: center;">  <p><b>Physical Adsorption</b></p> </div> <div style="text-align: center;">  <p><b>Chemical Adsorption</b></p> </div> </div> <p><b>Backfill and match transducer.</b> Backfills the sample tube to 760 mmHg at the beginning of the analysis and to recalibrate the sample port pressure transducer scale to match the manifold pressure transducer. Select the backfill gas to be used.</p> <p><b>Degas in situ.</b>  Degases the sample on the analysis port prior to analysis.</p> <ul style="list-style-type: none"> <li>• <b>Evacuation Temperature.</b> Temperature of the sample during evacuation.</li> <li>• <b>Hold pressure.</b> Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the <i>Hold pressure</i>. This feature prevents damage to the sample structure due to 'steaming,' as well as sample elutriation due to excessive escaping gas velocity.</li> </ul>

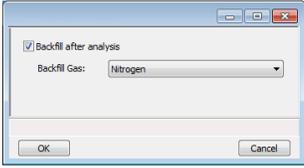
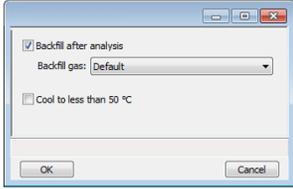
## Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	<ul style="list-style-type: none"> <li>• <b>Ramp Rate.</b> Rate at which the temperature is to change when advancing to the target temperature.</li> </ul> <p><b>Elevator.</b> <b>P</b> Select the appropriate elevator control option.</p> <ul style="list-style-type: none"> <li>• <b>Automatic.</b> The elevator is raised and lowered automatically.</li> <li>• <b>Wait for operator.</b> Prompts the operator to set the elevator or analysis bath to the preferred height. When the prompt is acknowledged, the analysis will continue. This option should be used if the analysis bath must be placed manually in the preferred position, or the elevator must be raised to a height other than the standard analysis height.</li> <li>• <b>Do not move.</b> Use to have the analysis proceed without pausing or moving the elevator. This option should be used when the analysis bath is already in position and should not be moved during analysis. If <b>Unit [n] &gt; Enable CryoStat</b> is enabled, the elevator option is automatically set to <i>Do not move</i>.</li> </ul> <p><b>Evacuation rate.</b> The rate for restricted evacuation.</p> <p><b>Evacuation time.</b> The length of time for preliminary evacuation. If this field is blank, the time entered in the <i>Pre-analysis evacuation</i> field on the <i>Analysis Conditions</i> window is used.</p> <p><b>In situ activation.</b> <b>SC</b> When selected, preparation steps will be done. If not selected, the task table is disabled and analysis starts after the preliminary evacuation.</p> <p><b>Leak Test.</b> <b>P</b> 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.</p> <p><b>Leak test duration.</b> <b>P</b> Enter the duration of the leak test.</p> <p><b>Vacuum level.</b> The pressure for unrestricted evacuation.</p> <p><b>Unrestricted evac. from.</b> The pressure at which unrestricted evacuation is to begin.</p>

## Analysis Conditions Fields and Buttons Table (continued)

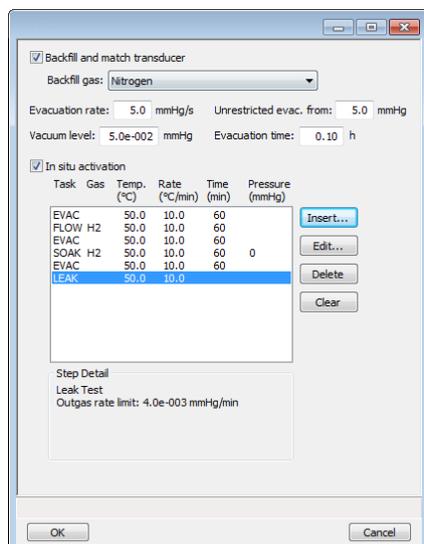
Field or Button	Description
<b>Pre-analysis evacuation time</b> [text box]	Evacuation is required prior to analysis. The default setting is 30 minutes.  <b>Preparation.</b> Use to set the evacuation rate, unrestricted pressure, and setpoint.  <b>Temperature.</b> Use to set the temperature and ramp rate.
<b>Repeat analysis</b> [selection]	Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the <i>Hold pressure</i> . This feature prevents damage to the sample structure due to 'steaming,' as well as sample elutriation due to excessive escaping gas velocity.
<b>Set external trigger during data collection</b>	Select to set the external trigger signal for the Mass Spectrometer (if connected). Chemical adsorption option is required.
<b>Temperature</b> [button] 	Provides access to furnace and accessory temperature control.    <b>Furnace temperature control.</b> Enter the analysis temperature rate.  <b>Accessory temperature control.</b> <i>For user supplied temperature control only.</i> Enter the intended analysis temperature. The operator will be prompted to establish the analysis temperature before analysis begins.

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
<b>Termination [button]</b>	<p>Select the backfill gas if backfill is to be done after the analysis.</p> <p>If using a cryostat, deselect <i>Backfill after analysis</i>.</p> <div style="display: flex; justify-content: space-around; align-items: flex-start;"> <div style="text-align: center;">  <p><b>Physical Adsorption</b></p> </div> <div style="text-align: center;">  <p><b>Chemical Adsorption</b></p> </div> </div> <p><b>Cool to less than 50 °C.</b>  Select to enable the cool down option.</p>
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

## CHEMICAL ADSORPTION TASKS

**File > Open > [.SMP File] > Analysis Conditions tab > Preparation > Insert**



To ensure safe operation and reliable results, an evacuation task should be included:

- between tasks using different gases
- preceding a leak test

An evacuation will be performed at the analysis temperature for the Pre-analysis Evacuation Time after the last task and before analysis.

### Chemical Adsorption Tasks Table

Task	Description
Evacuation [ <i>selection</i> ]	<p><b>Evacuate for ___ below ____</b> The minutes and pressure for preliminary evacuation.</p> <p><b>Evacuation rate.</b> The rate for restricted evacuation.</p>

Chemical Adsorption Tasks Table (continued)

Task	Description
	<p><b>Temperature.</b> The temperature to reach during evacuation.</p> <p><b>Temperature rate.</b> Enter the analysis temperature rate.</p> <p><b>Unrestricted evac. pressure.</b> The pressure at which unrestricted evacuation is to begin.</p>
<p><b>Flow [selection]</b></p>	<div data-bbox="558 508 915 869" style="border: 1px solid gray; padding: 5px; margin-bottom: 10px;"> </div> <p><b>Flow Rate.</b> The rate at which gas is to flow.</p> <p><b>Gas.</b> Gas used for the flow task.</p> <p><b>Set external trigger.</b> If selected, the contact closure used to trigger an external Mass Spectrometer or Calorimeter will be activated during the temperature ramp.</p> <p><b>Temperature.</b> The temperature at which the gas will flow for the specified time.</p> <p><b>Temperature rate.</b> Enter the analysis temperature rate.</p> <p><b>Time.</b> The duration of time the sample should remain at the specified temperature.</p>
<p><b>Leak Test [selection]</b></p>	<div data-bbox="558 1400 932 1661" style="border: 1px solid gray; padding: 5px; margin-bottom: 10px;"> </div> <p><b>Outgas rate limit.</b> If a measured leak or outgas rate exceeds the entered value, the test will be reported as failed. Analysis will not be canceled.</p>

## Chemical Adsorption Tasks Table (continued)

Task	Description
	<p><b>Temperature.</b> The target temperature for the leak test.</p> <p><b>Temperature rate.</b> Enter the analysis temperature rate.</p>
<p><b>Soak</b> [<i>selection</i>]</p>	<div data-bbox="558 407 911 709" style="border: 1px solid gray; padding: 5px; margin-bottom: 10px;"> <p>Gas: Hydrogen</p> <p>Minimum pressure: 0.0 kPa</p> <p>Time: 60 min</p> <p>Temperature: 323.1 K</p> <p>Temperature rate: 10.0 K/min</p> <p>OK Cancel</p> </div> <p><b>Gas.</b> Gas used during the soak task.</p> <p><b>Minimum pressure.</b> The minimum pressure to be maintained over the sample during the soak.</p> <p><b>Temperature.</b> The temperature at which sample is to be soaked.</p> <p><b>Temperature rate.</b> Enter the analysis temperature rate.</p> <p><b>Time.</b> The duration of time the sample is to soak at the specified temperature.</p>

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## ***EVACUATION RULES FOR CHEMICAL ADSORPTION***

Evacuation parameters apply to all four stages of evacuation with the exception of evacuation time. Evacuation time is set using the fields specified in the following evacuation rules.

When an analysis starts, evacuation begins:

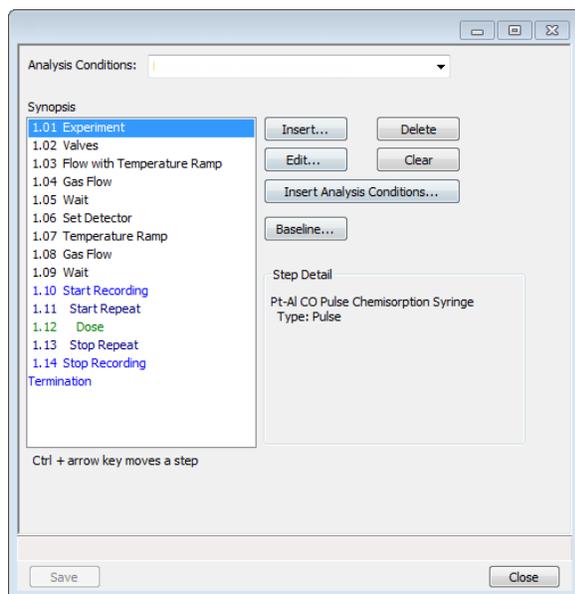
1. at ambient temperature before the first preparation step. This step uses the *Evacuation time* field on the *Preparation Options* window.
2. at analysis temperature after preparation and prior to the start of the analysis stage. This step uses the *Pre-analysis evacuation time* field on the *Analysis Conditions* window.
3. at analysis temperature before the repeat analysis if *Repeat analysis* is selected on the *Analysis Conditions* window. This step uses the *Repeat Analysis / Evacuation time* field.
4. at analysis temperature before the free space measurement. This step uses the *Evacuation time* field on the *Free Space Options* window. Measure after analysis must be selected to enable the *Evacuation time* field.

## ANALYSIS CONDITIONS FOR DYNAMIC ANALYSIS

### File > Open > [.ANC File]

Analysis conditions specify the data used to guide an analysis.

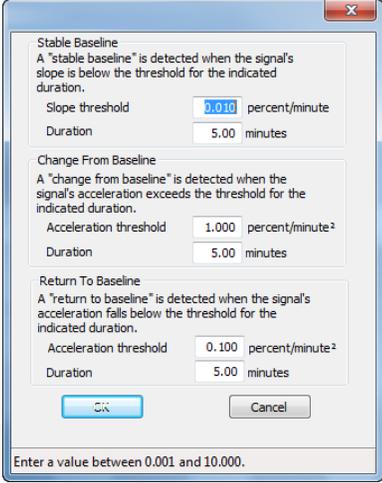
See [Analysis Conditions for Physical Adsorption and Chemical Adsorption on page 4 - 5](#)



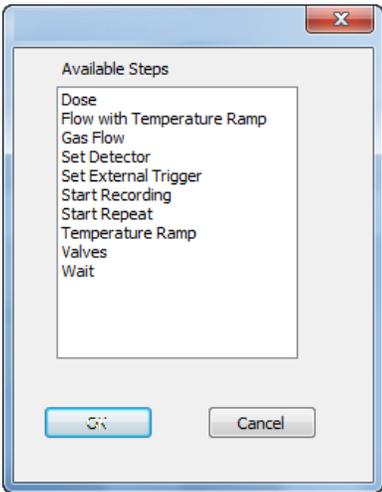
### Analysis Conditions Fields and Buttons Table

Field or Button	Description
<b>Analysis Conditions</b> [drop-down box]	Use to browse for an <i>Analysis Conditions</i> file that contains analysis condition parameters to be used in the analysis.

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
<p><b>Baseline</b> [<i>button</i>]</p>	<p>Specifies baseline settings if a <i>Wait</i> step depends upon the <i>Baseline</i>.</p>  <p>Establishes the <i>Slope</i> or <i>Acceleration</i> threshold and <i>Duration</i> for determining what constitutes a <i>Stable Baseline</i>, a <i>Change From Baseline</i>, and a <i>Return To Baseline</i>. These values control whether a particular change in the signal is significant to the current experiment — such as defining a stable baseline.</p> <p>Some <i>Wait</i> steps are contingent upon the values selected — such as if the experiment contains a <i>Wait until Baseline is stable</i> step, the signal is compared to these values to determine if a stable baseline has been established. Lower slope/acceleration values and longer durations create a more rigorous definition of these factors than higher values and shorter durations.</p> <p><b>Stable Baseline.</b> Detected when the signal slope is below the threshold for the indicated duration.</p> <p><b>Change from Baseline.</b> Detected when the signal acceleration exceeds the threshold for the indicated duration.</p> <p><b>Return to Baseline.</b> Detected when the signal acceleration falls below the threshold for the indicated duration.</p>

## Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
<b>Insert</b> [ <i>button</i> ]	<p>Insert a new step into the <i>Synopsis</i>. Steps can be inserted after the analysis has started by suspending the analysis.</p> <p>See <a href="#">Insert Experiment Step on page 4 - 21</a>.</p>  <p>To ensure safe operation and reliable results, a chemically inert gas flow should be inserted between flows of two chemically reactive gases such as hydrogen and oxygen.</p>
<b>Insert Analysis Conditions</b> [ <i>button</i> ]	<p>Click to load an entire list of steps, baseline parameters, and peaks parameters from the selected dynamic analysis conditions file.</p>
<b>Step Detail</b> [ <i>group box</i> ]	<p>Displays summary information about the highlighted step in the <i>Synopsis</i> box.</p>
<b>Synopsis</b> [ <i>group box</i> ]	<p>Contains a list of inserted experiment steps for the analysis. See <a href="#">Synopsis on the next page</a>.</p> <p>To edit steps that have not been started, the analysis must be suspended.</p> <ul style="list-style-type: none"> <li>• Upcoming steps - display as black or blue</li> <li>• Current step - step is highlighted and displays as light blue</li> <li>• Completed steps - display as light green</li> </ul>

Analysis Conditions Fields and Buttons Table (continued)

Field or Button	Description
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

**SYNOPSIS**

An analysis set is created by inserting up to 99 experiments in the sample file synopsis.

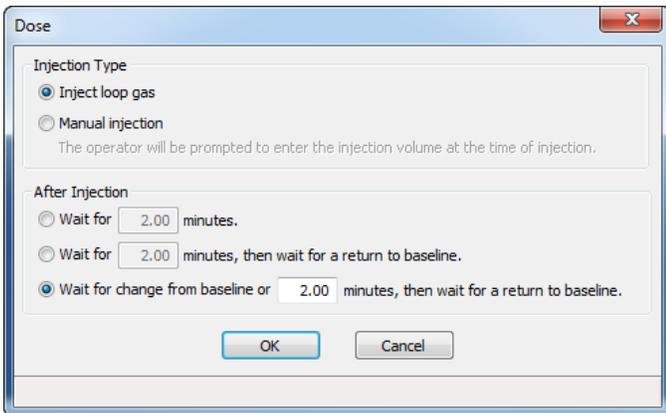
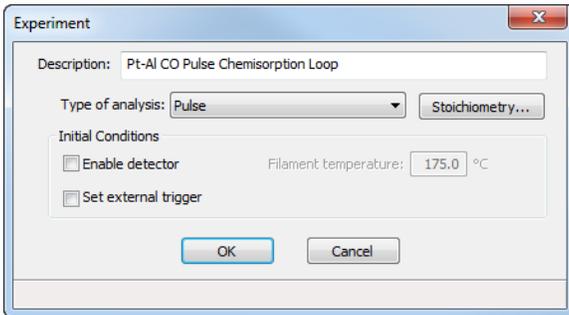
- Note that Loop Calibration, Loop Gas, Inject loop gas, and Loop Valve selections are only applicable to instruments that have the Loop option. Do not select these for analyses to be performed on an instrument without the Loop option.
- Data from one experiment are not available for editing until the next experiment in the analysis has begun recording.

## INSERT EXPERIMENT STEP

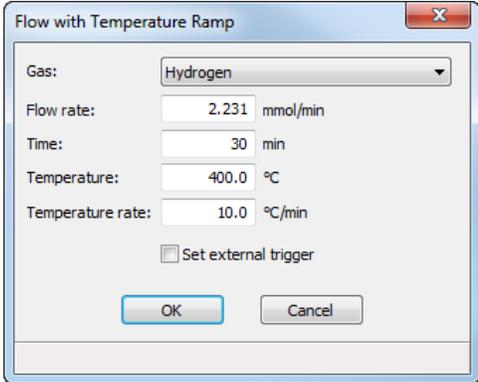
Experiments can be customized to control the analysis reaction. When an experiment is inserted, the initial conditions are specified first, then the individual steps.

On the *Analysis Conditions* tab, click **Insert** to insert an experiment. Select the *Experiment* step in the *Synopsis* box, then click **Edit** to modify settings.

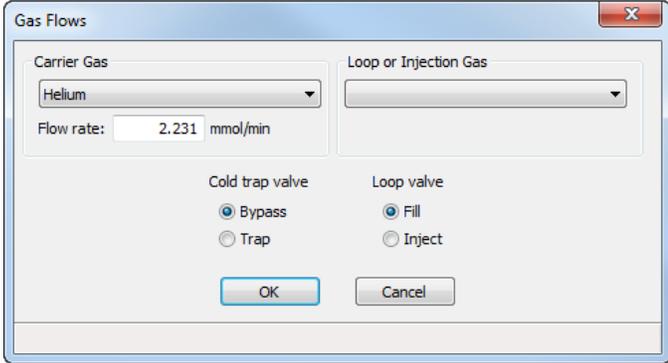
### Experiment Fields and Buttons Table

Field or Button	Description
<b>Dose</b>	 <p><b>Injection Type:</b></p> <ul style="list-style-type: none"> <li>• <b>Injection loop gas.</b> Automatically injects the contents of the loop into the path that leads to the sample. The contents of the loop are pushed out of the loop by the carrier gas.</li> <li>• <b>Manual injection.</b> Prompts the user to inject a dose of gas into the septum using a syringe.</li> </ul> <p><b>After Injection.</b> Specify the conditions for completion of this step.</p>
<b>Experiment</b>	 <p><b>Description.</b> Description of the experiment.</p>

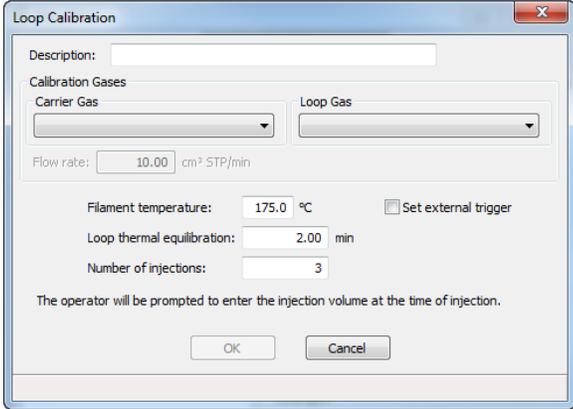
## Experiment Fields and Buttons Table (continued)

Field or Button	Description
	<p><b>Enable Detector.</b> Enables the <i>Filament temperature</i> field and enables the thermal conductivity detector (TCD).</p> <p><b>Filament temperature.</b> Set the filament temperature from 20 °C to 250 °C. A temperature of 175 °C is commonly used.</p> <p><b>Set external trigger.</b> If selected, the contact closure used to trigger an external mass spectrometer will be activated at the beginning of the experiment.</p> <p><b>Type of analysis.</b> See <a href="#">Analysis Types for TCD Analyzers on page 2 - 32</a>. If <i>Pulse</i> is selected, the <b>Active Metals</b> button is enabled. When modifying the <i>Active Metals</i> table during the insertion of a <i>Pulse</i> experiment, changes to the <i>Active Metals</i> table on the <i>Sample Description</i> tab are not affected. This is useful when sequencing multiple experiments and Stoichiometry is different from one experiment to the next.</p>
<b>Flow with Temperature Ramp</b>	 <p><b>Flow Rate.</b> The rate at which gas is to flow.</p> <p><b>Gas.</b> Gas used for the flow task.</p> <p><b>Set external trigger.</b> If selected, the contact closure used to trigger an external mass spectrometer will be activated at the beginning of the experiment.</p> <p><b>Temperature.</b> The temperature at which the gas will flow for the specified time.</p> <p><b>Temperature rate.</b> Enter the analysis temperature rate.</p> <p><b>Time.</b> The duration of time the sample should remain at the specified temperature.</p>

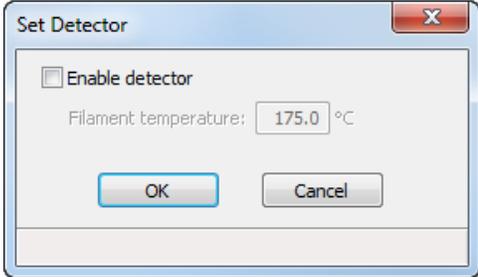
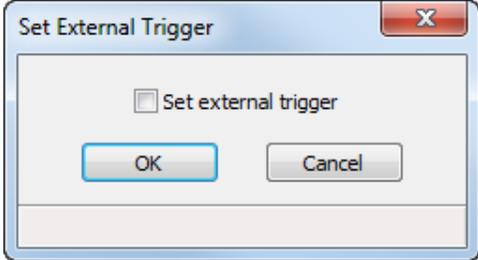
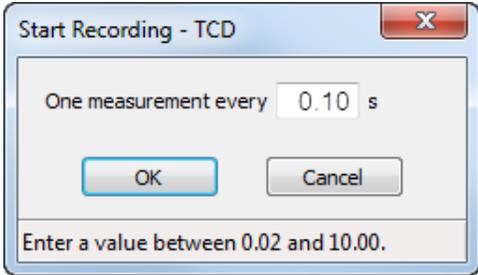
## Experiment Fields and Buttons Table (continued)

Field or Button	Description
<b>Gas Flow</b>	 <p><b>Carrier Gas / Loop or Injection Gas.</b> Select the gas for each set of inlet ports. The gases listed are those with defined Adsorptive Properties. If manual injections are to be programmed, the gas to be injected must be selected as <i>Loop or Injection Gas</i>. If loop injections are to be programmed, this gas must be connected to a loop gas inlet and assigned in <b>Unit [n] &gt; Unit Configuration</b>. See <a href="#">Configure the Analyzer on page 2 - 15</a>.</p> <p>When an analysis is started, the application verifies that the selected gases are connected to the appropriate ports. If there is a discrepancy between a gas selected for the current sample file and the gas indicated in the <i>Unit Configuration</i> window, an error message is displayed when the analysis is started.</p> <p><b>Cold trap valve.</b> Select the status of the cold trap valve.</p> <p><b>Flow rate.</b> The rate at which gas is to flow.</p> <p><b>Loop valve.</b> Select the status of the loop valve.</p>
<b>Loop Calibration</b>	<p>Use to calibrate the gas injection loop.</p> <p>This experiment verifies the exact volume of the loop for use in calculations on analyses in which the loop is used. Sample analysis data yield signal vs. time or temperature data and peak areas. After the analysis, select the <i>Loop Calibration</i> report and enter the <i>Loop volume</i> from the report on the <i>Unit Configuration</i> window. Entering the calibrated <i>Loop volume</i> on the <i>Unit Configuration</i> window makes it possible for the application to convert sample data to volume values. See <i>Loop volume</i> located in <a href="#">Specify Gas Ports on page 2 - 15</a>.</p>

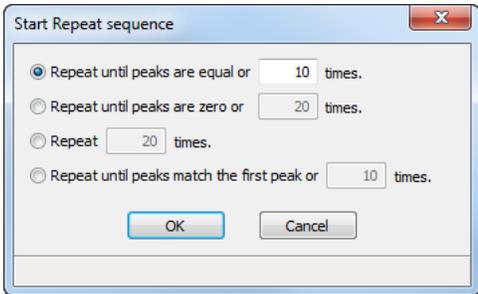
Experiment Fields and Buttons Table (continued)

Field or Button	Description
	 <p><b>Description.</b> Description of the experiment.</p> <p><b>Calibration Gases.</b> Select the carrier and loop gases.</p> <p><b>Filament temperature.</b> Set the filament temperature from 20 °C to 250 °C. A temperature of 175 °C is commonly used.</p> <p><b>Flow rate.</b> The rate at which gas is to flow.</p> <p><b>Loop thermal equilibration.</b> Specify the duration of the equilibration delay. This delay allows extra time for the instrument to stabilize thermally after the baseline has fully stabilized.</p> <p><b>Number of injections.</b> Indicate the number of gas injections are made into the gas stream.</p> <p><b>Set external trigger.</b> If selected, the contact closure used to trigger an external mass spectrometer will be activated.</p>

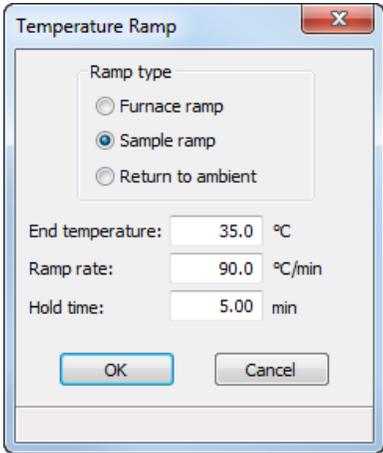
## Experiment Fields and Buttons Table (continued)

Field or Button	Description
<b>Set Detector</b>	 <p><b>Enable detector.</b> Enables the <i>Filament temperature</i> field and enables the thermal conductivity detector (TCD).</p> <p><b>Filament temperature.</b> Set the filament temperature from 20 °C to 250 °C. A temperature of 175 °C is commonly used.</p>
<b>Set External Trigger</b>	 <p><b>Set external trigger.</b> If selected, the contact closure used to trigger an external Mass Spectrometer or Calorimeter will be activated during the temperature ramp.</p>
<b>Start Recording</b>	<p>Specifies how frequently the signal reading is recorded. A <i>Stop Recording</i> step is inserted in the steps automatically when a <i>Start Recording</i> step is inserted. Multiple steps can be inserted between the <i>Start Recording</i> and <i>Stop Recording</i> steps.</p>  <p><b>One measurement every ____ seconds.</b> Specify the frequency of measurements.</p>

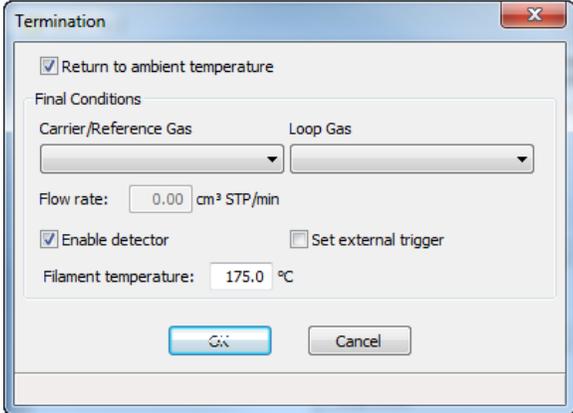
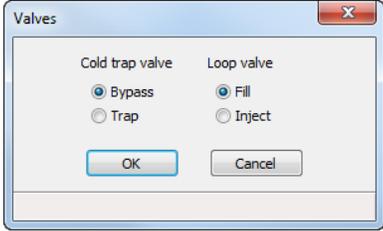
## Experiment Fields and Buttons Table (continued)

Field or Button	Description
	 <p>If a <i>Start Recording</i> step is immediately followed by a step that prompts an immediate peak, peak data are recorded before any baseline readings can be collected. To collect some baseline data before the first peak, insert a <i>Wait for ____ minutes</i> step after the <i>Start Recording</i> step but before the step which causes the peak.</p>
<b>Start Repeat</b>	<p>Specifies the duration of the repeat sequence. Automatically inserts a <i>Start Repeat</i> and a <i>Stop Repeat</i> in the list of steps. Multiple experiment steps can be inserted within the <i>Repeat</i> loop.</p>  <p><b>Repeat ____ times.</b> Stops repeating the steps within the loop when the specified number of times is reached.</p> <p><b>Repeat until peaks are equal or ____ times.</b> Stops repeating the steps within the loop when the last two peaks are equal to within 5% of the area, or when the maximum number of repeats is reached. This option is useful when performing H<sub>2</sub> or CO pulse chemisorption on supported metal catalysts.</p> <p><b>Repeat until peaks are zero or ____ times.</b> Stops repeating the steps within the loop when the last two peaks are each less than 10% of the area of the first peak, or when the maximum number of repeats is reached. This option is useful when performing an N<sub>2</sub>O decomposition for characterizing copper catalysts.</p> <p><b>Repeat until peaks match the first peak or ____ times.</b> Stops repeating the steps within the loop when the last two peaks each match the first peak to within 10% of its area, or when the maximum number of repeats is reached.</p>

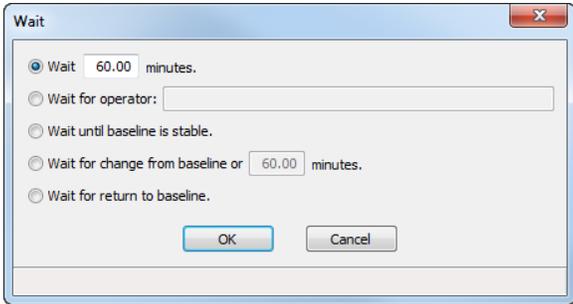
## Experiment Fields and Buttons Table (continued)

Field or Button	Description
Temperature Ramp	<p>Changes the sample temperature.</p>  <p><b>End temperature.</b> The ending temperature for the ramping procedure. If the CryoCooler is installed, it is automatically enabled if an ending temperature below 20 °C is used.</p> <p><b>Furnace ramp.</b> Ramps the furnace temperature directly to the <i>End</i> temperature, ignoring the sample temperature.</p> <p><b>Hold time.</b> How long the sample is to be held at the specified temperature before beginning to cool down.</p> <p><b>Ramp rate.</b> The rate at which the temperature will change after evacuation while advancing to the hold temperature.</p> <p><b>Return to ambient.</b> Allows the furnace temperature only (not the sample temperature) to return rapidly to a temperature to below 45 °C.</p> <p><b>Sample ramp.</b> Ramps the sample temperature to the <i>End</i> temperature. The actual furnace temperature is adjusted to meet this target.</p>

Experiment Fields and Buttons Table (continued)

Field or Button	Description
<b>Termination</b>	 <p><b>Carrier / Reference / Loop Gas.</b> Select the gas to be used during the termination stage of the analysis.</p> <p><b>Enable detector.</b> Enables the <i>Filament temperature</i> field and enables the thermal conductivity detector (TCD).</p> <p><b>Filament temperature.</b> Set the filament temperature from 20 °C to 250 °C. A temperature of 175 °C is commonly used.</p> <p><b>Flow rate.</b> The rate at which gas is to flow.</p> <p><b>Return to ambient temperature.</b> Allows the furnace temperature only (not the sample temperature) to return rapidly to a temperature between 14 °C and 50 °C.</p> <p><b>Set external trigger.</b> If selected, the contact closure used to trigger an external Mass Spectrometer or Calorimeter will be activated during the temperature ramp.</p>
<b>Valves</b>	<p>Sets the cold trap valve and the loop valve as specified.</p> 

## Experiment Fields and Buttons Table (continued)

Field or Button	Description
<b>Wait</b>	<p>Specify a waiting routine.</p>  <p><b>Wait _____ minutes.</b> Specify the time to wait.</p> <p><b>Wait for change from baseline or _____ minutes.</b> Specify the time to wait, click <b>OK</b>, then click <b>Baseline</b> to specify the settings.</p> <p><b>Wait for operator.</b> Enter a description of the operator task. During an analysis, the entered message displays at the appropriate time. The analysis continues after the operator clicks <b>OK</b>.</p> <p><b>Wait for return to baseline.</b> Waits for a return to baseline. If enabled, click <b>OK</b>, then click <b>Baseline</b> to specify the settings.</p> <p><b>Wait until Baseline is stable.</b> Specify if the analysis should wait until the baseline becomes stable. then click <b>Baseline</b> to specify the settings.</p>
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## DEGAS CONDITIONS

### File > Open > [.DEG File]

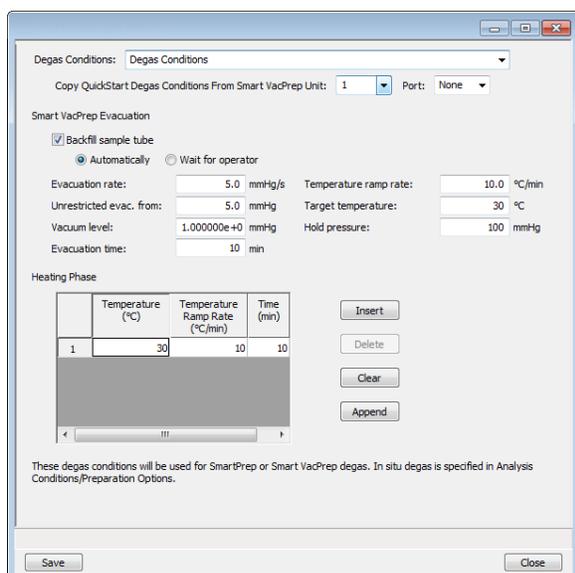
(or click the *Degas Conditions* tab when using *Advanced* presentation display)



Use this option only when the Smart Prep or Smart VacPrep is installed.

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

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.



### Degas Conditions Fields and Buttons Table

Field or Button	Description
<b>Copy QuickStart Degas Conditions from the Smart VacPrep Unit</b> [drop-down box]	Use to copy the degas conditions settings from the selected Smart VacPrep unit and port.
<b>Degas Conditions</b> [drop-down box]	Use to browse for a .DEG file that contains degas condition parameters to be used in the analysis.
<b>Heating Phase</b> [table]	This option is applicable when degassing with either a Smart VacPrep or a SmartPrep.

## Degas Conditions Fields and Buttons Table (continued)

Field or Button	Description
	<p>Enter up to five stages of degas conditions.</p> <p><b>Hold temp.</b> Temperature at which the sample is to be held while degassing.</p> <p><b>Hold time.</b> How long the sample is to be held at the specified temperature before beginning to cool down.</p> <p><b>Ramp rate.</b> The rate at which the temperature will change after evacuation while advancing to the hold temperature.</p>
<p><b>Smart VacPrep Evacuation</b> [<i>group box</i>]</p>	<p><b>Backfill sample tube.</b> Indicate if the sample tube should be backfilled automatically or wait for operator response.</p> <p><b>Evacuation Rate.</b> Rate used for evacuation.</p> <p><b>Evacuation time.</b> Length of time for preliminary evacuation before proceeding with the <i>Heating Phase</i> temperature schedule. The timer starts when the vacuum level is reached.</p> <p><b>Hold pressure.</b> Pressure at which heating will stop and hold the sample temperature approximately constant until the pressure falls below the <i>Hold</i> pressure. This prevents damage to the sample structure due to 'steaming' and /or elutriation due to excessive escaping gas velocity.</p> <p><b>Target temperature.</b> Targeted pressure for evacuation.</p> <p><b>Temperature ramp rate.</b> Rate at which the temperature is to change when advancing to the target pressure.</p> <p><b>Unrestricted evac. from.</b> Pressure at which the unrestricted evacuation is to begin.</p> <p><b>Vacuum level.</b> Evacuation time starts when the vacuum level is reached.</p>
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

## REPORT OPTIONS

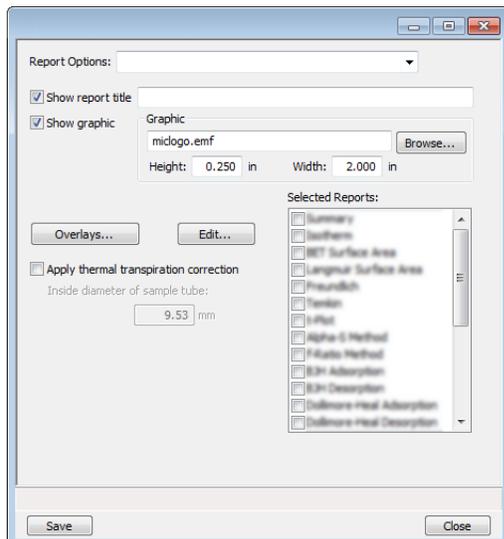
### File > Open > [.RPO File]

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

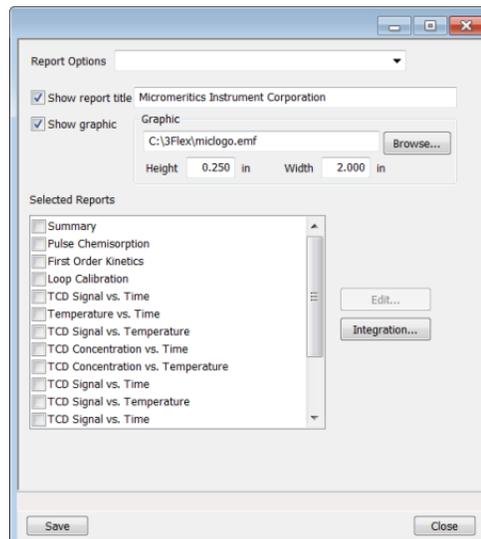
Use to specify report options for data collected from an analysis or manually entered data. *Report Options* files also help in customizing report details such as axis scale, axis range, column headings, and components of thickness curve equations. These files may contain tabular reports, plots, or both,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

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

*Report Options* files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis. See [Graph and Sample Overlays on page 7 - 33](#).



**Physical Adsorption and  
Chemical Adsorption**



**Dynamic Analysis**

## Report Options Fields and Buttons Table

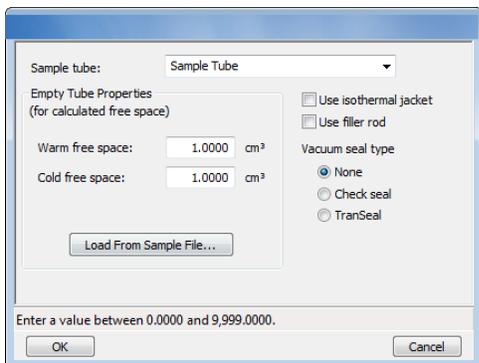
Field or Button	Description
<b>Apply thermal transpiration correction</b> [check box] <b>P SC</b>	<p>Use to correct the temperature-induced pressure difference between the manifold and the chilled sample tube. This option is most significant for pressures less than approximately 1.0 mmHg. Always use thermal transpiration when performing micropore analyses.</p> <p><b>Inside diameter of sample tube.</b> Enabled when <i>Apply thermal transpiration correction</i> is selected. Enter the inside diameter of the sample tube used in the analysis.</p> <p>See the <i>Thermal Transpiration Correction</i> section of the <i>Calculations</i> document. A link to the <i>Calculations</i> document is found in <a href="#">3Flex Links on page iv</a>.</p>
<b>Overlays</b> [button] <b>P SC</b>	See <a href="#">Graph and Sample Overlays on page 7 - 33</a> .
<b>Report Options</b> [drop-down box]	Browse for a .RPO file that contains report options parameters to be used in the report.
<b>Selected Reports</b> [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 <b>Options &gt; Units</b> to specify default calculations.
<b>Show graphic</b> [text box]	Use to show a graphic on the report header.  <b>Height / Width.</b> Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
<b>Show report title</b> [check box]	Select and enter a report title to appear on the report header.
<div style="border: 1px solid green; padding: 5px;">  <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p> </div>	

## SAMPLE TUBE FOR GAS ADSORPTION

### File > Open > [.STB File]

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

*Sample Tube* files specify information about the sample tube.



### Sample Tube Fields and Buttons Table

Field or Button	Description
<b>Cold free space</b> [text box]	Empty sample tube gas capacity measured with the dewar raised.
<b>Load from Sample File</b> [button]	Loads parameters from the selected sample file.
<b>Sample Tube</b> [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 <i>Sample Tube</i> drop-down list on the <i>Sample Description</i> tab.
<b>Use filler rod</b> [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.
<b>Use isothermal jacket</b> [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.
<b>Vacuum seal type</b> [group box]	Select the seal type to be used.

Sample Tube Fields and Buttons Table (continued)

Field or Button	Description
<b>Warm free space</b> [text box]	Empty sample tube gas capacity measured at room temperature.
<div style="border: 1px solid green; padding: 5px;">  <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p> </div>	

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## 5 DEGASSING

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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 to remove the moisture and contaminants.

After the sample has been weighed, degas the sample on:

- the analysis port (see [Degas in Situ on page 5 - 4](#)),
- a Smart VacPrep (see [3Flex Links on page iv](#) for a link to the Smart VacPrep Operator Manual)
- a SmartPrep (see [Degas on the SmartPrep on page 5 - 8](#)), or
- a user supplied degasser

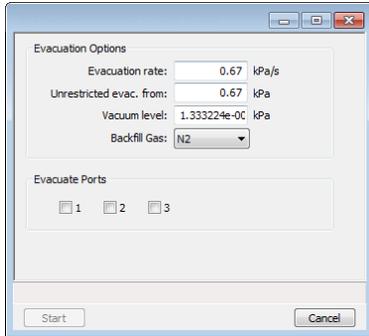
The Check Seal or TranSeal sample tube closures can be used with the Smart VacPrep to minimize sample contamination when transferring the sample tube from the Smart VacPrep to the analyzer port.



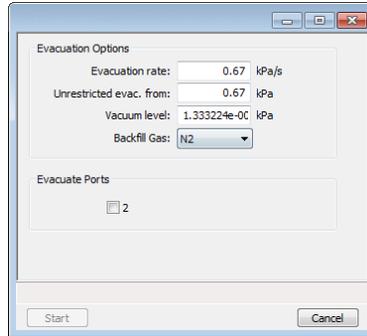
- If using the TranSeal, see the instructions included with the TranSeal (part number 350-42803-00).
- If using the Check Seal, see the instructions included with the Check Seal (part number 350-42802-00).

## EVACUATE PORTS

### Unit [n] > Evacuate Ports



Physical Adsorption



Chemical Adsorption or  
Dynamic Analysis

Allows manual evacuation of degas ports.

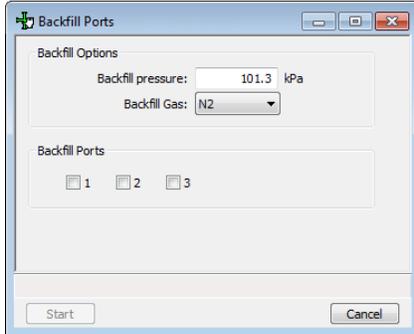
### Evacuate Ports Fields and Buttons Table

Field or Button	Description
<b>Backfill Gas</b> [drop-down box]	Select the backfill gas from the drop-down list.
<b>Evacuate Options</b> [group box]	<b>Evacuation rate. Unrestricted evac. pressure.</b> Pressure value at which unrestricted sample evacuation should begin  <b>Vacuum level.</b> Specify the vacuum level to be achieved before evacuation begins.
<b>Evacuate Port[s]</b> [group box]	Select the ports to evacuate.
	For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4.</a>

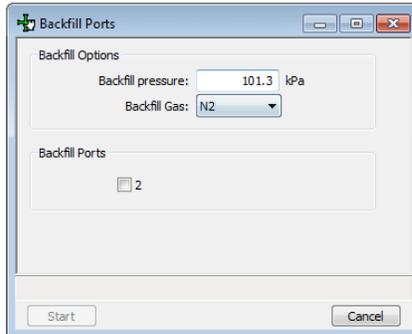
## ***BACKFILL PORTS***

### ***Unit [n] > Backfill Ports***

Use to backfill ports with gas.



**Physical Adsorption**



**Chemical Adsorption or  
Dynamic Analysis**

## DEGAS IN SITU

### Unit [n] > Sample Analysis

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 to remove the moisture and contaminants.

Physical adsorption samples can be degassed on either the analyzer's analysis port (in situ) or on a separate device such as a Smart VacPrep. Degassing should be performed prior to starting analysis.



Samples containing excessive amounts of moisture or significant amounts of other contaminants must be degassed on a separate degas system before attaching to the analyzer to prevent contamination of the analyzer high vacuum system.



Microporous samples should receive a secondary in situ degas on the analyzer to remove any moisture readmitted during transfer from the separate degas system to the analyzer.

When the degas is completed, observe the temperatures on the analyzer schematic. When the mantle has cooled, the *Sample Analysis* window will display a prompt to remove the degas heating mantle, properly position the isothermal jackets and dewar lid, then install the dewar.

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



To prevent potential burns, do not touch the sample tube or the heating mantle until they have cooled below 45 °C.

## TRANSFER A DEGASSED SAMPLE TO AN ANALYSIS PORT

When degassing on a separate degasser such as a Smart VacPrep or SmartPrep, the sample tube must be removed from the degas port, weighed, and then installed onto the analysis port for analysis.



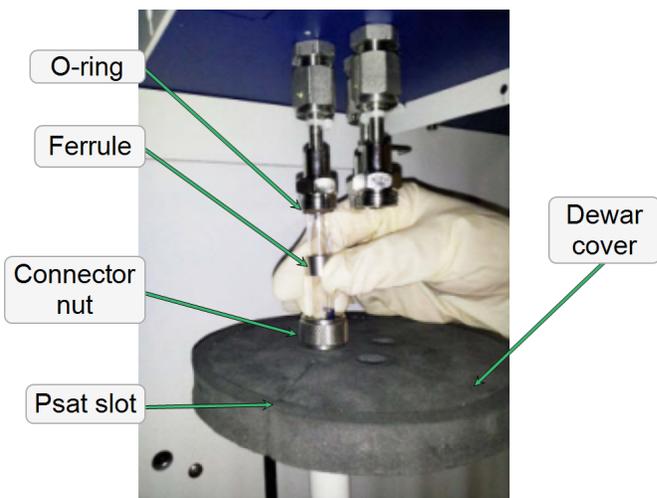
If the sample tube is not mounted on the analysis port immediately, leave it on the degas port. If it is necessary to remove the sample tube and a Check Seal or TranSeal was not used, insert a rubber stopper into the sample tube.

1. Allow the sample tube to cool.

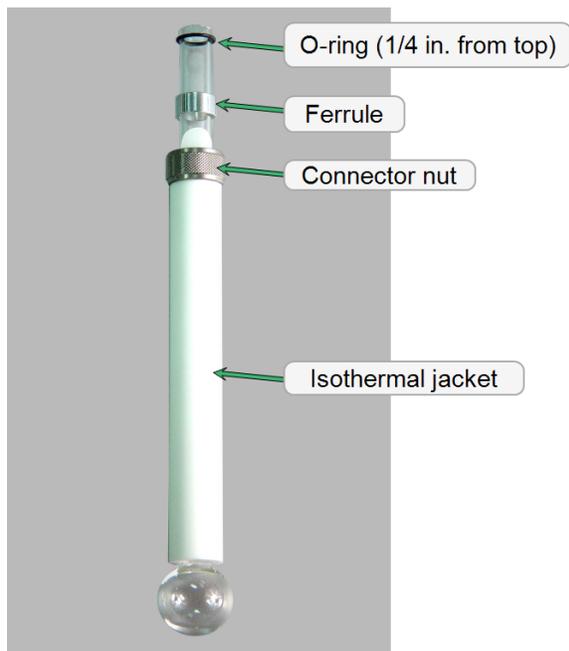
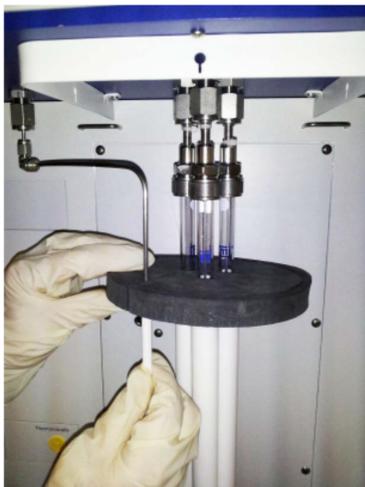


Do not touch the sample tube or the heating mantle until they have reached room temperature. Touching the sample tube, heating mantle, or heating mantle clip before they have cooled could result in burns.

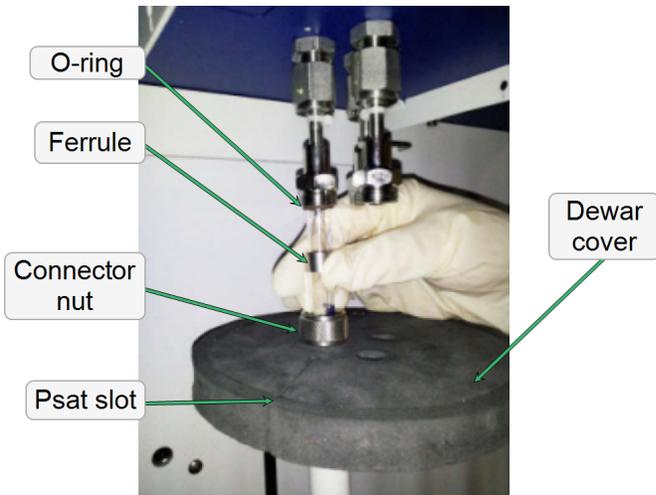
2. Carefully remove the heating mantle clip and the heating mantle from the sample tube and allow the sample tube to cool to room temperature (approximately fifteen minutes).
3. While holding the sample tube, loosen the port connector nut and remove the sample tube from the degas port. If a Check Seal or TranSeal was not inserted prior to degassing, immediately insert a rubber stopper into the sample tube.
4. Complete the [Sample Data Worksheet on page L - 2](#).
5. If using a Check Seal, ensure that the Check Seal opener is installed in the analyzer sample port. If a rubber stopper was used, remove it from the sample tube.
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. On the analyzer, loosen the connector nut on the Psat tube and rotate it out of the way.
9. Position the dewar lid so that the slot for the Psat tube is on the left between port 1 and port 2.



10. Insert the sample tube through one of the holes in the dewar lid.
11. Place the sample port nut, ferrule and O-ring onto the sample tube stem.
12. Insert the sample tube into the analysis port and ensure it is completely in the port. Securely hand tighten the sample port nut onto the analysis port.
13. Repeat all previous steps for each sample tube.
14. Position the dewar lid approximately 3/4 in. (19 mm) below the sample port nut.
15. Slide the Psat tube into the Psat slot in the dewar lid and retighten the Psat tube connector nut.
16. Insert the jacket onto the Psat tube. Ensure that the Psat tube jacket is level with the sample tube isothermal jackets.



17. Attach the sample tube to the analysis port, pushing it fully up. Turn the connector nut clockwise and hand tighten.
18. If degassing a sample on the sample port, see [Degas in Situ on page 5 - 4](#). Otherwise, place the dewar lid over the sample tube stem just above the isothermal jacket. Ensure the Po tube is next to the sample tube.



19. Install the dewar onto the elevator and place the safety shield over the sample tube and dewar.
20. Begin the analysis.

## ***DEGAS ON THE SMARTPREP***

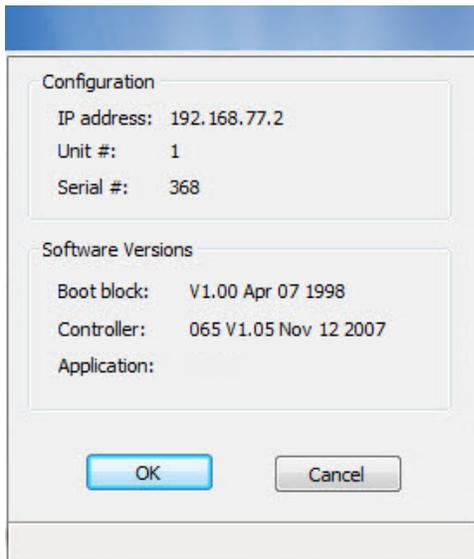
### ***Unit [n] > Degas***

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

## ***SMARTPREP CONFIGURATION***

### ***Unit [n] > Degas > SmartPrep Configuration***

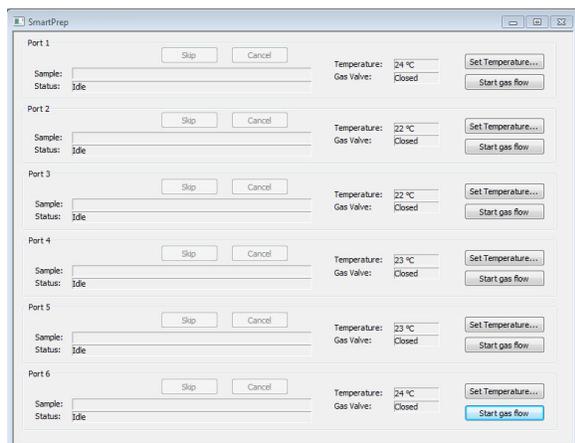
Displays the SmartPrep configuration and software versions.



## SHOW SMARTPREP STATUS

### Unit [n] > Degas > Start SmartPrep Degas

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



### Show SmartPrep Status Fields and Buttons Table

Field or Button	Description
<b>Cancel [button]</b>	Discards any changes or cancels the current process.
<b>Set Temperature [button]</b>	Use to set the temperature of the selected port.
<b>Skip [button]</b>	Use to bypass the current stage.
<b>Stop Gas flow [button]</b>	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 Fields and Buttons Table

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

## ***DEGAS ON THE SMART VACPREP***

See [\*Degasser Options on page 1 - 5\*](#)

Degassing on the Smart VacPrep requires updated analyzer application. If you have not already done so, update the analyzer software for Smart VacPrep support by logging in to your customer portal at [www.Micromeritics.com](http://www.Micromeritics.com). See [\*3Flex Links on page iv\*](#) for a link to the Smart VacPrep Operator Manual.

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## 6 PERFORM AN ANALYSIS

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If using a cryostat for analysis, see [CryoStat on page B - 1](#) prior to use.

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### **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.

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When handling dewars containing liquefied gases or cryogenic liquids:

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

### **FOR GLASS DEWARS**

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

## MIXING AN IPA/LN<sub>2</sub> SLURRY



Improperly mixing an IPA/LN<sub>2</sub> slurry could cause injury. If the mixture is not stirred continuously, gas may build up under the surface, causing the liquids to splash out of the dewar.

An isopropyl alcohol (IPA) /liquid nitrogen (LN<sub>2</sub>) slurry is used to maintain cold trap temperatures of approximately -80 °C. Improperly mixing an IPA/LN<sub>2</sub> slurry could cause injury. If the mixture is not stirred continuously, gas may build up under the surface, causing the liquids to splash out of the dewar.

1. Chill a 600 ml dewar by rinsing it with LN<sub>2</sub>. Allow a small amount of LN<sub>2</sub> to remain in the bottom of the dewar (approximately 1 cm deep).
2. Stirring constantly, slowly add approximately 500 ml of IPA. For greatest safety, use a laboratory squirt bottle. Squirt the stream of IPA along the inside edge of the dewar, close to the top of the dewar, allowing the IPA to flow down the inside wall of the dewar. Do not stop stirring the mixture in the bottom of the dewar, even if stirring becomes difficult. As stirring and adding IPA continues, the mixture will loosen and become easier to stir.
3. When all the IPA has been placed in the dewar, slowly begin pouring LN<sub>2</sub> into the dewar. Approximately 1 liter is needed. Continue to stir the mixture as LN<sub>2</sub> is added. As ice chunks form, break them up and stir them down into the mixture. Avoid splashing. Gently knock ice chunks away from the sides of the dewar and continue stirring. Add liquid nitrogen until the slurry is within 25 mm (1 in.) of the top of the dewar.
4. Lift the stirrer out of the slurry and observe as the slurry drips into the dewar; the stirrer should be thickly coated with slurry.

The goal is to achieve a slurry that has a thick, syrupy consistency without large chunks. When the slurry is cold enough, small pieces of ice may be present (10 - 20% of the mixture). A little ice accumulation along the sides of the dewar is acceptable.

## CHECK THE CRYOGEN LEVEL

If performing an analysis that requires use of the cold trap and cryogen, check the cryogen level in the dewar. It should be about 25 mm (1 in.) from the top.



The cryogen must not be cold enough to trap the carrier gas or analysis gas. Do not use liquid nitrogen with argon carrier gas. For example, use an alcohol and liquid nitrogen slurry (-80 °C).



Use appropriate safety procedures when handling all cryogenes. Be sure to wear safety glasses and gloves, and observe the precautions listed earlier.

## ***DYNAMIC ANALYSIS***

To surround the cold trap, the dewar must rest on a small stand. Hold the dewar beneath the cold trap, then raise it to slide the stand underneath the dewar. Lower the dewar until it rests on the stand. Ensure the cold trap is immersed.

## ***PREPARE FOR ANALYSIS***

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



For dynamic analyses that include manual or loop injections of a chemically active gas, selection of an active gas / inert gas pair is required. The gas to be injected may be a blend of chemically active gas and inert gas, or a pure active gas. If a blend is used for injections, the inert gas in the blend should be the same as the carrier gas. See [Adsorptive Properties on page 4 - 2](#) and [Gas Charts on page E - 1](#). The inert carrier gas should be selected to have a very different thermal conductivity from the active gas so as to provide a large change in the thermal conductivity detector signal, for instance helium (inert, T.C.=5.84) with carbon monoxide (active, T.C.=0.97) or argon (inert, T.C.=0.68) with hydrogen (active, T.C.=7.07). The selected carrier gas must be connected to one of the 12 primary gas inlets on the back panel of the instrument. For manual injections, the gas to be injected must be connected to an external septum (provided in the accessory kit) to allow filling the syringe. For loop injections, the gas to be injected must be connected to one of the four loop gas inlets on the side of the instrument. Be sure to enter the mnemonics for the connected gases under [Specify Gas Ports on page 2 - 15](#).

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

[Step 1 - Clean and Label Sample Tubes on the next page](#)

[Step 2 - Create the Sample File on page 6 - 5](#)

[Step 3 - Determine the Sample Mass on page 6 - 6](#)

[Step 4 - Degas the Sample on page 6 - 11](#)

[Step 5 - Install the Sample Tube on page 6 - 11](#)

[Step 6 - Fill and Install the Dewar on page 6 - 18](#)

## STEP 1 - CLEAN AND LABEL SAMPLE TUBES

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

Supplied by Micromeritics	Supplied by User
<ul style="list-style-type: none"> <li>• Filler rod</li> <li>• Funnel</li> <li>• Sample data worksheet</li> <li>• Sample tube</li> <li>• Sample tube brush</li> <li>• Sample tube rack</li> <li>• Sample weighing support (required for chemical adsorption sample tubes)</li> <li>• Stopper for sample tube (Check Seal, rubber stopper, or TranSeal)</li> </ul>	<ul style="list-style-type: none"> <li>• Acetone or isopropyl alcohol</li> <li>• Analytical balance</li> <li>• Detergent</li> <li>• Drying oven</li> <li>• Pipe cleaners</li> <li>• Rubber gloves or lint-free cloth</li> <li>• Safety glasses</li> <li>• Ultrasonic cleaning unit</li> <li>• Waste container</li> </ul>

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



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



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



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

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



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

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

## STEP 2 - CREATE THE SAMPLE FILE

See [Sample Files on page 3 - 1](#)



For dynamic analyses, the *Loop Volume* and *Environmental Default* values must be correct before starting an analysis.

### STEP 3 - DETERMINE THE SAMPLE MASS

See [Worksheets on page L - 1](#)

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

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

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

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

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

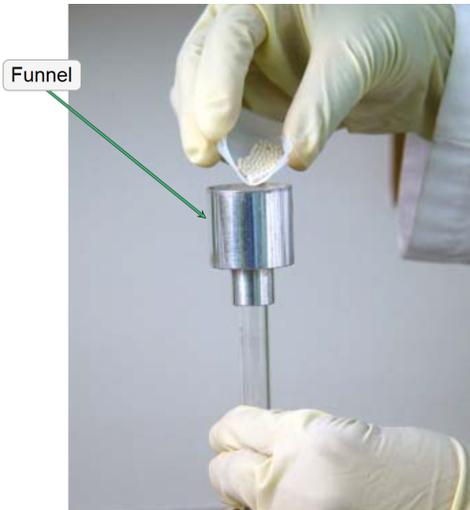
### DETERMINE SAMPLE MASS FOR PHYSICAL ADSORPTION

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



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

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



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

## DETERMINE SAMPLE MASS FOR CHEMICAL ADSORPTION



Bulb sample tubes are for pellets and other samples without loose particles. Using powder samples in bulb tubes may cause the loose particles to go into the analyzer's exhaust.

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.
3. If analyzing a powder or sample made of fine particles, push a piece of quartz wool all the way down into the sample tube. See [Use Quartz Filter Discs for Chemical Adsorption on the facing page](#).
4. If using quartz wool, put a second piece of quartz wool just inside the sample tube. If using filter discs, push a filter disc down into the tube until it sits on top of the quartz wool. Place a second filter disc just inside the sample tube.
5. Place the sample tube set (sample tube with quartz wool or filter discs and stoppers) on the sample support. Record the stabilized mass as the *[A] Mass for empty sample tube set* on the *Sample Data Worksheet*.



6. Remove the sample weighing support and sample tube set from the balance.
7. Place the sample container on the balance and allow the balance to stabilize at zero.



Do not touch the sample with bare hands. Oil from hands could affect the accuracy of results.

8. Slowly add approximately 0.5 to 1.0 gram of sample to the sample container.
9. If a second piece of quartz wool or filter disc was inserted, remove the top portion of the quartz wool or the filter disc from the sample tube.

10. Use a funnel to slowly pour sample from the container into the sample tube on top of the quartz wool in the tube.



Ensure all sample in the container is placed in the sample tube to avoid errors caused by incorrect sample mass.

11. If using quartz wool, insert the top portion of quartz wool into the tube and press it down. If using filter discs, insert the filter disc into the tube and press it down.



Ensure the disc is flat on top of the sample. A seal must be created around the edge to prevent the sample from escaping.

12. Wipe the top of the sample tube with a lint-free cloth, such as a Kimwipe<sup>®</sup>, to remove any quartz wool that may have adhered to the surface.
13. Weigh the sample tube set containing the sample and the stoppers. Record this mass as the *Sample + tube*.

### **Use Quartz Filter Discs for Chemical Adsorption**



The use of quartz wool is not mandatory, however it can provide extra protection for light powdered samples.



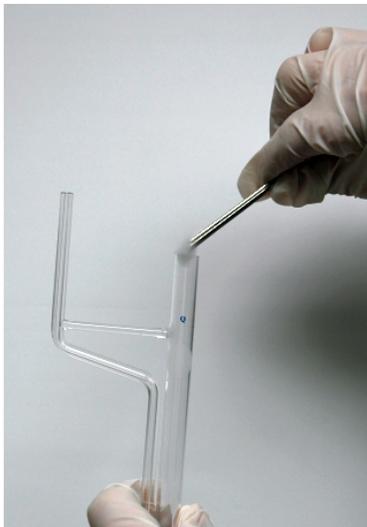
Wear latex gloves when handling the sample tube. The natural oils in human skin can chemically damage and weaken the quartz tube. It is also important that the sample tube and its components, as well as the sample and exhaust ports, be clean and free of debris. Dust particles from quartz wool or the insulator disc of previous analyses may adhere to the port and/or components, preventing a proper seal of the sample tube.



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

Use quartz filter discs or quartz wool to aid in chemical adsorption sample preparation. Quartz filter discs (placed both below and above powdered samples) not only provide a more uniform sample surface but also keep the analyzer free of sample debris. The filters can be used up to 900 °C.

1. Insert a small portion of quartz wool into the sample tube to serve as a support for the powdered sample. Use a filler rod or smaller sample tube to push the quartz wool to the bottom of the sample tube.



Insert quartz wool and disc



Top disc, powdered sample, bottom disc, quartz wool

2. Insert a quartz disc into the sample tube and push it into the tube until it rests on top of the quartz wool. Inspect the disc to ensure that there is a good seal and that the sample will not go past the filter. An additional filter can be inserted if needed.
3. Insert a second filter disc on top of the quartz wool. Ensure that the filter is placed high enough into the sample tube for easy retrieval.
4. Take the initial tube weight (with both filters).
5. Remove the top filter disc. Place it on a clean surface, then use a funnel to add the powdered sample on the bottom filter disc.
6. Reinsert the top filter disc into the sample tube, then use a rod or smaller sample tube to push it down until it reaches the top of the sample.
7. To remove the quartz wool and disc after analysis, use the quartz wool extractor tool.

## STEP 4 - DEGAS THE SAMPLE



For instructions on degassing on the SmartPrep, see [Degas on the SmartPrep on page 5 - 8](#).



If using the Smart VacPrep degasser, go to **Smart VacPrep > Prep [n] > Start Degas**, then degas the sample using menu commands and information entered on the *Degas Conditions* tab. See [3Flex Links on page iv](#) for a link to the Smart VacPrep Operator Manual.

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

## STEP 5 - INSTALL THE SAMPLE TUBE

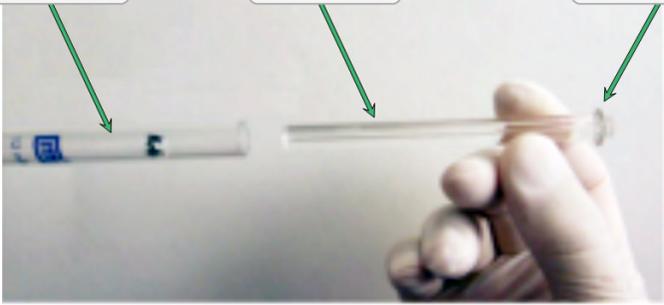
### Sample Tube Installation for Physical Adsorption



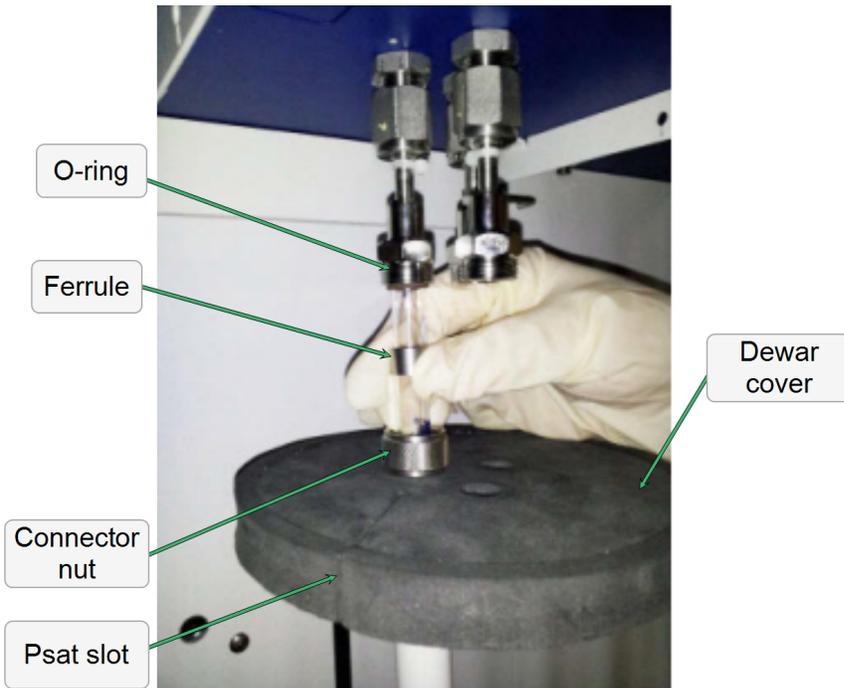
The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

Repeat the following steps for each sample to be installed.

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

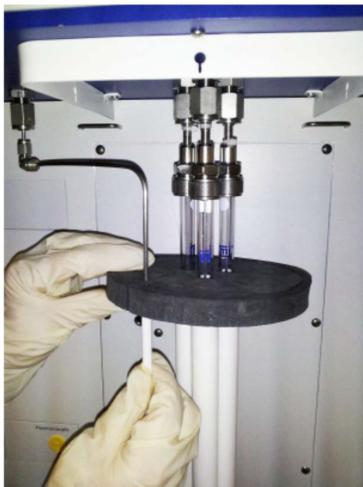
If using...	Then...
	<div style="display: flex; justify-content: space-around; margin-bottom: 10px;"> <span>Sample tube</span> <span>Filler rod</span> <span>Metal clip</span> </div>  <div style="border: 1px solid black; padding: 5px; margin-top: 10px;">  <p>Do not hold the rod vertically and drop the rod into the tube; this could break the rod and/or tube.</p> </div>

1. Loosen the connector nut on the Psat tube and rotate it out of the way.
2. If using a Check Seal, verify that the port has the Check Seal opener installed or if using a TranSeal, install it at this time.
3. Position the dewar lid so that the slot for the Psat tube is on the left between ports 1 and 2.



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

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

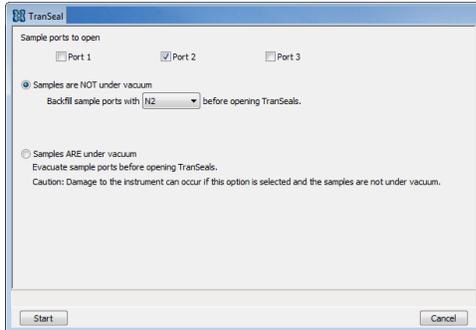


- If using the TranSeal, see the instructions included with the TranSeal (part number 350-42803-00).
- If using the Check Seal, see the instructions included with the Check Seal (part number 350-42802-00).

## **Open the TranSeal**

### **Unit > Open TranSeal**

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



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

### **TranSeal Fields and Buttons Table**

<b>Field or Button</b>	<b>Description</b>
<b>Sample ports to open</b> [group box]	Select the ports to open during analysis.
<b>Samples are NOT under vacuum</b> [selection]	Select if samples are NOT under vacuum and specify the amount of backfill and adsorptive to be used prior to opening TranSeals.
<b>Samples ARE under vacuum</b> [selection]	Select if the samples ARE under vacuum. This option evacuates sample ports prior to opening TranSeals. Do not select this option if the samples are not under vacuum as analyzer damage may occur.
<b>Start</b> [button]	Opens the TranSeals. The selected sample port will be either backfilled or evacuated as specified. The user will be prompted to open the TranSeals. For each selected port, an event is recorded in the <i>Instrument Log</i> file with the port pressure before and after opening the TranSeal.



For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 4](#).

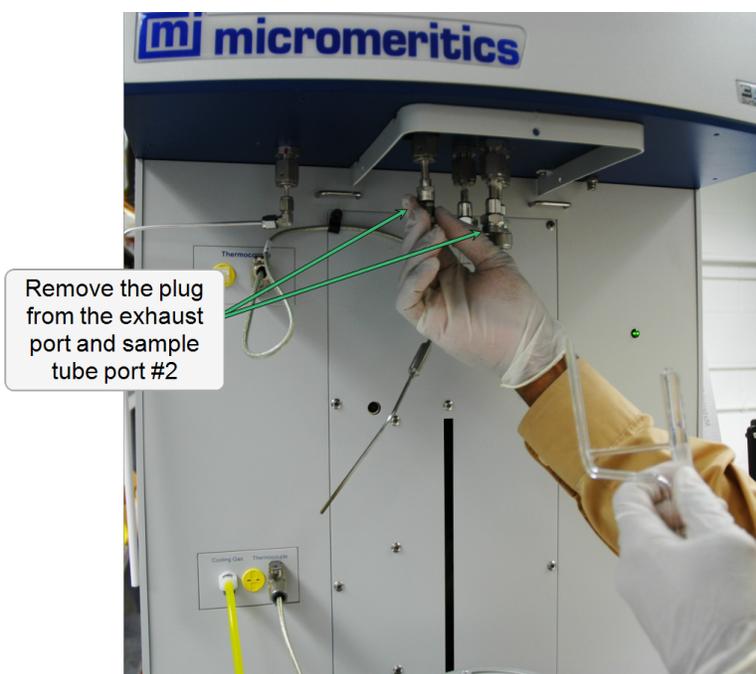
## SAMPLE TUBE INSTALLATION FOR CHEMICAL ADSORPTION



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



Wear latex gloves when handling the sample tube. The natural oils in human skin can chemically damage and weaken the quartz tube. It is also important that the sample tube and its components, as well as the sample and exhaust ports, be clean and free of debris. Dust particles from quartz wool or the insulator disc of previous analyses may adhere to the port and/or components, preventing a proper seal of the sample tube.

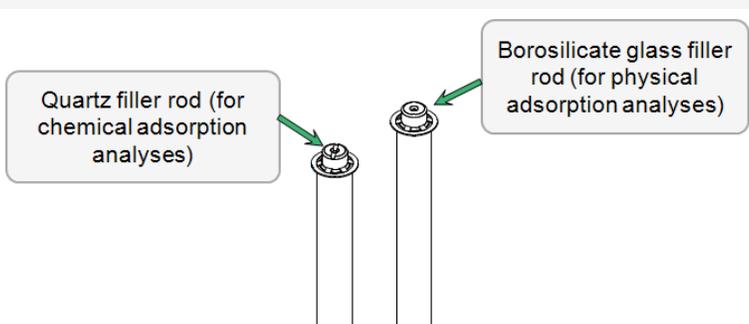


1. Remove the stopper from the sample tube stem and the cap from the exhaust stem.
2. Use a lint-free swab moistened with IPA and wipe the interior rims of the sample and exhaust ports.
3. Use a lint-free tissue moistened with IPA and wipe the O-ring, ferrule, and connector nuts for the sample and exhaust tubes. Place on a lint-free tissue.



Sample and exhaust ports, as well as all components that contact the sample and exhaust ports, must be clean, therefore it is recommended that the previous steps be repeated each time a sample tube is installed onto a port

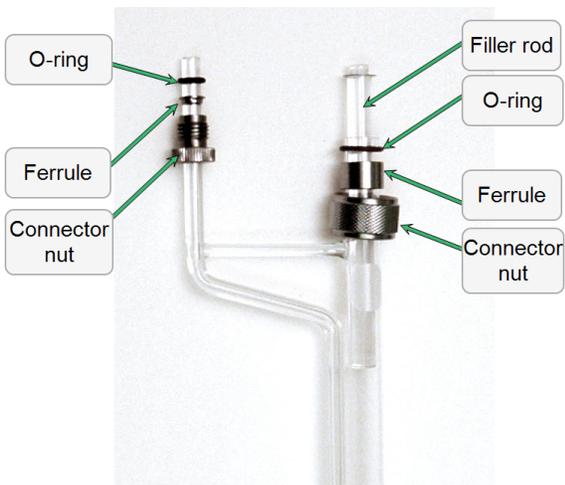
4. Remove the stopper from the sample tube stem and the cap from the exhaust stem.
5. If using a hanging filler rod (recommended), hold the sample tube slightly tilted and carefully place the filler rod into the tube.



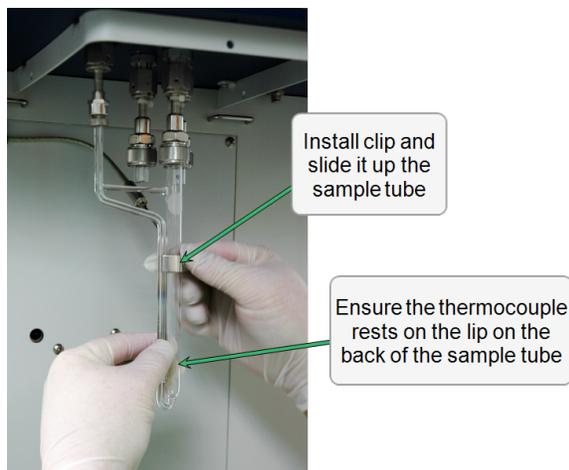
The quartz filler rod can be used for both physical adsorption and chemical adsorption analyses; however, the borosilicate glass filler rod can only be used with physical adsorption analyses. The quartz filler rod can be identified by the notch across the top of the rod.

Use of the borosilicate glass filler rod in a chemical adsorption analysis can cause damage to the instrument.

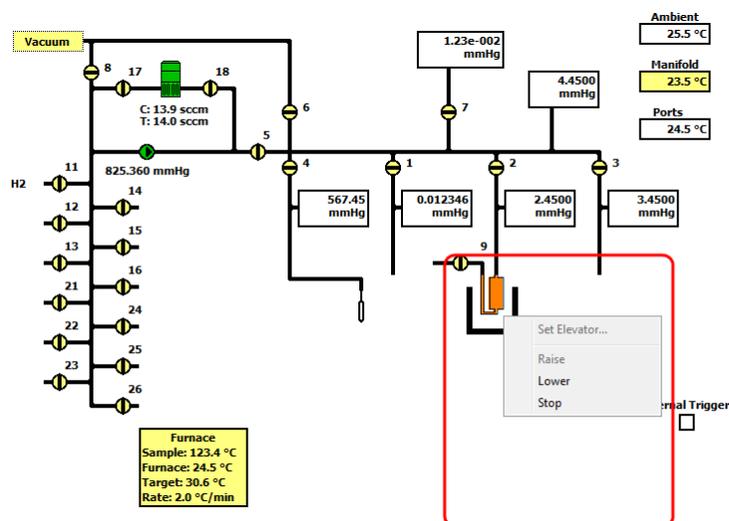
6. Assemble and install the sample and exhaust tube components.



7. Insert the assembled sample tube into the exhaust port and analysis port #2.
8. Slide the connector nuts up the stems and screw the nuts clockwise to secure the tube in place. Hand tighten both connector nuts until snug.



9. Rest the sample thermocouple tip on the lip on the back of the sample tube and install the sample tube clip around the sample tube and thermocouple. Ensure the clip is high enough on the sample tube to clear the furnace disk when the elevator and furnace are raised.
10. Ensure the furnace is on the elevator shelf and manually raise the elevator. To raise the elevator, go to **Unit [n]** and ensure there is a check mark to the left of the chemical adsorption option. If not, select **Unit [n] > [Chemical Analysis]** to select it.
11. Go to **Unit [n] > Enable Manual Control**. Ensure a checkmark displays to the left of the menu item. If the instrument schematic does not display, go to **Unit [n] > Show Instrument Schematic**.



12. On the schematic, right click the furnace icon and select *Raise* to raise the elevator. If it is necessary to stop the elevator, right click the elevator icon again and select *Stop*.
13. When the elevator reaches the top, insert the two furnace disk halves on top of the furnace opening. Place the first disk behind the sample tube and the second disk in front of the sample tube. Ensure the clip remains above the furnace disks.



### **STEP 6 - FILL AND INSTALL THE DEWAR**



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

See [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. If using isothermal jackets, slide the jackets down the sample tube until the jackets touch the sample tube bulbs.
5. Slide the dewar lid to approximately 3/4 in. (19 mm) from the sample port nuts to ensure a proper seal on the top of the dewar.
6. Attach the safety shield to the brackets on the front of the analyzer.

## PERFORM AN ANALYSIS SEQUENCE

### Unit [n] > Analysis Sequence



If using a cryostat for analysis, see [CryoStat on page B - 1](#) prior to use.



After running a chemical adsorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physical adsorption tubes until the system vacuum level is at  $9 \times 10^{-6}$  mmHg or lower.

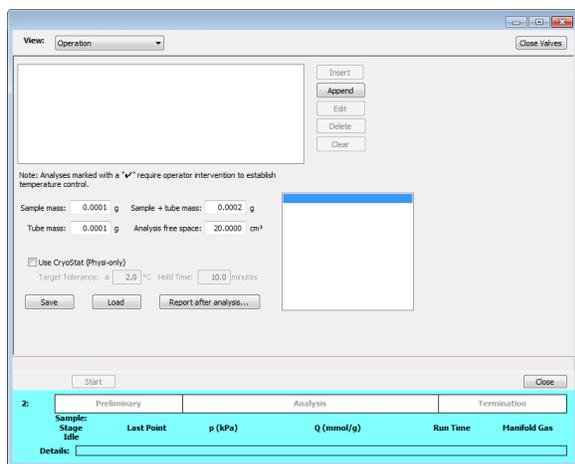
Use to perform a sequence of analyses on port 2. If the *Analysis Sequence* option is selected and a sequence analysis is already in progress, the program continues to run the four steps for the analysis in progress. Files can be added and removed from the sequence while it is in progress.

The analysis sequence can:

- run a sequence of only physical adsorption files
- run a physical adsorption analysis as a part of the chemical adsorption analysis sequence
- run a physical adsorption file in any position in the sequence

The physical adsorption live graph will be used for the physical adsorption analysis within a chemical adsorption sequence.

Operator intervention will be required between analyses in the sequence if the temperature control type changes. Intervention will be required if at the beginning of the analysis.



1. Click **Close Valves**.
2. **Insert** a sample file.

3. Edit the fields below the sample file selections.
4. Click **Save** to save the selected files as an analysis sequence file (.SEQ) for future analyses.
5. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.
6. When the analysis is complete, remove the sample tube and store (or dispose of) the sample material as applicable.



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

### Analysis Sequence Fields and Buttons Table

Field or Button	Description
<b>Close Valves</b> [ <i>button</i> ]	Closes all valves on the unit.
<b>Report after analysis</b> [ <i>button</i> ]	Generates reports to the selected destination when the analysis is complete.
<b>Sample mass / Sample + tube mass / Tube mass / Analysis free space</b> [ <i>text box</i> ]	Enter the values for the sample's mass and free space.
<b>Use CryoStat</b> [ <i>checkbox</i> ] 	<b>For cryostat use only.</b> If using a cryostat, select and enter the <i>Target Tolerance</i> and <i>Hold Time</i> . The cryostat temperature will be ramped (if ramp is enabled) to the sample analysis temperature. Once the cryostat temperature is within the entered tolerance, the analysis will wait for the hold time before proceeding.



For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 4](#).

## PERFORM A BLANK ANALYSIS

### Unit [n] > Reference Analysis

See [Perform a Reference Analysis on page 6 - 25](#)



After running a chemical adsorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physical adsorption tubes until the system vacuum level is at  $9 \times 10^{-6}$  mmHg or lower.

The *Blank Analysis* is run in the same manner as a *Reference Material* analysis except a blank method is selected and no sample material is placed in the sample tube.

## PERFORM A PULSE CHEMICAL ADSORPTION ANALYSIS



This topic provides an example of Pulse Chemisorption being performed on a sample of Pt/Al<sub>2</sub>O<sub>3</sub> with CO. Make the appropriate modifications for the material you are analyzing. Platinum Alumina Reference Material can be ordered from Micromeritics (part number 004-16825-00).

A Pulse Chemisorption analysis determines the quantity of active gas irreversibly adsorbed. By applying pulses of known quantity of active gas to the sample, calculations of active surface area, percent metal dispersion, and active particle size can be made for supported metals. The sample is dosed with the analysis gas using the injection loop or a syringe until all accessible active sites are covered. The amount chemisorbed is the difference between the total amount of active gas injected and the amount that does not interact irreversibly with the sample.

The number of injections depends on the quantity in each pulse and total number of active sites present

### LOOP VOLUME

The gas quantity in each pulse is determined by active gas concentration, the loop or syringe volume, temperature, and pressure. The loop temperature is controlled by the instrument. The ambient (syringe) temperature and atmospheric pressure are entered by the operator (see [Environmental Defaults for TCD Analyzers on page 2 - 20](#)). If a syringe is used, the volume injected is determined by the operator. In general, it is desirable for the sample to require at least two doses of gas, but no more than ten doses, before the reaction ends (although this may vary from lab to lab). Some factors that influence the number of doses required are sample size, the density of active sites, the concentration of active gas, and the size of the loop.

A 0.5 cm<sup>3</sup> loop is provided with the analyzer and an optional 1 ml loop is available.

## LOOP CALIBRATION

For the most accurate determination of the injected gas quantity, a loop calibration experiment should be performed. An independent loop calibration can be performed before or after the analysis.

The following example assumes that the loop calibration will be performed after the analysis. A *Loop Calibration* step can be included in a pulse chemisorption analysis. See [Loop Calibration for TCD Analyzers on page 10 - 12](#).

## PREPARATION

<b>Pretreatment</b>	Degas by flowing inert gas (such as helium, argon, or nitrogen) over the sample while ramping the temperature. Hydrogen of at least 10% concentration is generally used for reduction.
<b>Analysis</b>	Pulse the loop (analysis) gas over the sample until the peak area remains constant.
<b>Cold Trap</b>	The cold trap does not need to be cooled during the reference material analysis. This may vary depending on application or analysis method. The cold trap tube should remain on the instrument. Including the cold trap in the flow path is optional.
<b>Pressure regulator</b>	Gas cylinders should be set to a level between 10 and 15 psig (69 and 103.5 kPag).
<b>Furnace temperature</b>	Select a temperature high enough to remove any contaminants or moisture, but not so high as to cause sintering or fusing of the sample. Ensure the <i>Termination</i> step is set to return the sample temperature to ambient.



Before performing an analysis, ensure the sample and analyzer are adequately prepared using the instructions in [Prepare for Analysis on page 6 - 3](#).

## PROCEDURE

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer. Raise the furnace around the sample tube and install the insulator disc and shield.
2. Create a new sample file for this analysis. In the *Analysis Conditions* drop-down box, select either *Pt-AI CO Pulse Chemisorption Loop* or *Pt-AI CO Pulse Chemisorption Syringe* and modify the steps as needed depending on the analysis to be performed.
3. On the *Report Options* tab, select the required report options for the analysis.
4. Save and close the file.

5. Go to **Unit [n] > Sample Analysis** and select the sample file saved in the previous step. chemical adsorption can only be performed on Port 2, therefore Port 2 is the only selection available.
6. Click **Start** to start the analysis.
7. When prompted, select the calibrations associated with each experiment in the sample file (if applicable). For this example, select *None*. Calibration files can also be associated with a sample file after analysis using the *Peak Editor*. See [Peak Editor for Dynamic Analysis on page 7 - 11](#).



When the analysis ends, the furnace begins to lower the sample temperature to room temperature. When the sample has cooled, lower the furnace and remove the sample tube when the analysis is complete.



Use the cotton gloves provided in the accessory kit to remove the sample tube before it has cooled. Rubber gloves may be used to handle the sample tube when it has cooled.

## GENERATE THE REPORT

Open the Peak Editor and ensure that peaks are properly marked. See [Peak Editor for Dynamic Analysis on page 7 - 11](#).

There are three possibilities for each dose of gas injected during Pulse chemisorption :

- all of the gas is taken up by the sample,
- some of the gas is taken up by the sample, or
- none of the gas is taken up by the sample.

When Pulse chemisorption is properly performed, there will be some injections of each type. When the data is viewed using the Peak Editor, however, only those injections in which some or none of the gas is taken up will appear as peaks. When all of the gas is taken up by the sample, none of it reaches the detector and, therefore, the peak area is zero. These types of peaks are detected automatically by the application and do not require marking.

To generate the report, go to **Reports > Start Report> [.SMP file created above]** or click **Preview** and select the signal view.

## PERFORM A REFERENCE ANALYSIS

### Unit [n] > Reference Analysis



If using a cryostat for analysis, see [CryoStat on page B - 1](#) prior to use.



After running a chemical adsorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physical adsorption tubes until the system vacuum level is at  $9 \times 10^{-6}$  mmHg or lower.

A reference analysis is used to verify the analyzer is operating properly and producing optimum results. These methods provide specifications for critical report quantities and reporting of whether quantities are in or out of specification.

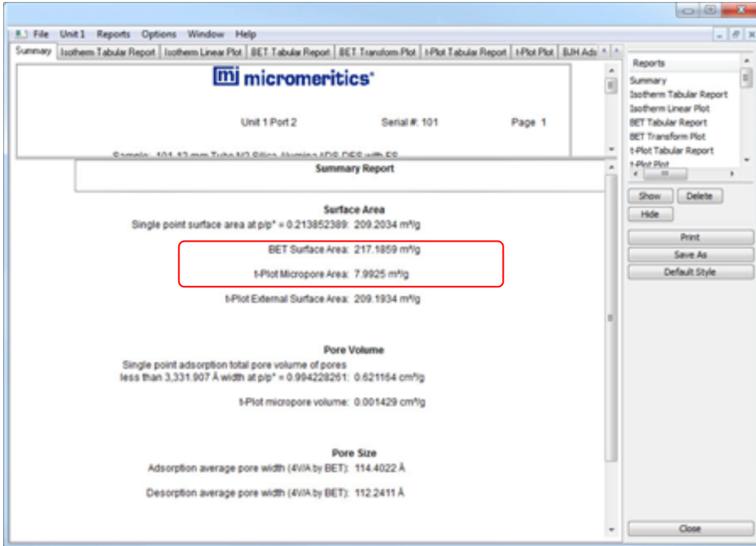
When running a reference analysis, use the appropriate reference material provided in the accessories kit to perform this analysis. The results should match those shown on the label of the reference material bottle, within the tolerance level.

**P** If using a cryostat, select *Use CryoStat*, enter a *Target Tolerance* and a *Hold Time*. The cryostat temperature will be ramped (if ramp is enabled) to the sample analysis temperature. Once the cryostat temperature is within the entered tolerance, the analysis will wait for the hold time before proceeding.

**Physical Adsorption**

**Chemical Adsorption or  
Dynamic Analysis**

If a *Blank Analysis* was selected, look at the *Blank Analysis Report*. The isotherm points should all fall between the minimum and maximum specification lines.



- If the results are within tolerance, the analyzer is operating properly. Click **Close**.
- If the results are not within tolerance, refer to the following *Cause and Action* table. After performing the action, perform the reference analysis again.

**Cause and Action Table**

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

## PERFORM A SAMPLE ANALYSIS

### Unit [n] > Sample Analysis

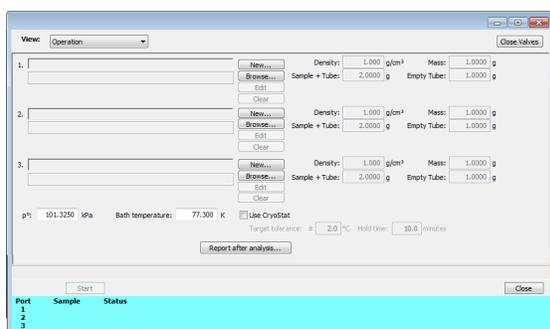


If using a cryostat for analysis, see [CryoStat on page B - 1](#) prior to use.

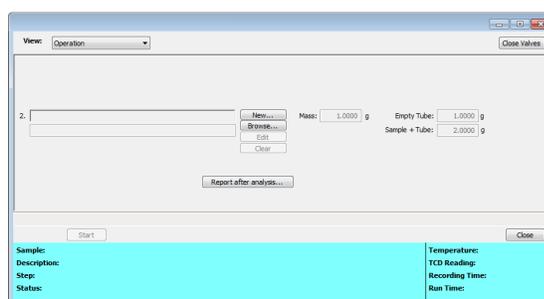


After running a chemical adsorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physical adsorption tubes until the system vacuum level is at  $9 \times 10^{-6}$  mmHg or lower.

See [Prepare for Analysis on page 6 - 3](#)



**Physical Adsorption**



**Chemical Adsorption or  
Dynamic Analysis**

**P** Use to perform up to three analyses with different analysis conditions and/or report options. Sample files can be loaded into ports 1, 2, and 3 allowing one analysis using different analysis conditions to run on each port.

**C** Use to perform an analysis on port 2.

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

- The minimum target pressure must be no less than the *Minimum Record Pressure* for the minimum range transducer present on the port.
- The selected sample files will be checked for matching adsorptive gases, matching  $P_{sat}$  or  $P_0$  gases if measured, and matching backfill gases. All selected gases, except those in a pre-charged vapor source, must be connected to gas inlets and identified on the *Unit [n] > Unit Configuration > Gas Selections* window. See [Specify Gas Ports on page 2 - 15](#).

- If any selected sample file specifies an *Adsorptive Dose Method* from port 3 and a sample file is selected for port 3, an error message displays indicating the problem and the *Start* window will remain active.



- If *in-situ Degas* is selected for any samples, the operator is prompted to raise the isothermal jackets, then connect and install the degas heating mantle on the sample tubes. If this occurs, the operator will be prompted after degas to remove the heating mantle and properly position both the isothermal jackets and dewar lid.
- If *Vapor Source Temperature Control* is selected and the vapor heating mantle is not connected, the operator is prompted to install and connect the vapor heating mantle. If *Degas* is selected for any samples, this will occur after the prompt to remove the degas heating mantle. Otherwise, this will occur immediately at the start of analysis.

Click **Next** to schedule additional analyses after the completion of the first series of analyses. The **Next** button displays after the first set of analyses is complete. Samples cannot be removed from or added to ports until the full set of analyses has completed.

1. Click **Close Valves** to manually close all analyzer valves.
2. For a selected port, either click **Browse** and select a sample file, or click **New** to create a new sample file.



**P** On Port 1, up to three sample files may be selected. The files will be loaded into ports 1, 2, and 3 in the order they appear in the file selector.

**P** A sample cannot be attached to port 3 if port 3 is being used as the vapor source.

**C** Port 2 is the only available port.

3. Verify the information populated into the sample identification. This information is pulled from the selected or newly created sample file. The *Density* value is applicable only if using the *Calculate* method for the free space determination.
4. **P** Edit the  $p^{\circ}$  and *Bath temperature* fields, if necessary.
5. If using a cryostat, select *Use CryoStat* and complete the *Target tolerance* and *Hold time* fields.
6. 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.
7. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.



**DA** When running a dynamic analysis, after starting the analysis several windows display with various warnings, messages, and prompts for the user to perform a specific task. Tasks will depend on the experiments in the current analysis — such as: wait status, manual injections, or calibration file selection.

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



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

### Analysis Fields and Buttons Table

Field or Button	Description
<b>Bath temperature</b> [text box] <b>P</b>	Enter the temperature for the analysis bath.
<b>Close Valves</b> [button]	Closes all valves on the unit.
<b>Density / Mass / Sample + Tube / Empty Tube</b> [text box]	Enter values for the sample's mass and density. These values may be edited after analysis.
<b>New</b> [button]	Creates a new sample file.
<b>p<sup>o</sup></b> [text box] <b>P</b>	Enabled if <i>Entered</i> is selected for the p <sub>0</sub> measurement for at least one file.
<b>Report after analysis</b> [button]	Generates reports to the selected destination when the analysis is complete.
<b>Use CryoStat</b> [checkbox] <b>P</b>	<b>For cryostat use only.</b> If using a cryostat, select and enter the <i>Target Tolerance</i> and <i>Hold Time</i> . The cryostat temperature will be ramped (if ramp is enabled) to the sample analysis temperature. Once the cryostat temperature is within the entered tolerance, the analysis will wait for the hold time before proceeding.



For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 4](#).



Sample Analysis Graph Fields and Buttons Table

Field or Button	Description
<b>Live Graph Settings</b> [button]	Select Thermal transpiration, X-axis Quantity (relative or absolute pressure) and the X-Axis Scale (linear or logarithmic).
<b>Report after analysis</b> [button]	Generates reports to the selected destination when the analysis is complete.
<b>Port</b> [button]	Generates a report on data being collected. The reports are printed to the screen only.
<b>Resume</b> [button]	Restarts the suspended analysis.
<b>Skip</b> [button]	Moves to the next step. This button is visible only when an analysis is in progress. Select the ports to skip.
<b>Status window</b>	Displays the last point pressure and relative pressure for each port.
<b>Suspend</b> [button]	Suspends an analysis in progress.

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

## ***PERFORM A TEMPERATURE PROGRAMMED DESORPTION ANALYSIS***



This topic provides an example of how to perform a Temperature Programmed Desorption analysis using  $\text{NH}_3$  on calcium oxalate under helium with a  $10\text{ }^\circ\text{C}/\text{min}$  temperature ramp. Make the appropriate modifications for the material you are analyzing.

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Temperature Programmed Desorption (TPD) analyses determine the quantity, type, and strength of active sites available on the surface of a catalyst from measurement of the amount of gas desorbed at various temperatures.

After the sample has been outgassed, reduced, or otherwise prepared, a steady stream of analysis gas flows over the sample and reacts with the active sites. (Alternatively, Pulse chemisorption can be used to react with active sites.) Programmed desorption begins when the temperature is ramped linearly over time while a constant stream of inert carrier gas passes over the sample.

At a certain temperature, the heat will overcome the activation energy, breaking the bond between the adsorbate and adsorbent. The adsorbed species will then desorb. If different active metals are present, they usually will desorb the reacted species at different temperatures. The desorbed molecules enter the stream of inert carrier gas and are swept to the detector where the detector response is proportional to the gas concentrations. The quantity of desorbed species, combined with the stoichiometry factor, and the temperature at which pre-adsorbed species desorb, yield the quantity and strength of active sites, respectively.

If TPD is performed after coverage of the active sites by flow or Pulse chemisorption, additional information about the distribution of active sites and the strength of active sites is collected.

## PREPARATION



Before performing an analysis, ensure the sample and analyzer are adequately prepared using the instructions in [Prepare for Analysis on page 6 - 3](#).

## PROCEDURE

1. For  $\text{NH}_3$  on zeolite, a sample mass of 50 mg  $\pm$  10 mg should be used. Install the loaded sample tube and thermocouple on the analyzer. Raise the furnace around the sample tube and install the insulator disc and shield.
2. Create a new sample file for this analysis. In the *Analysis Conditions* drop-down box, select *Y Zeolite with nitrogen, measure freespace* and modify the steps as needed depending on the analysis to be performed.
3. On the *Report Options* tab, select the required report options for the analysis.
4. Save and close the file.
5. Go to **Unit [n] > Sample Analysis** and select the sample file saved in the previous step.
6. Click **Start** to start the analysis.
7. When prompted, select the calibrations associated with each experiment in the sample file (if applicable). For this example, select *None*. See [Peak Editor for Dynamic Analysis on page 7 - 11](#).



When the analysis ends, the furnace begins to lower the sample temperature to room temperature. When the sample has cooled, remove the sample tube when the analysis is complete.



Use the cotton gloves provided in the accessory kit to remove the sample tube before it has cooled. Rubber gloves may be used to handle the sample tube when it has cooled.

8. Verify that the peak temperatures are consistent with specifications in the *Reference Material* booklet.

## PERFORM A TEMPERATURE PROGRAMMED OXIDATION ANALYSIS



This topic provides an example of performing a TPO on  $WO_3$ . Because TPR is often used as the preparation for TPO, the TPR process was included in this example.

Temperature Programmed Oxidation (TPO) examines the extent to which a catalyst can be re-oxidized. Generally, TPO analyses are used to measure the degree of reduction of certain oxides.

Usually the sample is pretreated and the metal oxides are reduced to the base metal. Then the reactant gas is applied to the sample in pulses or (alternatively) as a steady stream. The analyzer measures the uptake of the reactant gas.

TPO is often performed after TPR is performed. When the TPR experiment concludes, the sample is returned to room temperature. Then, the analysis gas is changed to 2-5%  $O_2$  + He. This gas mixture is flowed after the sample at ambient temperature, then the temperature is ramped up to the same maximum temperature used for the preceding TPR analysis. The portion of the sample that had been reduced is re-oxidized, and the degree of reduction can be calculated.

If the TPR and TPO results are different, there are several possible causes: the sample material sintered such that only a surface oxide (and not a bulk oxide) is formed, or part of the sample was re-oxidized at room temperature while the TCD baseline was stabilizing.

### PREPARATION

<b>Pretreatment</b>	TPR
<b>Analysis</b>	2 to 5% oxygen/helium is flowed through the sample while temperature is ramped, beginning at ambient temperature.
<b>Cold Trap</b>	The cold trap does not need to be cooled during the reference material analysis. This may vary depending on application or analysis method. The cold trap tube should remain on the instrument. Including the cold trap in the flow path is optional.



Before performing an analysis, ensure the sample and analyzer are adequately prepared using the instructions in [Prepare for Analysis on page 6 - 3](#).

## PROCEDURE

1. Obtain the sample mass, then install the loaded sample tube and thermocouple on the analyzer. Raise the furnace around the sample tube.
2. Create a new sample file for this analysis. In the *Analysis Conditions* drop-down box, select the analysis conditions file and modify the steps as needed depending on the analysis to be performed.
4. On the *Report Options* tab, select the required report options for the analysis.
5. Save and close the file.
6. Go to **Unit [n] > Sample Analysis** and select the sample file saved in the previous step.
7. Click **Start** to start the analysis.
8. When prompted, select the calibrations associated with each experiment in the sample file (if applicable). For this example, select *None*. Calibration files can also be associated with a sample file after analysis using the *Peak Editor*. See [Peak Editor for Dynamic Analysis on page 7 - 11](#).



When the analysis ends, the furnace begins to lower the sample to room temperature. When the sample has cooled, remove the sample tube when the analysis is complete.

9. As the temperature increases, the sample is oxidized, and the application calculates the volume of oxygen taken up.
10. Allow the TCD signal to return to the initial baseline after the peak has been displayed.



Use the cotton gloves provided in the accessory kit to remove the sample tube before it has cooled. Rubber gloves may be used to handle the sample tube when it has cooled.

11. When the displayed sample temperature reaches the ambient temperature, open the furnace. Using gloves, remove the sample tube.

## PERFORM A TEMPERATURE PROGRAMMED REDUCTION ANALYSIS



This topic provides an example of how to perform a TPR analysis of silver oxide. Silver Oxide Reference Material can be ordered from Micromeritics (part number 004-16836-00).

Temperature Programmed Reduction (TPR) determines the quantity of reducing gas consumed during reaction with the catalyst and reveals the temperature at which reduction occurs. For the reference material analysis, hydrogen (5-10%) is the reducing gas. The following method provides an overview of the steps to successfully analyze the reference material only. For other materials, the sample mass, gas flow rate, temperature ramp, active gas, or active gas concentration may need to be changed. Refer to the reference material booklet and the reference material bottle label for the expected results.

### PREPARATION

<b>Pretreatment</b>	Not required for reference material.
<b>Analysis</b>	Flow 5-10% hydrogen/argon or 5-10% hydrogen/ nitrogen, while ramping the temperature. The analyzer records hydrogen consumption as a function of temperature. Nitrogen is sometimes used because it may be more economical than argon.
<b>Cold Trap</b>	A cooling of the cold trap is generally needed for the entire analysis. A mixture of liquid nitrogen or dry ice and acetone or isopropanol provides the best performance. A cooling bath with dry ice will last longer than a cooling bath with liquid nitrogen. See step 2 in the following procedure for preparation details.



Before performing an analysis, ensure the sample and analyzer are adequately prepared using the instructions in [Prepare for Analysis on page 6 - 3](#).

### PROCEDURE

1. Obtain the sample mass (see the *Silver Oxide Reference Material* booklet for proper mass range), then install the loaded sample tube and thermocouple on the analyzer. Raise the furnace around the sample tube and install the insulator disc and shield.
2. Place the isopropyl alcohol in a cold trap dewar and slowly pour LN<sub>2</sub> into the dewar while stirring the mixture. Continue to add and stir until the mixture becomes slushy. The mixture must be capable of achieving a temperature of approximately -90 °C.
3. Place the cold trap dewar filled with cooling fluid around the cold trap tube. Ensure that the cold trap dewar contains enough coolant to immerse the lower part of the cold trap tube.



Extreme caution should be used when mixing the IPA/LN<sub>2</sub>. See [Dewar Precautions on page 6 - 1](#).

4. Create a new sample file for this analysis. (See [Sample Files on page 3 - 1](#).) In the *Analysis Conditions* drop-down box, select *AgO TPR with H2Ar*.
4. On the *Report Options* tab, select *Summary* in the *Selected Reports* list box.
5. Save and close the file.
6. Go to **Unit [n] > Sample Analysis** and select the sample file saved in the previous step.
7. Click **Start** to start the analysis.
8. When prompted, select the calibrations associated with each experiment in the sample file (if applicable). For this example, select *None*. Calibration files can also be associated with a sample file after analysis using the *Peak Editor*. See [Peak Editor for Dynamic Analysis on page 7 - 11](#).

As the temperature increases, the silver oxide is reduced, the water produced by the reaction is collected in the cold trap, and the amount of hydrogen consumed is detected.

A hydrogen consumption peak which corresponds to the reduction capacity of silver oxide is displayed. Refer to the *Silver Oxide Reference Material* booklet for the maximum peak temperature.

9. When the analysis concludes, remove the sample tube.



Use the cotton gloves provided in the accessory kit to remove the sample tube before it has cooled. Rubber gloves may be used to handle the sample tube when it has cooled.

10. Close the analysis window and open the sample file with the TPR data.
11. Click the drop-down at the bottom of the window and select the peak editor for the TPR experiment. See [Peak Editor for Dynamic Analysis on page 7 - 11](#).
12. Apply calibration file. See [Peak Editor for Dynamic Analysis on page 7 - 11](#).
13. Verify that the peak temperature and consumption are consistent with specifications in the *Silver Oxide Reference Material* booklet.

## PERFORM A VAPOR ANALYSIS



This option is for physical adsorption or chemical adsorption only.

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



After running a chemical adsorption analysis, port 2 can become contaminated with use. Clean the system by evacuating all three ports that were fitted with empty physical adsorption tubes until the system vacuum level is at  $9 \times 10^{-6}$  mmHg or lower.

A vapor analysis requires that a vapor source container be installed. See [Vapor Source Container Installation on page 11-11](#).



Micromeritics offers two methods of installing a vapor source container - one method for analyzers with a shelf support and another method for analyzers without a shelf support.

1. On the *Adsorptive Properties* window, select *Vapor source* in the *Dosing Method* group box. If running the vapor analysis on Port 3, select *From sample port 3* and proceed to Step 3.
2. Set the *Vapor Source Temperature* to the correct value if *Vapor Source* is selected. If sample port 3 is selected, the analysis bath temperature is assumed.
3. Select *Controlled by instrument using heating mantle* if the vapor source is to be automatically heated to this temperature.



If this option is selected for a chemical adsorption analysis, sample temperature control must be set to *Accessory temperature control*. See [Temperature \[button\] on page 4 - 11](#).

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## 7 ABOUT REPORTS

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

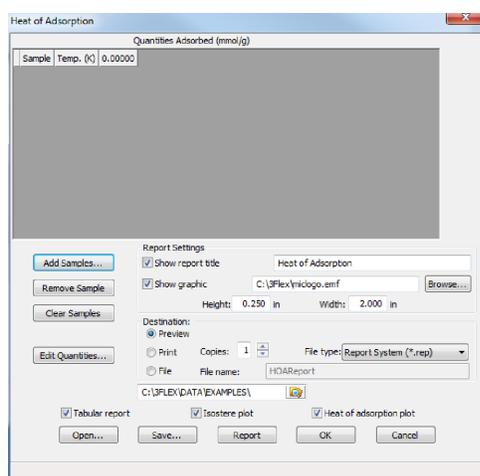
**Reports > Close Reports**

**Reports > Start Report**

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

### HEAT OF ADSORPTION REPORT

**Reports > Heat of Adsorption**

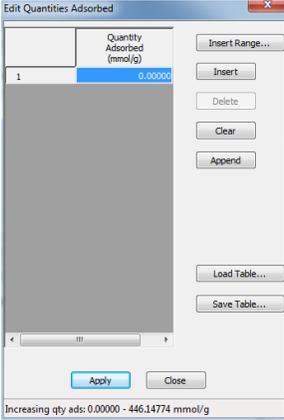


Use to select sample files, define quantities, and generate a *Heat of Adsorption* report. The isosteric heat of adsorption is an important parameter for characterizing the surface heterogeneity and for providing information about the adsorbent and the adsorption capacity. Multiple adsorption isotherms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

#### Heat of Adsorption Fields and Buttons Table

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

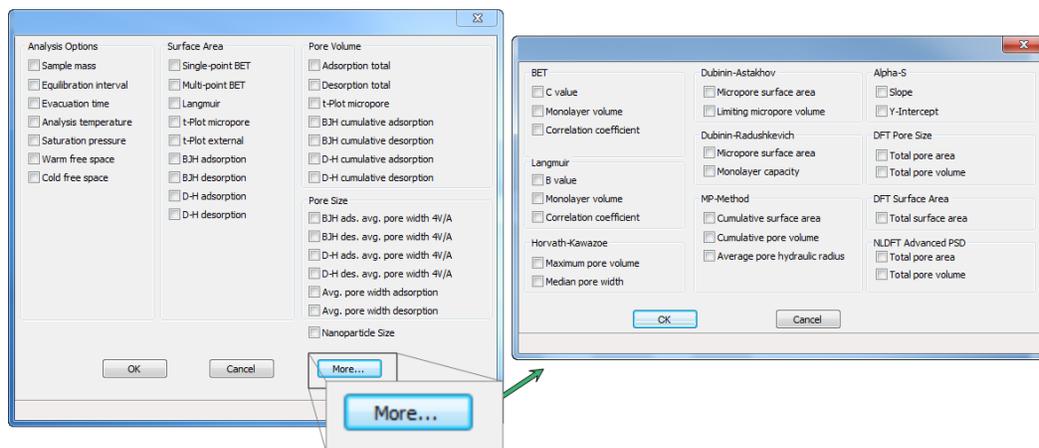
## Heat of Adsorption Fields and Buttons Table (continued)

Field or Button	Description
	 <p><b>Insert Range.</b> Click to specify the starting and ending quantities adsorbed and number of points to insert.</p> <p><b>Load Table.</b> Imports values from another file.</p> <p><b>Save Table.</b> Saves the current table as a .QNT file.</p> <p><b>Apply.</b> Applies all table changes.</p>
<b>Heat of adsorption plot</b> [selection]	Generates the <i>Heat of Adsorption</i> data in a graphical format.
<b>Isostere plot</b> [selection]	Generates a graph showing quantities of gas adsorbed versus the temperature.
<b>Remove Sample</b> [button]	Removes the selected sample from the list.
<b>Show graphic</b> [checkbox]	Use to show a graphic on the report header.  <b>Height / Width.</b> Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
<b>Show report title</b> [checkbox]	Select and enter a report title to appear on the report header.
<b>Tabular report</b> [checkbox]	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## SPC REPORT FOR PHYSICAL ADSORPTION

### Reports > SPC Report Options

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

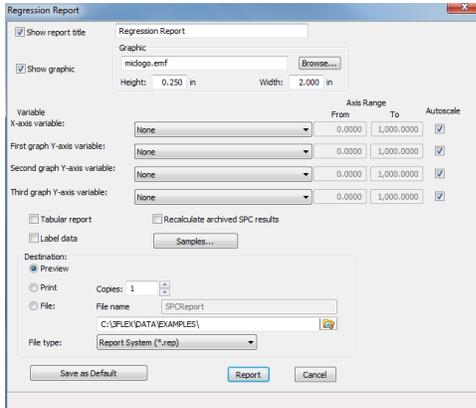


The selected items display as graph variable selections in **Reports > Regression Report** and graph selections in **Reports > Control Chart**. If additional report options are required, click [More](#).

## REGRESSION REPORT FOR PHYSICAL ADSORPTION

### Reports > Regression Report

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



Regression Report Fields and Buttons Table

Field or Button	Description
<b>Autoscale</b> [ <i>check box</i> ]	When enabled, allows the x- and y-axes to be scaled automatically.
<b>Axis Range</b> [ <i>text box</i> ]	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if <i>Autoscale</i> is selected.
<b>Label data</b> [ <i>check box</i> ]	Use to label the points on the plot to correspond with the values in the sample files.
<b>Recalculate archived SPC results</b> [ <i>check box</i> ]	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.  <div style="border: 1px solid green; padding: 5px; margin: 5px 0;">  <p>If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application.</p> </div> <p>When this option is selected, this message displays:</p> <div style="background-color: #e0e0e0; padding: 5px; margin: 5px 0; text-align: center;"> <p><b>Saving the recalculated SPC data may render some files</b></p> </div>

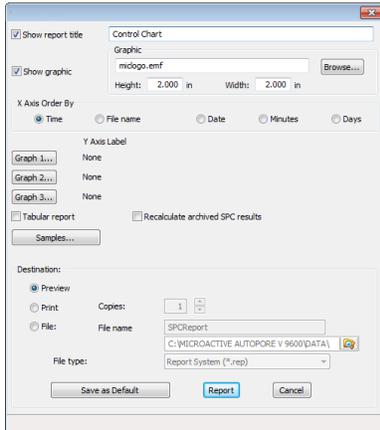
## Regression Report Fields and Buttons Table (continued)

Field or Button	Description
	<p><b>unreadable by the original application. Saving the SPC data speeds up future SPC reports.</b></p> <hr/>  <p>If <i>Do not show me this message again</i> is selected, the message cannot be redisplayed without Micromeritics assistance.</p> <hr/> <p>The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.</p>
<b>Samples</b> [ <i>button</i> ]	Select additional sample files to add to the report.
<b>Save as Default</b> [ <i>button</i> ]	Click to save selected report options as default report settings.
<b>Show graphic</b> [ <i>check box</i> ]	Use to show a graphic on the report header.  <b>Height / Width.</b> Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
<b>Show report title</b> [ <i>check box</i> ]	Select and enter a report title to appear on the report header.
<b>Tabular report</b> [ <i>check box</i> ]	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
<b>X- and Y-Axis Variable</b> [ <i>drop-down box</i> ]	Use to designate the x- and y-axes variables. The variables in the drop-down lists are those selected in the <b>Reports &gt; SPC Report Options</b> 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 <a href="#">Common Fields and Buttons on page 2 - 4</a> .

## CONTROL CHART REPORT FOR PHYSICAL ADSORPTION

### Reports > Control Chart

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



### Control Chart Fields and Buttons Table

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

## Control Chart Fields and Buttons Table (continued)

Field or Button	Description
<b>Recalculate archived SPC results</b> [ <i>checkbox</i> ]	<p>Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report.</p> <hr/> <div style="display: flex; align-items: center;">  <p>If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application.</p> </div> <hr/> <p>When this option is selected, this message displays:</p> <div style="border: 1px solid gray; padding: 5px; margin: 10px 0;"> <p><b>Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.</b></p> </div> <hr/> <div style="display: flex; align-items: center;">  <p>If <i>Do not show me this message again</i> is selected, the message cannot be redisplayed without Micromeritics assistance.</p> </div> <hr/> <p>The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.</p>
<b>Report</b> [ <i>button</i> ]	Generates the report.
<b>Samples</b> [ <i>button</i> ]	Select additional sample files to add to the report.
<b>Show graphic</b> [ <i>checkbox</i> ]	Use to show a graphic on the report header.  <b>Height / Width.</b> Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
<b>Show report title</b> [ <i>checkbox</i> ]	Select and enter a report title to appear on the report header.
<b>Tabular report</b> [ <i>checkbox</i> ]	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.

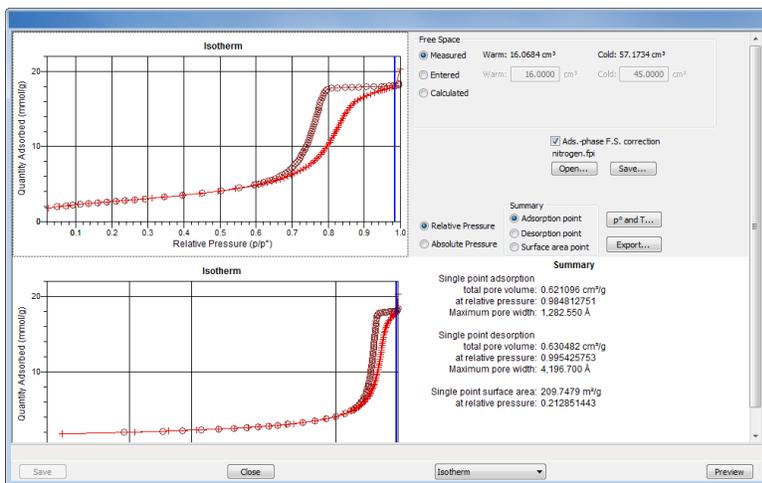
## Control Chart Fields and Buttons Table (continued)

Field or Button	Description
<b>X Axis Order by</b> [group box]	Select the order in which x-axis statistics are placed. Sort by:  <b>Time.</b> Time the files were analyzed.  <b>File name.</b> Alphanumeric order.  <b>Date.</b> Date the files were analyzed.  <b>Minutes.</b> Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file.  <b>Days.</b> 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 <a href="#">Common Fields and Buttons on page 2 - 4</a> .

## INTERACTIVE REPORTS

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

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



- To view the plots in either relative or absolute pressure, select either the *Relative Pressure* or *Absolute Pressure* option.
- To view the reports selected for generation during the analysis, click **Preview**.
- 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.
- 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
- Click **Save**.

## ***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.

Tutorials are available for some reports. These reports are available in online help and on the internet. For a list of available links, go to [3Flex Links on page iv](#).

## ***PEAK EDITOR FOR DYNAMIC ANALYSIS***

The *Peak Editor* feature provides the viewing and editing of up to 16 dynamic analysis experiments. The *Peak Editor* options are accessed by selecting a sample file that contains at least one experiment with signal data from the drop-down menu at the bottom of the report window. Peaks can be defined, edited, or deleted.

Peaks are defined by a baseline. If the **Find All Peaks** button is clicked (enabled when *Edit Peak* is selected), the *Peak Editor* will define baselines for all positive peaks detected according to the *Integration* window accessed from the **Integration** button. The baseline can be manually defined by double left clicking in the signal graph on the starting baseline point (this places the Peak Editor into baseline creation mode). The ending baseline point is then defined by left clicking in the signal graph on the ending baseline point. Baseline creation mode can be exited by right clicking in the signal graph.

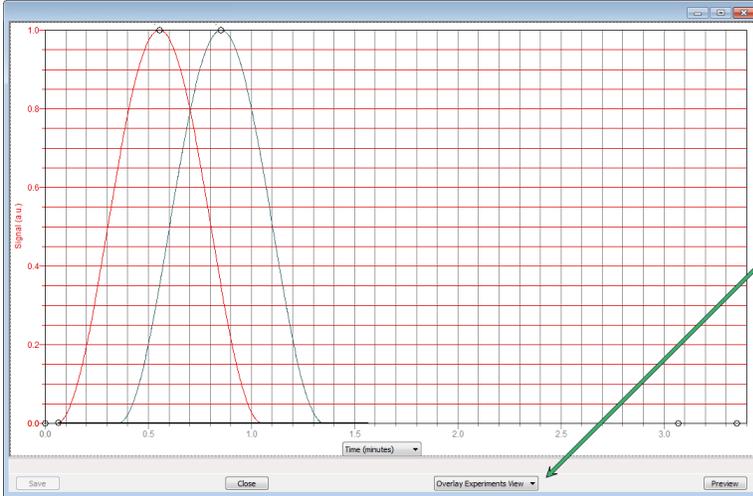
## ***PEAK EDITOR VIEW***

See [Open a Sample File on page 3 - 5](#)

When a sample file with a *Completed* status is opened, three views are available:

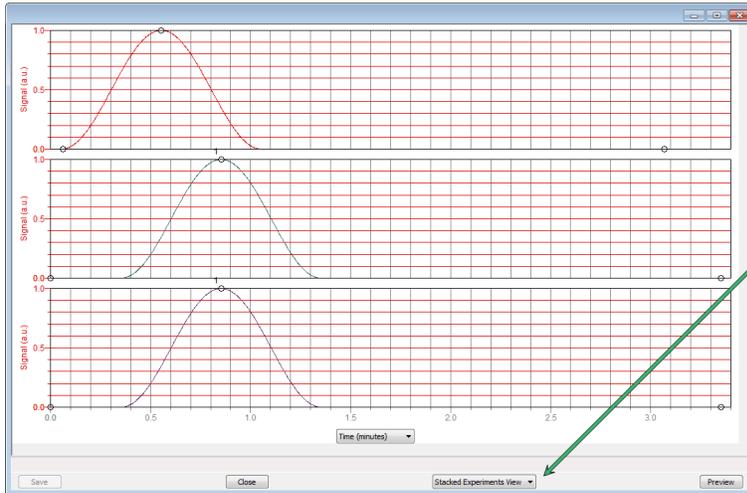
- Overlay Experiments View
- Stacked Experiments View
- Peak Editor - [*sample file name*]

To change the view, select the view from the drop-down list at the bottom of the graph window. Only the *Peak Editor* view allows editing of the experiment.



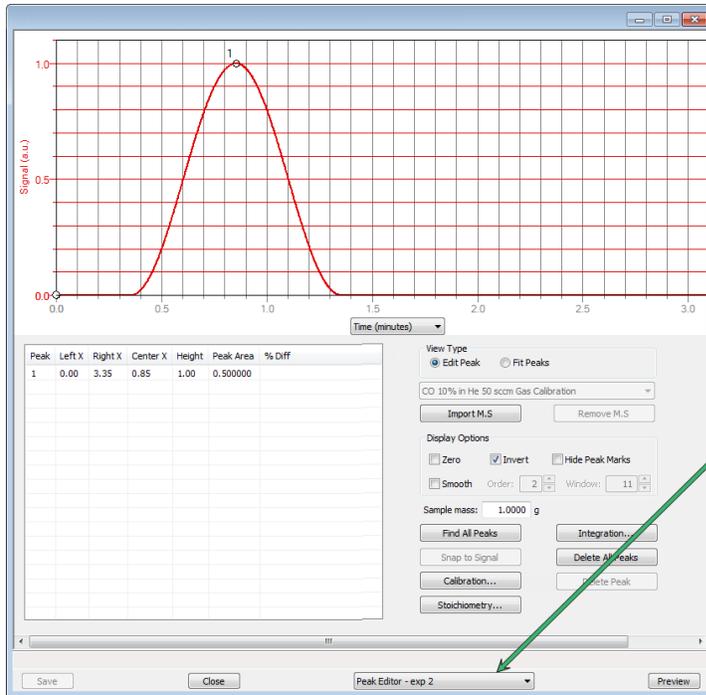
Select the view from the drop-down list

**Overlay Experiments View**



Select the view from the drop-down list

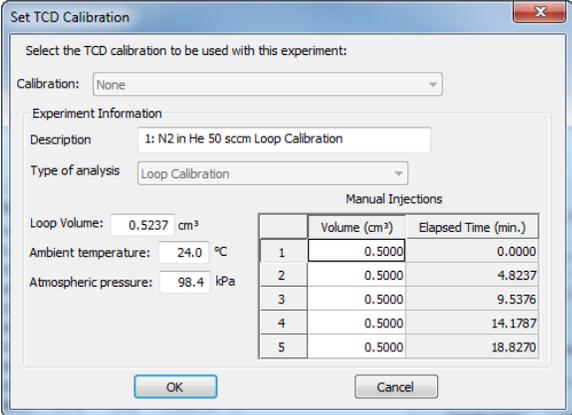
**Stacked Experiments View**



Select the view from the drop-down list

**Peak Editor View**

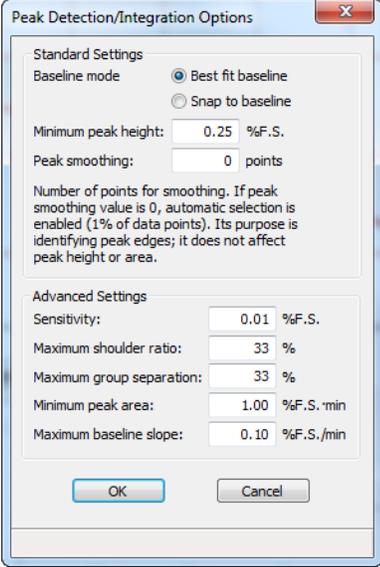
## Peak Editor Fields and Buttons Table

Field or Button	Description																		
<b>Calibration</b> [ <i>button</i> ]  <b>Delete All</b> [ <i>button</i> ] <b>Delete All Peaks</b> [ <i>button</i> ] <b>Delete Peak</b> [ <i>button</i> ]	 <p><b>Calibration.</b> Select a previously defined calibration file. If the experiment carrier gas and the calibration carrier gas differ, a warning displays. The system also compares the experiment flow rate and calibration flow rate. If they differ, a warning displays.</p> <p><b>Experiment Information.</b></p> <ul style="list-style-type: none"> <li>• <b>Description.</b> Enter a description of the current experiment.</li> <li>• <b>Type of analysis.</b> Select the analysis type that most closely describes the current experiment. This will affect what is reported as part of the Summary report data and the available reports.</li> </ul> <p><b>Ambient temperature.</b> Displays the ambient temperature entry from the <i>Options &gt; Environmental Defaults</i> setting. This field may be edited, if necessary.</p> <p><b>Atmospheric pressure.</b> Displays the atmospheric pressure entry from the <i>Options &gt; Environmental Defaults</i> setting. This field may be edited, if necessary.</p> <p><b>Manual Injections.</b> Displays the injection settings entered during analysis. This table may be edited, if necessary.</p> <table border="1" data-bbox="816 512 1101 657"> <thead> <tr> <th></th> <th>Volume (cm<sup>3</sup>)</th> <th>Elapsed Time (min.)</th> </tr> </thead> <tbody> <tr> <td>1</td> <td>0.5000</td> <td>0.0000</td> </tr> <tr> <td>2</td> <td>0.5000</td> <td>4.8237</td> </tr> <tr> <td>3</td> <td>0.5000</td> <td>9.5376</td> </tr> <tr> <td>4</td> <td>0.5000</td> <td>14.1787</td> </tr> <tr> <td>5</td> <td>0.5000</td> <td>18.8270</td> </tr> </tbody> </table>		Volume (cm <sup>3</sup> )	Elapsed Time (min.)	1	0.5000	0.0000	2	0.5000	4.8237	3	0.5000	9.5376	4	0.5000	14.1787	5	0.5000	18.8270
	Volume (cm <sup>3</sup> )	Elapsed Time (min.)																	
1	0.5000	0.0000																	
2	0.5000	4.8237																	
3	0.5000	9.5376																	
4	0.5000	14.1787																	
5	0.5000	18.8270																	
<b>Delete All</b> [ <i>button</i> ] <b>Delete All Peaks</b> [ <i>button</i> ] <b>Delete Peak</b> [ <i>button</i> ]	<p>Clears all peaks from the table and removes all markings from the peaks.</p> <p>To delete a single peak, select the peak from the peak table or left click the peak to enable the <b>Delete Peak</b> button.</p> <p><b>Delete All</b> is available only when the <i>Fit Peaks</i> selection is enabled.</p>																		

## Peak Editor Fields and Buttons Table (continued)

Field or Button	Description
	<p><b>Delete All Peaks</b> is available only when the <i>Edit Peak</i> selection is enabled.</p> <p><b>Delete Peak</b> is available only when the <i>Fit Peak</i> selection is enabled.</p>
<b>Delete Baseline</b> [button]	<p>Removes the baseline from the graph.</p> <p>Available only when the <i>Fit Peak</i> selection is enabled.</p>
<b>Display Options</b> [group box]	<p>Allows the user to change how the data are displayed.</p> <p><b>Zero.</b> Select to zero the signal (starting baseline).</p> <p><b>Invert.</b> Select to invert the signal (peak).</p> <p><b>Hide Peak Marks.</b> Select to hide all marks from peaks.</p> <p><b>Smooth.</b> Select to smooth the signal on the display.</p> <ul style="list-style-type: none"> <li>• <b>Order</b> and <b>Window.</b> Enabled when the <i>Smooth</i> checkbox is selected. The smoothing process uses the Savitzky-Golay filter to fit a polynomial order <math>n</math> into size of the specified window <math>[m]</math>.</li> </ul>
<b>Find All Peaks</b> [button]	<p>Defines baselines for all positive peaks detected according to the <i>Integration</i> window, accessed via the <b>Integration</b> button.</p> <p><b>Find All Peaks</b> automatically detects the peaks and draws the baseline for detection. Place the cursor over one of the baseline end points and double left-click to grab the baseline. Move the cursor to the new position and right click.</p> <p>Available only when the <i>Edit Peak</i> selection is enabled.</p>
<b>Import M.S</b> [button] <b>Remove M.S</b> [button]	<p>Mass spectrometer data can be imported and overlaid in the <i>Overlay Experiment</i> view. In the <i>Peak Editor</i> view, it is saved as a separate signal.</p> <p><b>Import M.S.</b> Click to import mass spectrometer data. In the bottom right-hand corner of the pop-up window, select the type of mass spectrometer file to import (Quadera, Quadstar, MKS, or TAMS). Select the file, then click <b>Open</b> to import the signals.</p> <p>A popup window prompts the user to sync the temperature data with the current experiment temperature data.</p> <p><b>Remove M.S.</b> Click to remove all previously imported mass spectrometer signals from the current experiment.</p>

## Peak Editor Fields and Buttons Table (continued)

Field or Button	Description
<b>Import standard peaks</b> [button]	Imports the saved peak parameters from the <i>Peak Edit</i> window and uses them for the initial estimates for the peak set.  Available only when the <i>Fit Peaks</i> selection is enabled.
<b>Integration Options</b> [button]	 <p>See <a href="#">Peak Detection / Integration Options on page G - 1</a>.</p> Available only when the <i>Edit Peak</i> selection is enabled.
<b>Optimize Peak Fit</b> [button]	Modifies the peak parameters of the current peak to minimize the residual.  Available only when the <i>Fit Peaks</i> selection is enabled.
<b>Peak fit model</b> [button]	Select the peak shape.  Available only when the <i>Fit Peaks</i> selection is enabled.  <b>Gaussian.</b> Standard Gaussian curve.  <b>Log Normal Skewed.</b> A 4-parameter, log-normal shape that allows for skewed peaks. This is the default peak.
<b>Sample mass</b> [text box]	The sample mass field is auto-filled with the sample mass from the sample file being used.
<b>Snap to Signal</b> [button]	Places the selected baseline point on the signal curve..

## Peak Editor Fields and Buttons Table (continued)

Field or Button	Description
Stoichiometry [ <i>button</i> ]	See <a href="#">Active Metals for Chemical Adsorption on page 3 - 10</a> .
View Type [ <i>group box</i> ]	<p><b>Edit Peak.</b> Locates peaks via signal integration over a baseline.</p> <p><b>Fit Peaks.</b> Locates peaks via function fitting to minimize residual over a baseline.</p> <p>See <a href="#">Report Features and Shortcuts on page 7 - 23</a>.</p>
X-axis [ <i>drop-down box</i> ]	<p>Change the x-axis to display as time or temperature.</p> <p>This feature is available in <i>Stacked Experiment</i> view, <i>Overlay Experiment</i> view, and <i>Peak Editor</i> view. In <i>Peak Editor</i> view, the <i>Edit Peak</i> selection must be enabled.</p> <p><b>Time (minutes).</b> Select to display the x-axis as minutes.</p> <p><b>Temperature.</b> Select to display the x-axis as temperature.</p>
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

## ***EVALUATE REPORT RESULTS***

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

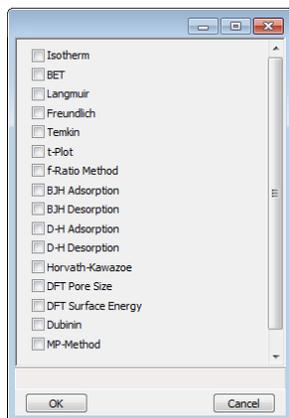
Regardless of the precautions exercised before the analysis, problems still may occur during the analysis, or as a result of using inappropriate parameters or even inappropriate methods. The analysis data should be inspected for evidence of experimental error. The traditional method of confirming the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

Analysis data can be evaluated by:

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

## VIEW THE VALIDATION REPORT

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



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

## INSPECT THE ISOTHERM PLOT

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

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

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

## ***EVALUATE THE ISOTHERM TABULAR DATA SET***

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

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

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

## ***REVIEW REDUCED DATA***

Isotherm data may be analyzed by any one of several reduction methods depending on instrument model and pressure range employed. The quality of the results depends on the quality of the isotherm, the congruity of the data reduction parameters with experimental conditions, the agreement of the theoretical model with the physical gas-solid system, and compliance to the pressure range over which the method is valid. Typically, results can be appraised by examining a few salient areas of the report as described in the following topics.<sup>3)</sup>

## ***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.

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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).

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

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<sup>1)</sup> 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  $\gamma$ , contact angle between solid and liquid phase  $\theta$ , molar volume of the adsorbate  $v_m$ , gas constant  $R$ , and sample temperature  $T$  are treated as one constant, the adsorbate property factor  $A$  expressed by:

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

using which, the Kelvin equation<sup>2)</sup> reduces to

$$\ln \frac{P^*}{P_0} = \frac{A}{r_m}$$

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<sup>3)</sup>, the Dubinin affinity coefficient or Astakhov exponent<sup>4)</sup>, 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.

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- 1) McClellan, A.L., and Harnsberger, H.F., Journal of Colloid and Interface Science, 23, 577 (1967).  
 2) Thomson, W., Phil. Mag. S., 42, 448 (1871).  
 3) Everett, D.H. and Powl, J.C., J. Chem Soc., Faraday Trans. 1, 72, 619 (1976).  
 4) Dubinin, M. and Radushkevich, L.V., Proc. Acad. Sci. USSR, 55, 331 (1947).
-

The t-Plot method plots quantity adsorbed ( $V_a$ ) against thickness ( $t$ ) derived from a thickness equation, and the Dubinin transform plots quantity adsorbed against  $\log(P/P_0)^n$ . 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.

## ***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.

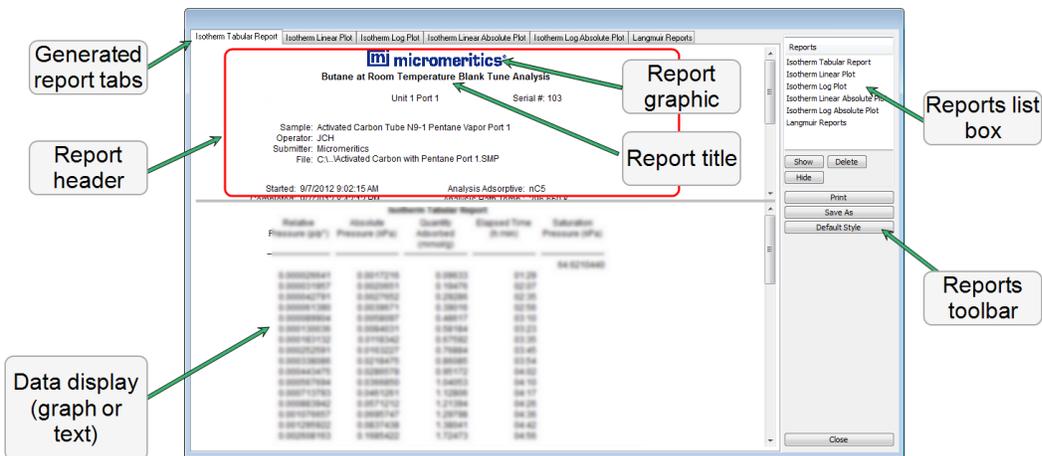
## ***DATA ANALYSES BY THE BJH METHOD***

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

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

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

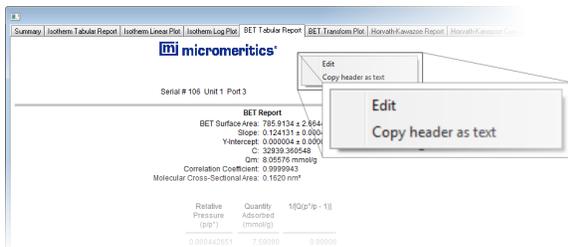


If configured, the report header can also contain a graphic and/or a title.

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

## REPORT HEADER SHORTCUTS

Display header shortcuts by right clicking in the report header.



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

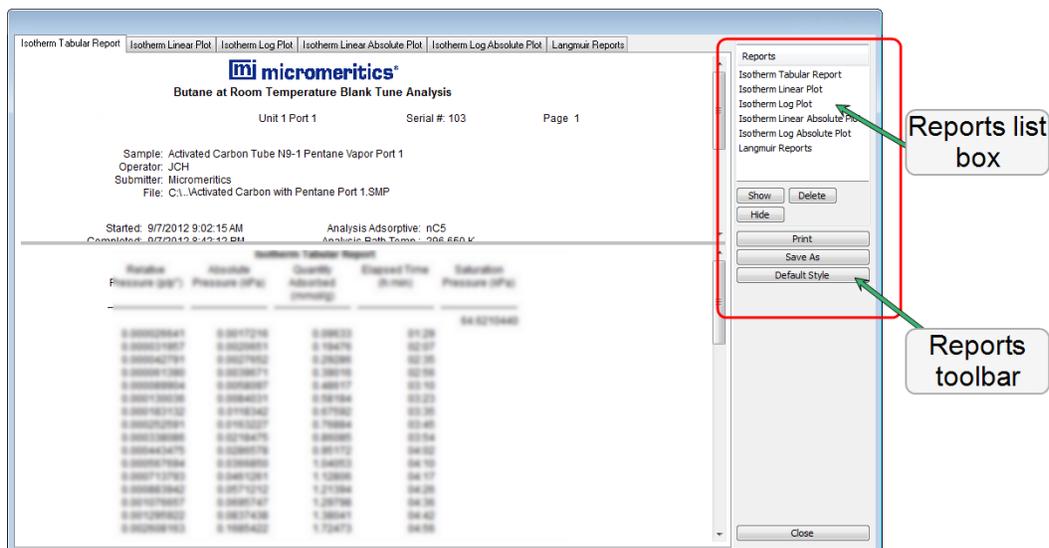
- Tabular and graphical reports contain sample and instrument statistics — such as analysis date, analysis time, analysis conditions, etc.
- The headers contain notes of sample file changes occurring after analysis.
- Summary report headers contain the same information in tabular and graphical reports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

### Report header Shortcut Field and Button Table

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

## REPORT TOOLBAR

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



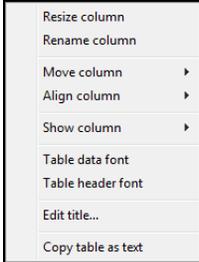
### Report Toolbar Fields and Buttons Table

Field or Button	Description
<b>Default Style</b> [button]	Specify default report parameters for fonts and curve properties.
<b>Delete</b> [button]	Deletes the selected report in the <i>Reports</i> list box. Deleted reports will have to be regenerated if deleted in error.
<b>Hide</b> [button]	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the <i>Reports</i> list box.
<b>Print</b> [button]	Displays the <i>Print</i> window for report output.
<b>Reports</b> [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 <b>Hide</b> button.
<b>Show</b> [button]	Displays the selected or hidden report in the <i>Reports</i> 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.



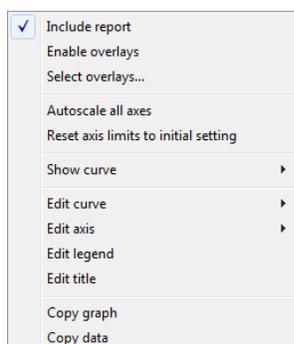
Tabular Reports Shortcut Options and Descriptions Table

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

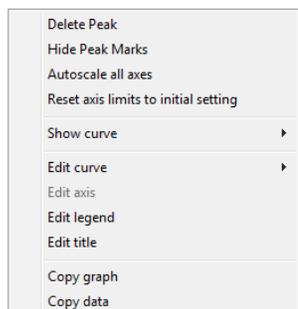
 For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 4](#).

## GRAPH FEATURES AND SHORTCUTS

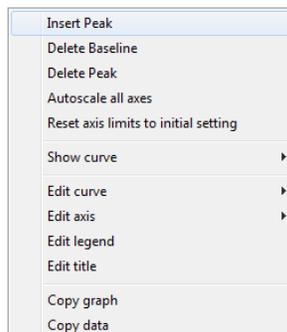
Display graph report shortcuts by right clicking in the body of the graph area.



**Standard Graph Shortcuts**

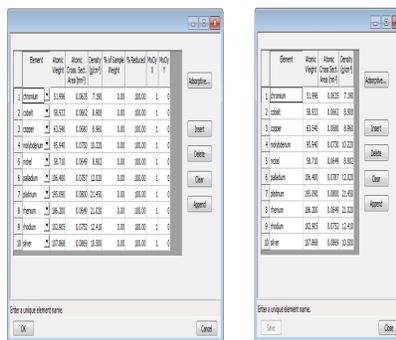


**Peak Editor Graph Shortcuts**

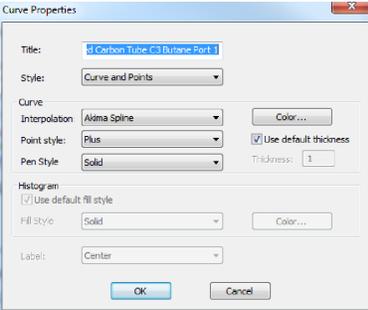


### Graph Shortcuts Options and Description Table

Field or Button	Description
<b>Autoscale all axes</b>	Returns the report to full view after using the zoom feature.
<b>Copy Data</b>	Copies the report data to the clipboard. It can then be pasted into other software programs as tab-delimited columns or copied as an overlay onto another graph.
<b>Copy Graph</b>	Copies the graph to the clipboard. It can then be pasted into other software programs.
<b>Delete Baseline <span style="background-color: blue; color: white; padding: 2px;">DA</span></b>	Deletes the baseline from the graph.
<b>Delete Peak <span style="background-color: blue; color: white; padding: 2px;">DA</span></b>	Deletes the selected peak from the table.
<b>Edit axis</b>	Use to edit the selected axis properties.



## Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
	<p><b>Autoscale minimum / maximum.</b> To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box.</p> <p><b>Degrees.</b>  Displays the axis in degrees.</p> <p><b>Grid lines.</b> Use to change how to display major / minor grid lines.</p> <p><b>Invert scale.</b> Use to invert the scale.</p> <p><b>Linear / Logarithmic.</b> Select the option to scale the graph as linear or logarithmic.</p> <p><b>Minutes.</b>  Displays the axis in minutes.</p> <p><b>Scale font.</b> Use to modify the font for the scale label. Deselect <i>Use default font</i> to enable font options.</p> <p><b>Signal (a.u.).</b>  Converts the signal from calibrated units to arbitrary units.</p> <p><b>Title.</b> Use to edit the selected axis label.</p> <p><b>Title font.</b> Use to modify the font for the selected axis label. Deselect <i>Use default font</i>. Select new font attributes for report data. Enable <i>Use default font</i> to reset default fonts.</p>
<b>Edit curve</b>	<p>Use to edit selected curve properties.</p>  <p><b>Color.</b> Click to change the curve color.</p> <ul style="list-style-type: none"> <li>• <b>Curve group box.</b> Use to change the interpolation, point style and pen style for the selected curve. These options are disabled</li> </ul>

## Graph Shortcuts Options and Description Table (continued)

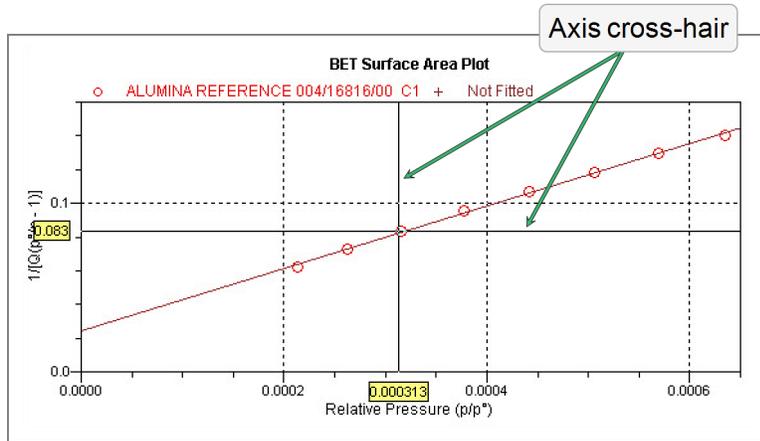
Field or Button	Description
	<p>if <i>Use default fill style</i> is selected in the <i>Histogram</i> group box.</p> <ul style="list-style-type: none"> <li><b>Histogram group box.</b> Enabled only if <i>Histogram</i> is selected in the <i>Style</i> drop-down list. Use to specify the type of fill, fill color and label position for the selected curve.</li> </ul> <p><b>Label.</b> Select where the graph point labels will display (left, right, center, etc.) on the SPC report.</p> <p><b>Style.</b> Use to select another style for the collected data curve.</p> <p><b>Title.</b> Use to change the title of the selected curve.</p> <p><b>Use default thickness.</b> Uses the default curve thickness. Deselect to enter a new thickness number in the <i>Thickness</i> text box.</p>
<b>Edit imported data</b> <b>P</b> <b>SC</b>	Used with pore distribution data reports only. Use to select ASCII text files for import onto the active graph.
<b>Edit legend</b>	Use to change the legend location and font. 
<b>Edit title</b>	Use to change the graph title and font.

## Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
<b>Enable Overlays</b> <b>P SC</b>	If overlays have been selected, this option displays (or hides) the overlays.
<b>Hide Peak Marks</b> <b>DA</b>	Select to remove all peak marks from the graph.
<b>Include report</b> <b>P SC</b>	When selected, places a checkmark to the left of the report in the <i>Select Reports</i> list box on the <i>Report Options</i> tab.
<b>Insert Peak</b> <b>DA</b>	Inserts a peak into the graph at the selected point .
<b>Reset axis limits to initial setting</b>	Removes the cross-hair and returns the graph back to the initial setting.
<b>Select overlays...</b> <b>P SC</b>	Displays the option to select files to overlay onto the active graph. To view the overlays, click <i>Enable Overlays</i> on the shortcut menu.
<b>Show curve</b>	Displays a list of all curves. Select the curve(s) to display.
	<b>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</b>

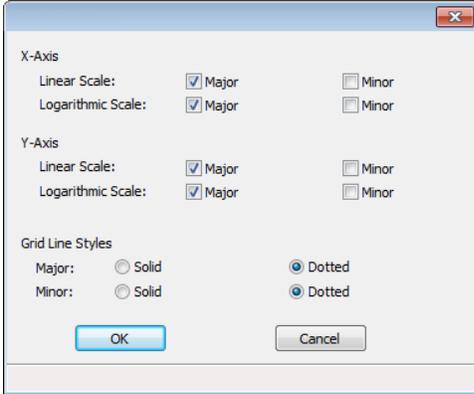
**Axis Cross-Hair**

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 Fields and Buttons Table**

Field or Button	Description
<b>Grid Line Styles</b> [selection]	Select if the major and/or minor grid lines should appear as solid or dotted lines.
<b>X-Axis / Y-Axis</b> [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.
 <b>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</b>	

**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.

## GRAPH AND SAMPLE OVERLAYS

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

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



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

## GENERATE PORE-SIZE DISTRIBUTION GRAPH OVERLAYS

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

Multiple graph overlays can only be generated for:

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

## ASCII TEXT FILE FORMAT RULES

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

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

Two examples of a header format:

My Title (A, cm<sup>3</sup>/g, incremental)

My Title (A, cm<sup>3</sup>/g, cumulative)

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

## Sample ASCII Text File

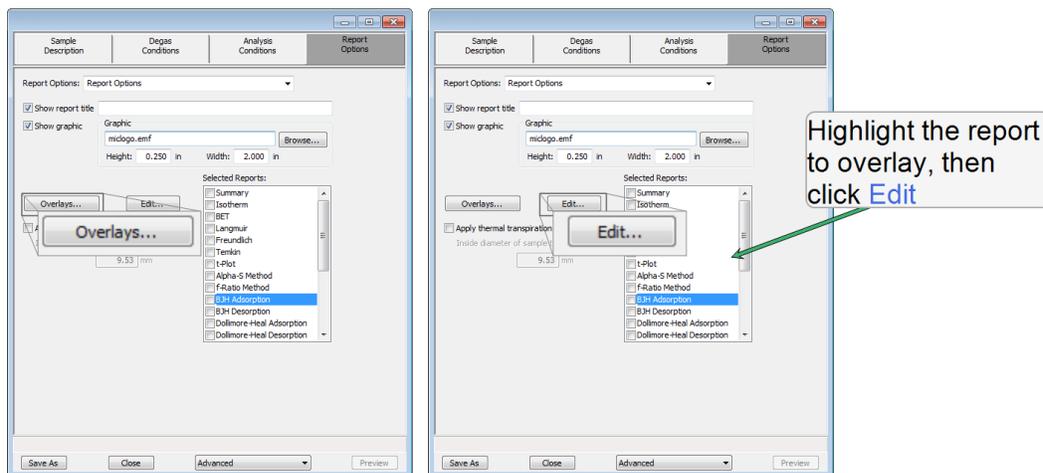
silica alumina bjh	(A, cm <sup>3</sup> /g, cumulative)
456.657	0.0133559
444.847	0.0546427
429.168	0.0869924
425.419	0.119721
419.629	0.132681
360.634	0.156611
340.859	0.197672
326.601	0.233092

### ***To IMPORT ASCII TEXT FILES TO GENERATE GRAPH OVERLAYS***

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

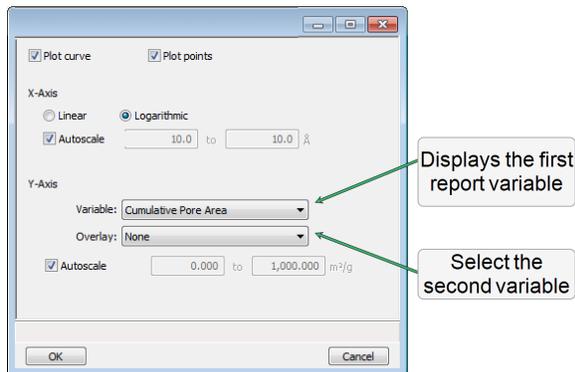
1. Go to **File > Open**. Select a sample file to overlay graphs onto other samples. Click **Open**.
2. Select *Advanced* from the drop-down list at the bottom of the window.
3. Select the *Report Options* tab, then click **Overlays** to browse for the .TXT file.

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



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

- Click the down arrow at the *Variable* field and select a variable to overlay. Click the down arrow of the *Overlay* field, then select *Imported Data*. Click **OK** to return to the *Report Options* window.

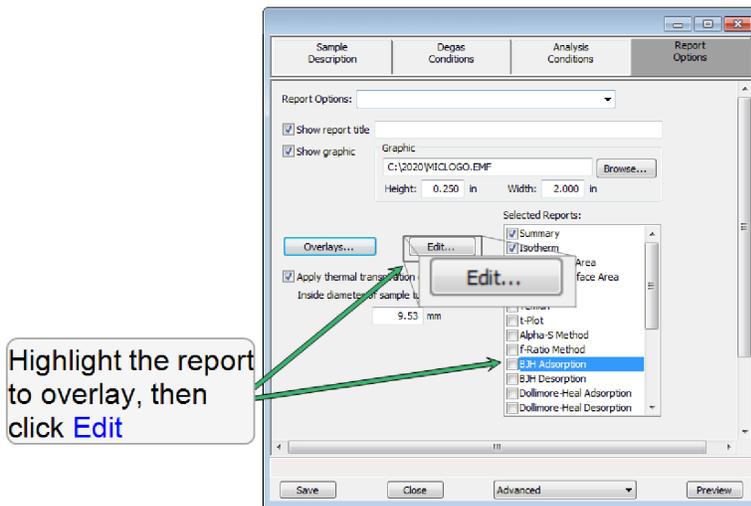


- Click **OK** again to return to the *Report Options* tab.
- Click **Save As** to save the selections.
- To view the report, click **Preview**.

## OVERLAY MULTIPLE PHYSICAL ADSORPTION SAMPLE FILES

To overlay the same type of graph on multiple samples:

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



### For Physical Adsorption Reports

If overlaying this type of report...	Then...
<ul style="list-style-type: none"> <li>• Isotherm</li> </ul>	<ol style="list-style-type: none"> <li>On the <i>Isotherm Report Options</i> window, select one or more plots in the <i>Selected Reports</i> group box, then click <b>Options</b> to the right of the selected plot.</li> <li>On the <i>Plot Options</i> window, select <i>Plot curve</i> and/or <i>Plot points</i> if they are to be included in the overlay. If the x- and/or y-axes are to be autoscaled, enable <i>Autoscale</i>; otherwise, enter the <i>From</i> and <i>To</i> points for the axes. Click <b>OK</b>.</li> <li>On the <i>Isotherm Report Options</i> window, in the <i>Plot Options</i> group box, select <i>Plot overlays</i>. Click <b>OK</b>.</li> <li>Continue to Step 5.</li> </ol>

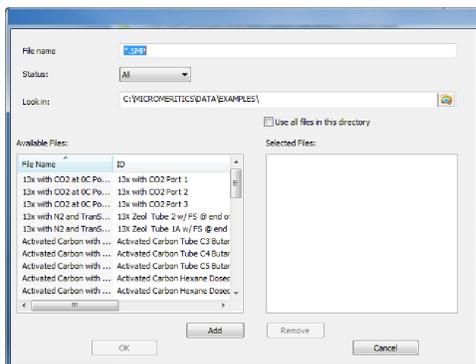
**For Physical Adsorption Reports (continued)**

If overlaying this type of report...	Then...
<ul style="list-style-type: none"> <li>• Alpha-S Method</li> <li>• BET Surface Area</li> <li>• <i>f</i>-Ratio Method</li> <li>• Freundlich</li> <li>• Langmuir Surface Area</li> <li>• <i>t</i>-plot</li> <li>• Temkin</li> </ul>	<ol style="list-style-type: none"> <li>On the pop-up window, select <i>Overlay samples</i>. Verify other fields. Click <b>OK</b>.</li> <li>Continue to Step 5.</li> </ol>
<ul style="list-style-type: none"> <li>• BJH</li> <li>• Dollimore-Heal</li> <li>• MP-Method</li> </ul>	<ol style="list-style-type: none"> <li>Select the report variable from the <i>Selected Reports</i> group box, then click <b>Edit</b>.</li> <li>Click the down arrow on the <i>Overlay</i> field, then select the <i>Samples</i> option. Verify other fields. Click <b>OK</b>.</li> <li>Click <b>OK</b> again.</li> </ol>

**For Chemical Adsorption Reports**

If overlaying this type of report...	Then...
<ul style="list-style-type: none"> <li>• Difference Method</li> <li>• Freundlich</li> <li>• Langmuir</li> <li>• Sinfelt Method</li> <li>• Temkin</li> </ul>	<ol style="list-style-type: none"> <li>On the pop-up window, select <i>Overlay samples</i>. Verify other fields. Click <b>OK</b>.</li> <li>Continue to Step 5.</li> </ol>

- On the *Report Options* tab, click **Overlays**.
- On the *Plot Overlay Sample Selection* window, move up to 25 files from the *Available Files* box to the *Selected Files* box:



- Click **OK**.
- To view the report, click **Preview**.

## **IMPORT ASCII PORE DISTRIBUTION DATA**

### **IMPORT AN ASCII TEXT FILE USING GRAPH SHORTCUTS**

1. Create an ASCII text file. See [Manually Enter Data on page 3 - 6](#).
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 - 6](#).
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**

**mi micromeritics®**

Unit 1 Port 2                      Serial #: 101                      Page 9

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

Started: 8/17/2012 8:44:16 PM Completed: 8/18/2012 6:33:42 PM Report Time: 8/30/2012 6:34:58 AM Sample Mass: 0.2555 g Cold Free Space: 57.0915 cm <sup>3</sup> Low Pressure Dose: None Automatic Degas: Yes	Analysis Adsorptive: N2 Analysis Bath Temp.: -196.058 °C Thermal Correction: No Warm Free Space: 16.0907 cm <sup>3</sup> Measured Equilibration Interval: 10 s Sample Density: 1.000 g/cm <sup>3</sup>
---	---

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

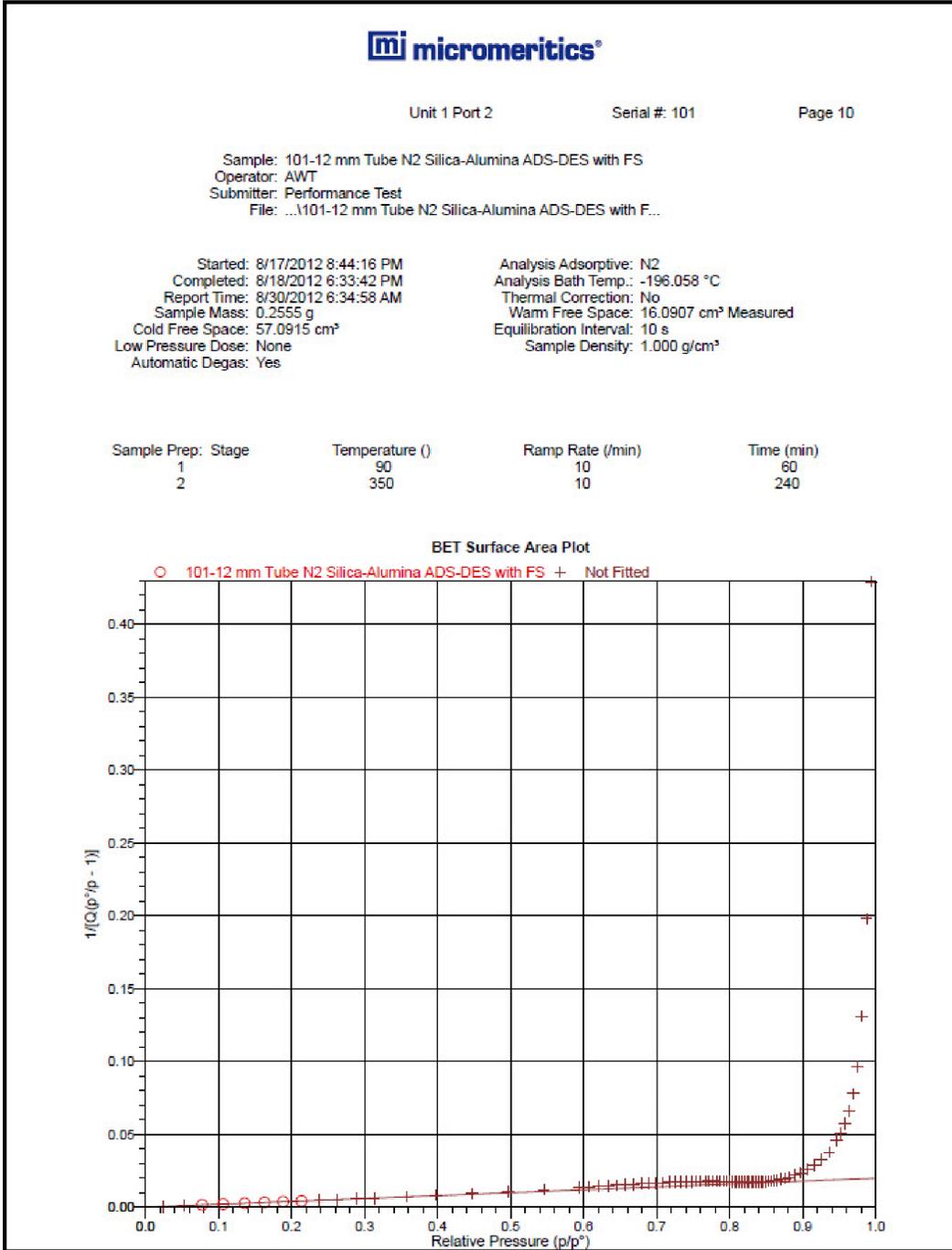
  

**BET Surface Area Report**

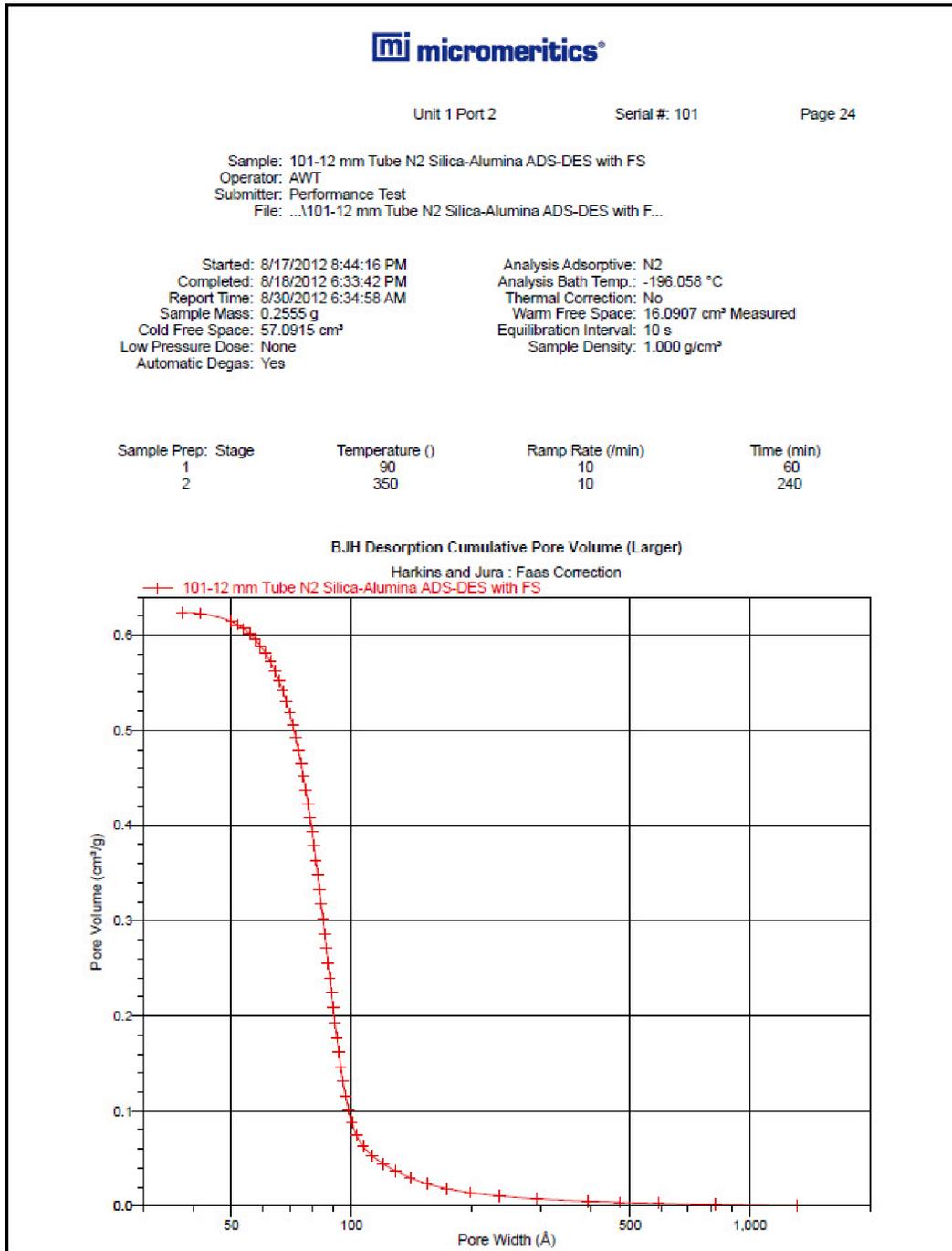
BET Surface Area: 217.1859 ± 0.2311 m<sup>2</sup>/g  
 Slope: 0.019839 ± 0.000021 g/cm<sup>3</sup> STP  
 Y-Intercept: 0.000205 ± 0.000003 g/cm<sup>3</sup> STP  
 C: 97.887751  
 Qm: 49.8911 cm<sup>3</sup>/g STP  
 Correlation Coefficient: 0.9999977  
 Molecular Cross-Sectional Area: 0.1620 nm<sup>2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

Relative Pressure (p/p <sup>0</sup> )	Quantity Adsorbed (cm <sup>3</sup> /g STP)	1/[Q(p <sup>0</sup> /p - 1)]
0.077824186	48.2830	0.001748
0.106574940	51.3624	0.002322
0.135877231	54.2113	0.002901
0.163237219	56.6908	0.003441
0.188595088	58.9330	0.003944
0.213852389	61.1303	0.004450

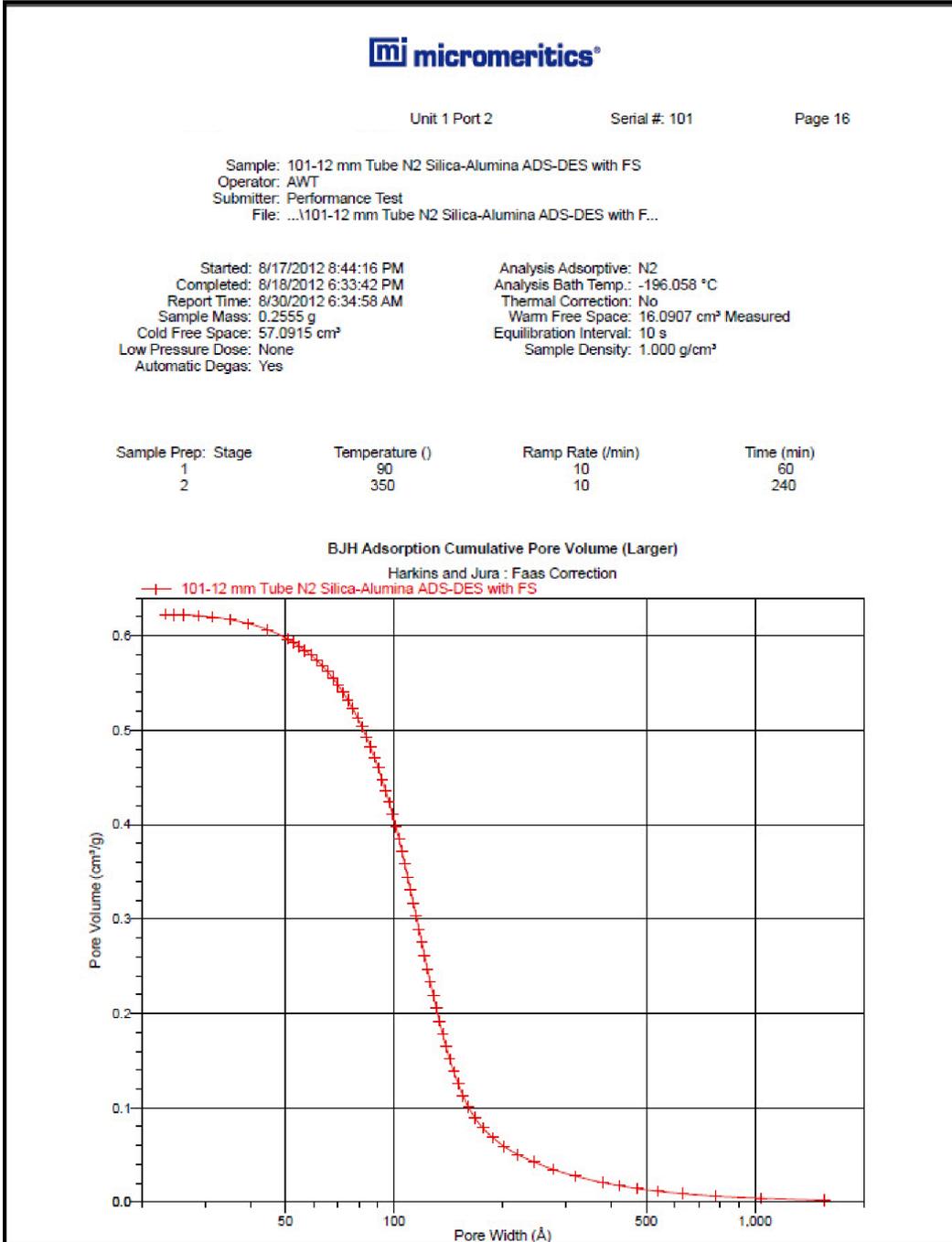
# BET SURFACE AREA PLOT



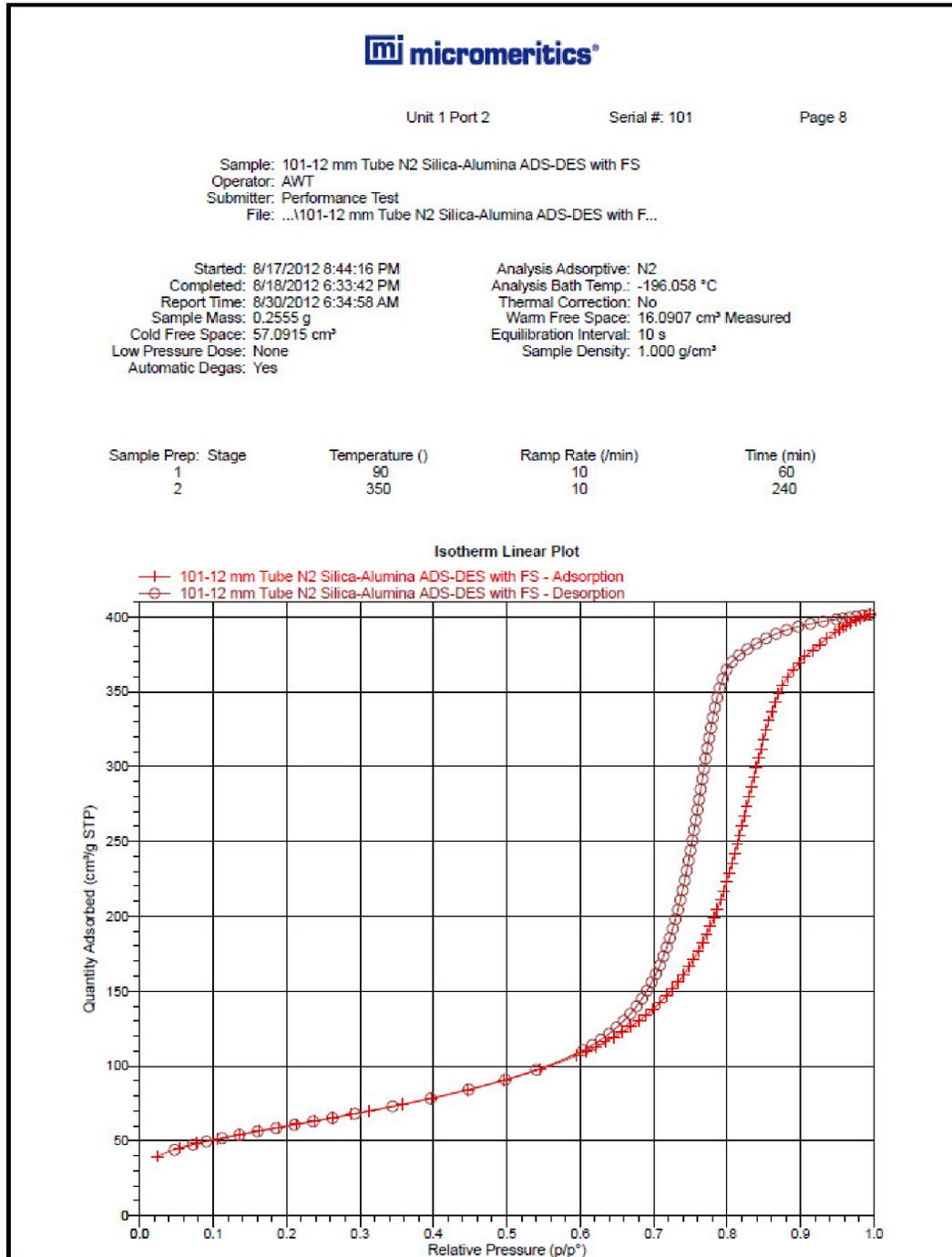
**BJH DESORPTION: CUMULATIVE PORE VOLUME**



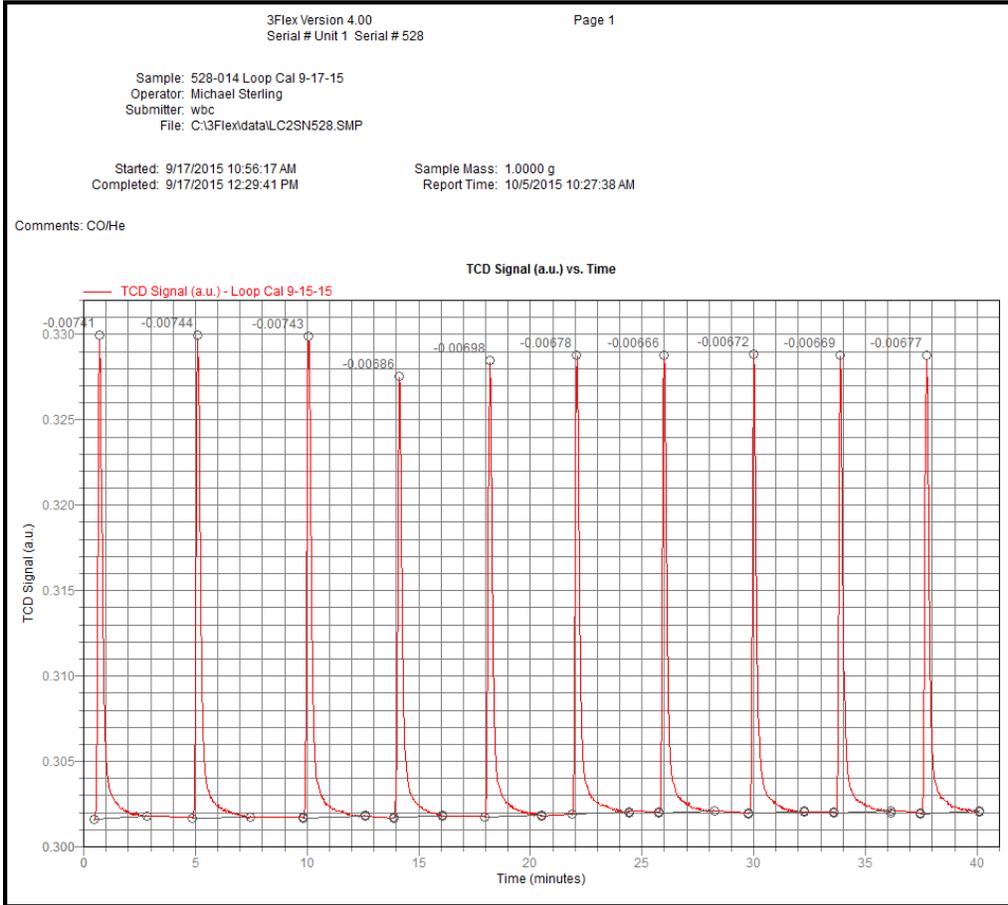
### BJH ADSORPTION: CUMULATIVE PORE VOLUME



## ISOTHERM LINEAR PLOT



# LOOP CALIBRATION



**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 cm <sup>3</sup> Low Pressure Dose: None Automatic Degas: Yes	Analysis Adsorptive: N2 Analysis Bath Temp.: -196.058 °C Thermal Correction: No Warm Free Space: 16.0907 cm <sup>3</sup> Measured Equilibration Interval: 10 s Sample Density: 1.000 g/cm <sup>3</sup>
---	---

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

**t-Plot Report**

Micropore Volume: 0.001429 cm<sup>3</sup>/g  
 Micropore Area: 7.9925 m<sup>2</sup>/g  
 External Surface Area: 209.1934 m<sup>2</sup>/g  
 Slope: 13.517568 ± 0.050021 cm<sup>3</sup>/g·Å STP  
 Y-Intercept: 0.923393 ± 0.218961 cm<sup>3</sup>/g STP  
 Correlation Coefficient: 0.999959  
 Surface Area Correction Factor: 1.000  
 Density Conversion Factor: 0.0015476  
 Total Surface Area (BET): 217.1859 m<sup>2</sup>/g  
 Thickness Range: 3.5000 Å to 5.0000 Å  
 Thickness Equation: Harkins and Jura

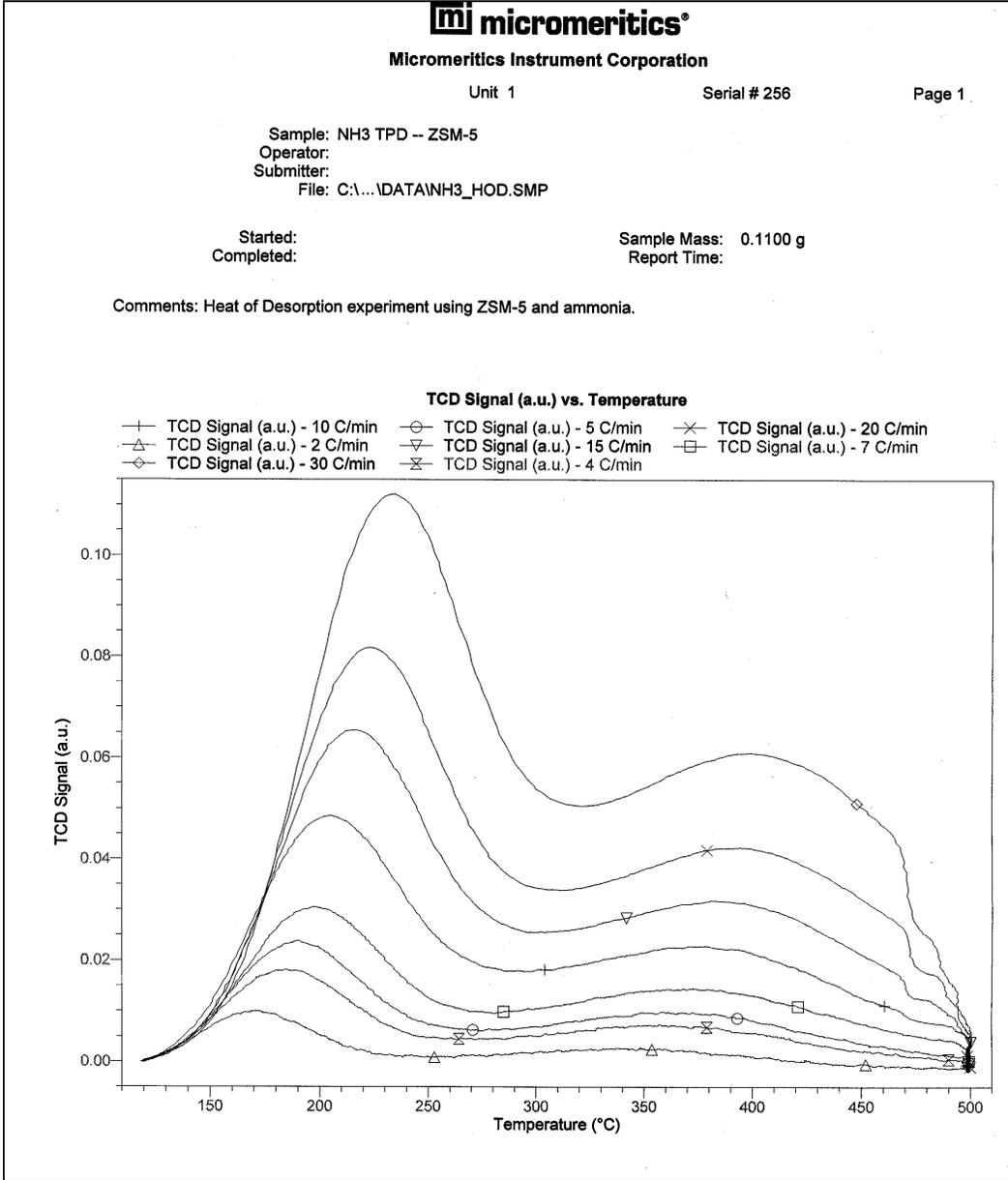
**Thickness Curve**

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

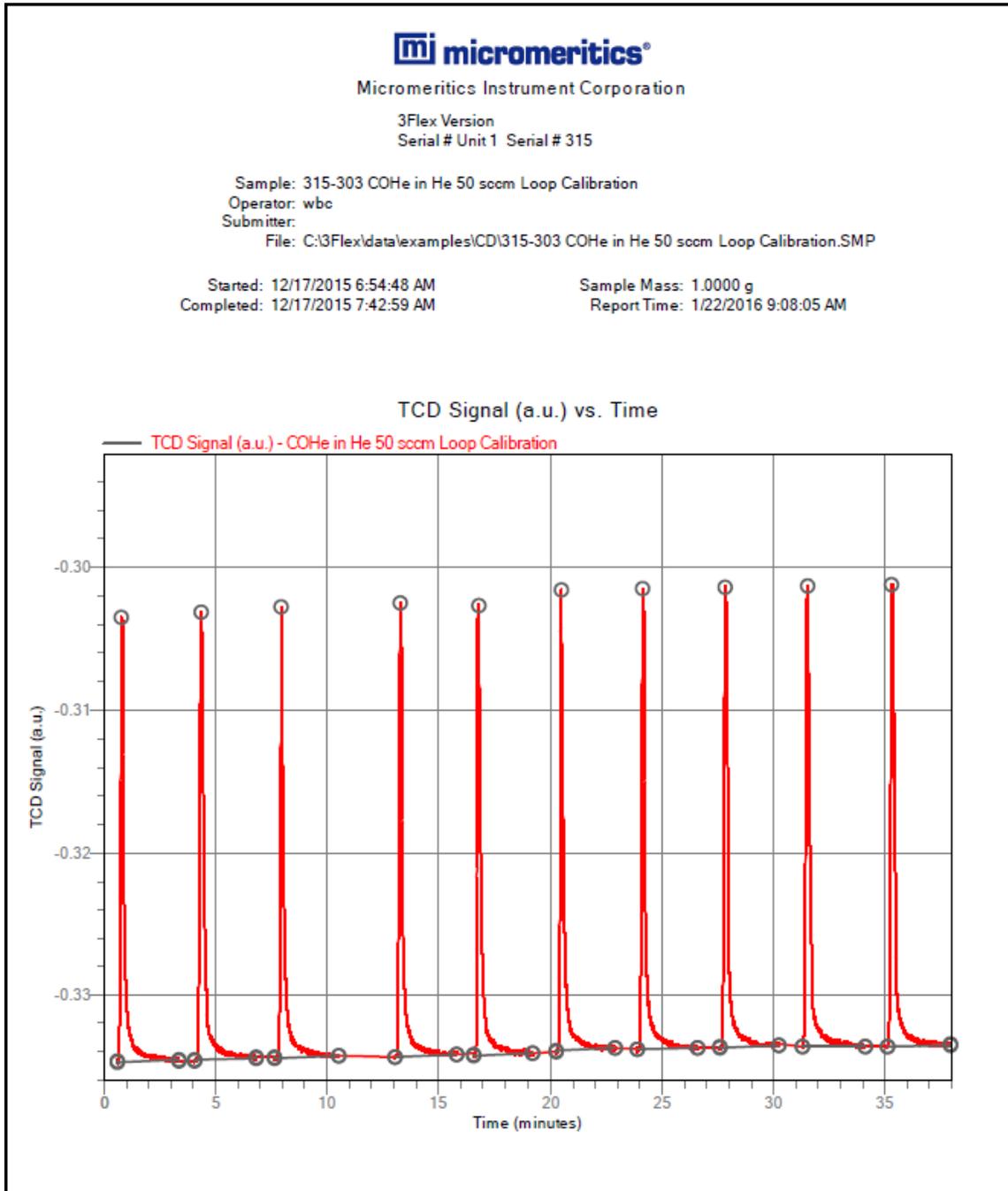
**t-Plot Report - Data**

Relative Pressure (p/p <sup>*</sup> )	Statistical Thickness (Å)	Quantity Adsorbed (cm <sup>3</sup> /g STP)	Fitted
0.053665461	3.2751	45.1706	
0.077624186	3.4967	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	8.0448	122.6982	

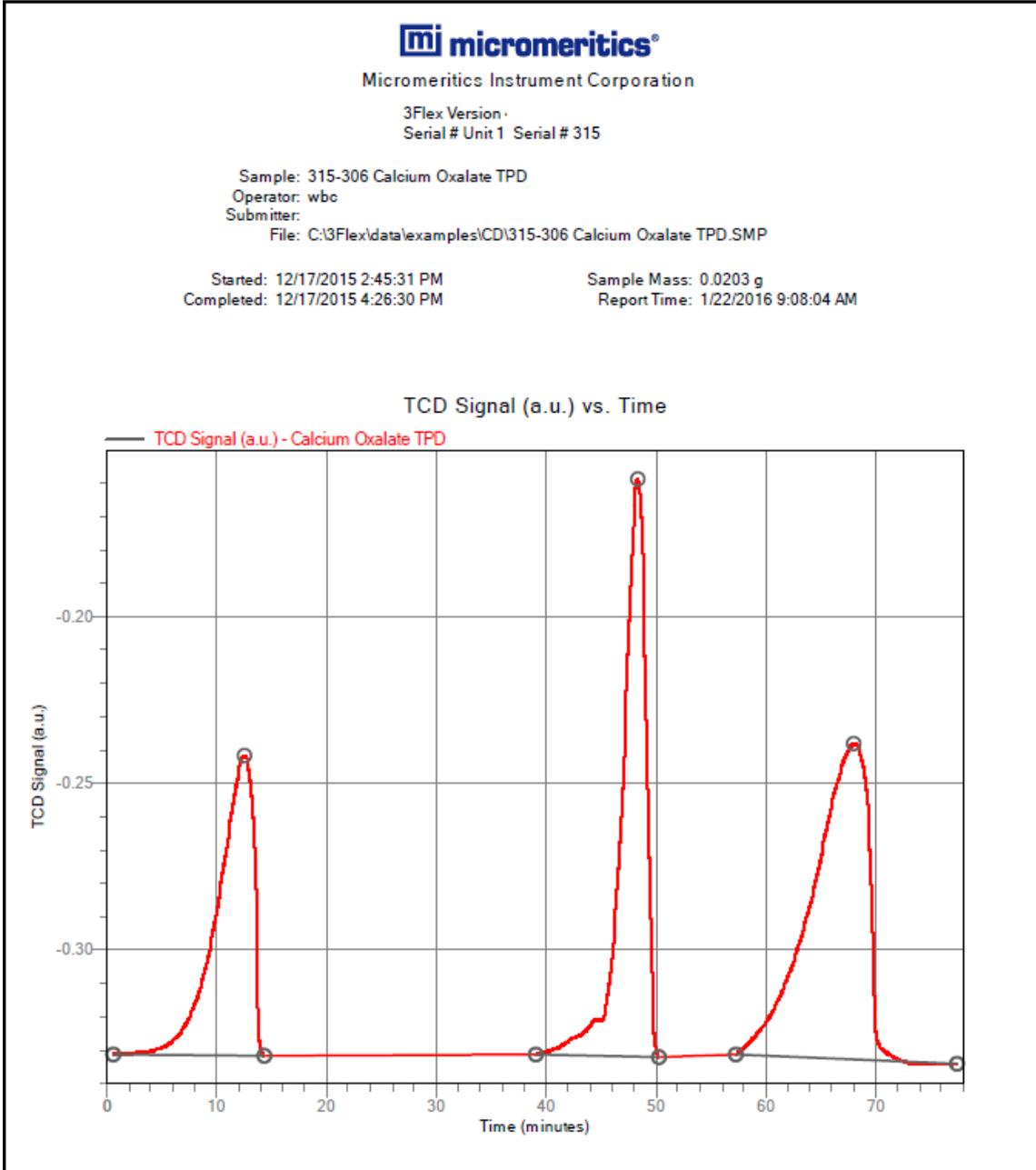
# TCD SIGNAL VS TEMPERATURE



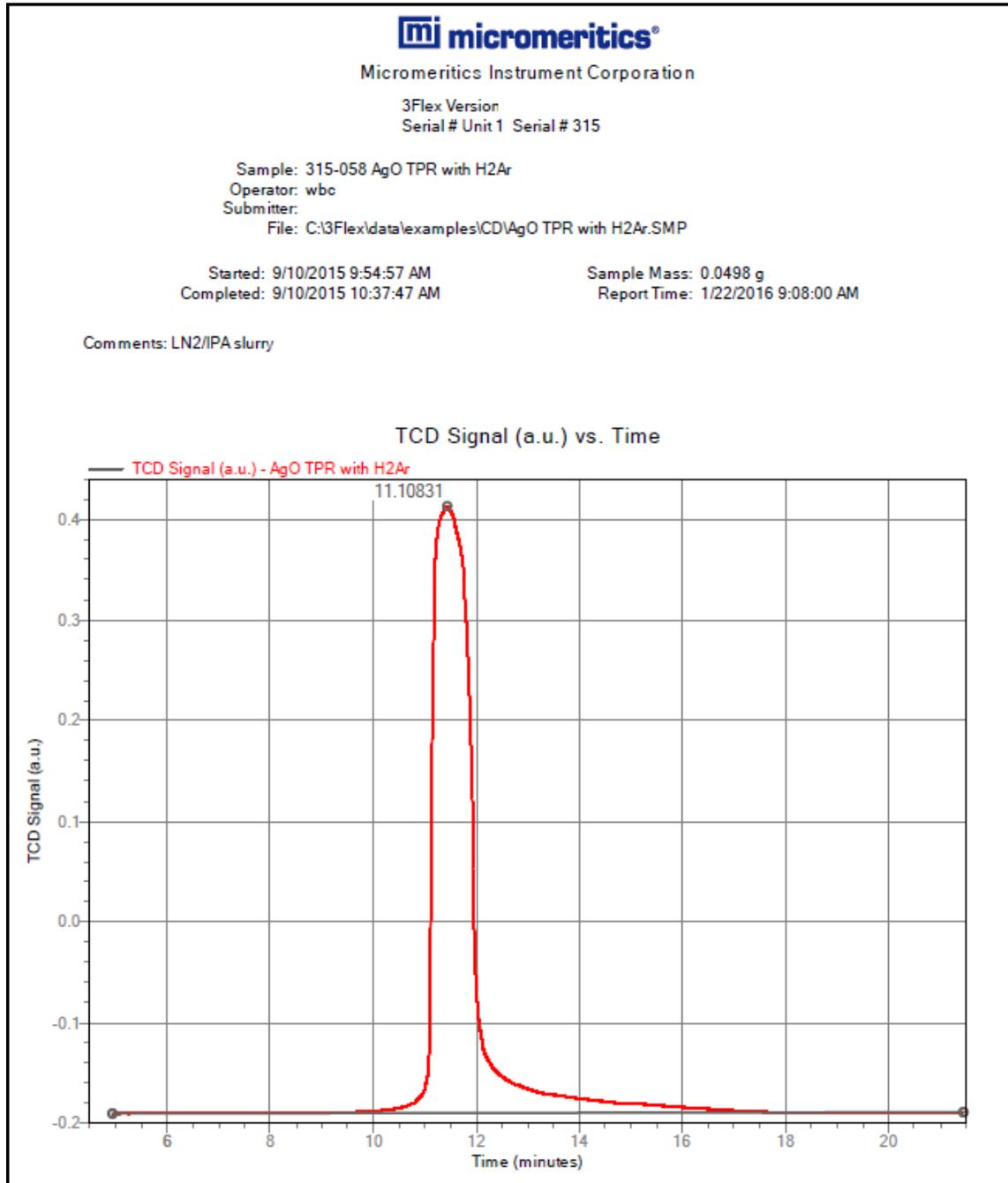
### TCD SIGNAL VS TIME



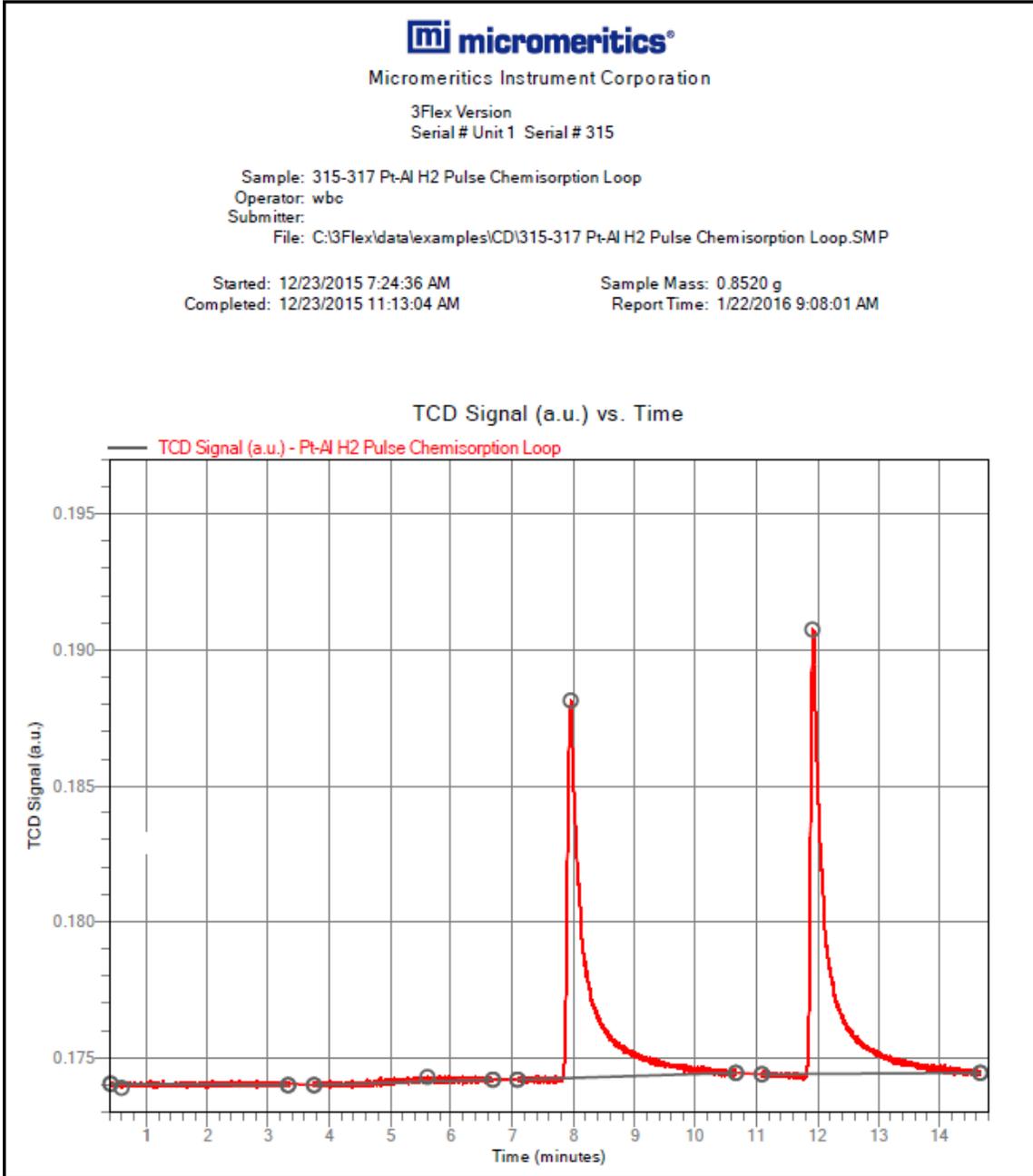
# TCD SIGNAL VS TIME



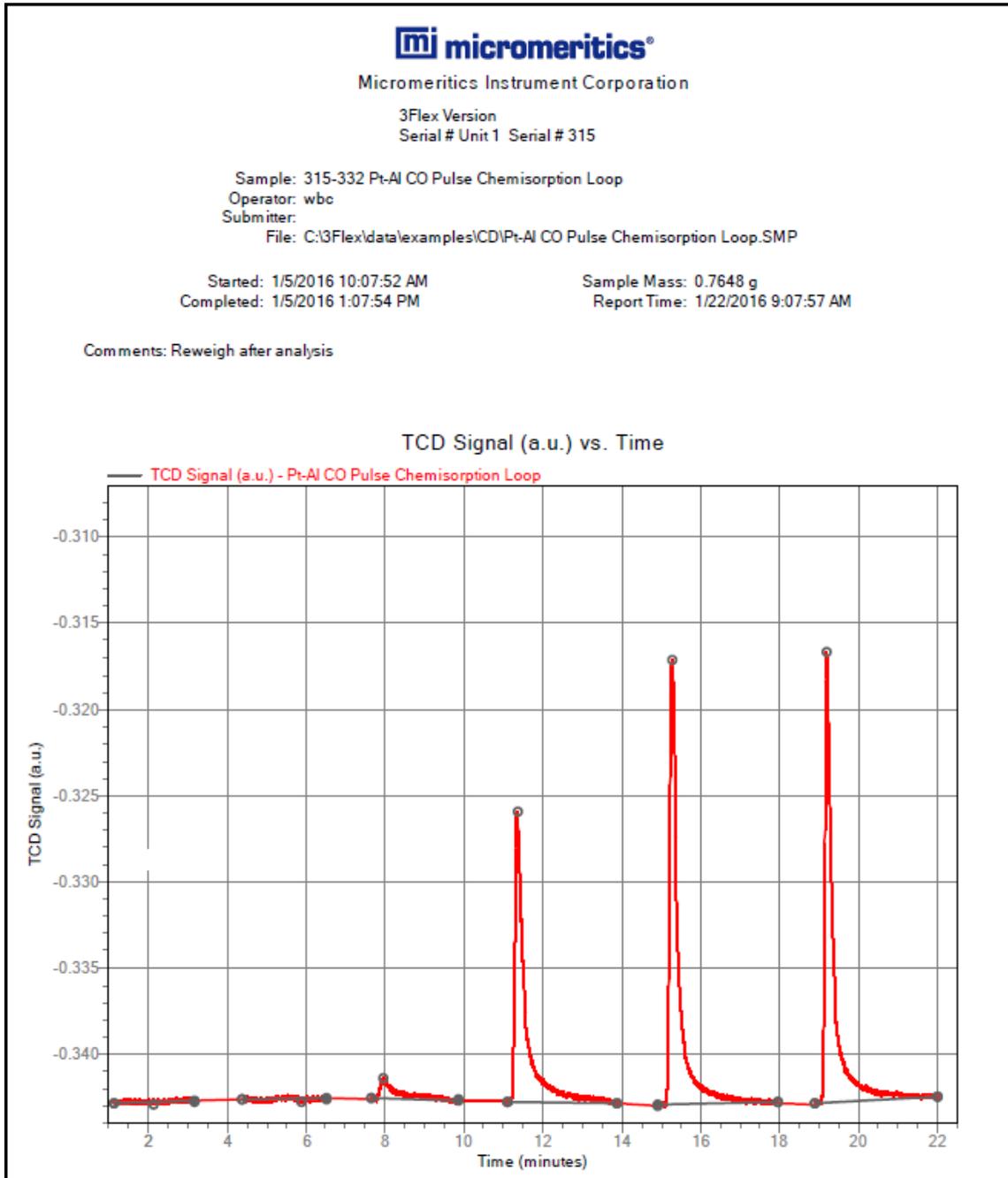
### TCD SIGNAL VS TIME



### TCD SIGNAL VS TIME



### TCD SIGNAL VS TIME



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## 8 SELECTED REPORTS

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



The *Selected Reports* list box may display a *User-Defined* option rather than an *Advanced* option. These options are the same.

Tutorials are available for some reports. These reports are available in online help and on the internet. For a list of available links, go to [3Flex Links on page iv](#).

### Physical Adsorption Reports

- Advanced (or User-Defined)
- Alpha-S Method
- BET Surface Area
- BJH Adsorption/Desorption
- DFT Pore Size
- DFT Surface Energy
- Dollimore Heal Adsorption/Desorption
- Dubinin
- f-Ratio Method
- Freundlich
- Horvath-Kawazoe
- Isotherm
- Langmuir
- MP-Method
- NLDFT Advanced PSD
- Options
- Sample Log
- Summary
- t-Plot
- Temkin
- Use-Defined (or Advanced)
- Validation

### Chemical Adsorption Reports

- Advanced (or User-Defined)
- Difference Method
- Freundlich
- Isotherm
- Langmuir
- Options
- Sample Log
- Sinfelt Method
- Temkin
- User-Defined (or Advanced)

## Dynamic Analysis Reports

See [TCD Report Options for Dynamic Analysis on page 8 - 61](#)

- Advanced Reports
- First Order Kinetics
- Loop Calibration
- Options Report
- Pulse Chemisorption
- Sample Log
- Summary
- TCD Concentration vs Temperature
- TCD Concentration vs Time
- TCD Signal and TCD Concentration vs Time
- TCD Signal vs Temperature
- TCD Signal vs Time
- Temperature vs Time

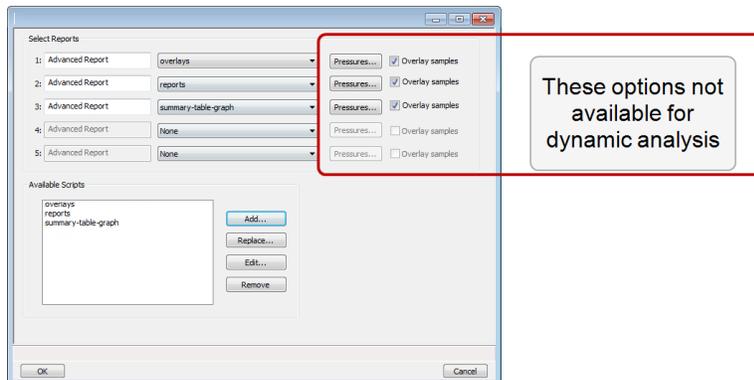
## ADVANCED REPORT OPTIONS



The *Selected Reports* list box may display a *User-Defined* option rather than an *Advanced* option. These options are the same.

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 H - 18](#) 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* 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.



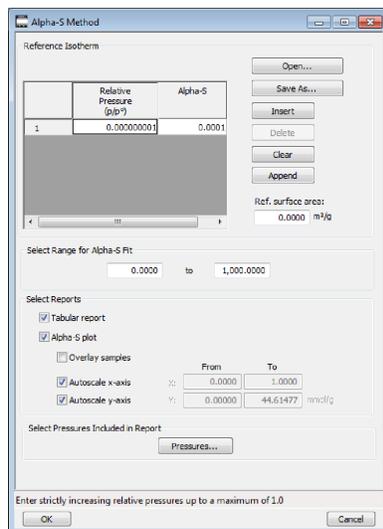
6. In the *Selected Reports* group box, click the drop-down arrows to select up to five Python scripts previously added in the *Available Scripts* box.
7. **P C** Click **Pressures** to add pressure points to the report. Click **OK** to return to the *Report Options* tab.
8. **P C** Select the *Overlay 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 Report Options Fields and Buttons Table

Field or Button	Description
<b>Add</b> [ <i>button</i> ]	Click to add additional Python reports.
<b>Available Scripts</b> [ <i>group box</i> ]	Lists the available reports and provides the option to add, replace, edit or remove reports.
<b>Overlay samples</b> (if shown) [ <i>checkbox</i> ]  	Use to overlay samples as defined by the function.
<b>Advanced Report 1 through 5</b> [ <i>drop-down box</i> ]	Use the drop-down lists to select currently-defined functions used to define the report calculations and output.
	For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .

## 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.

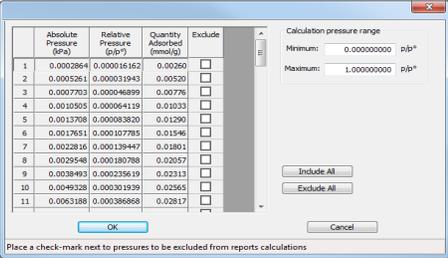


One predefined curve is located in the *Reference* file directory. Use the table buttons to enter relative pressure and the alpha-s values.

### Alpha-S Method Report Options Fields and Buttons Table

Field or Button	Description
<b>Alpha-S plot</b> [check box]	Use to plot data in graph format.  <b>Autoscale x-axis.</b> The x-axis field shows the relative pressure.  <b>Autoscale y-axis.</b> The y-axis field shows the quantity of gas adsorbed.  <b>Overlay samples.</b> Use to overlay sample files on the plot.
<b>Open</b> [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.
<b>Pressures</b> [button]	Use to select a pressure range for report calculations and points for exclusion from calculations.

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

Field or Button	Description
	 <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p>
<b>Ref. surface area</b> [text box]	Enter the surface area from the reference curve. This value is used to calculate the sample surface area.
<b>Select Range for Alpha-S Fit</b> [group box]	Enter minimum and maximum relative pressures to determine the fit.
<b>Selected Reports</b> [group box]	<p><b>Alpha-S Plot.</b> Use to plot data in graph format.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> The x-axis field shows the relative pressure.</li> <li>• <b>Autoscale y-axis.</b> The y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the plot.</li> </ul> <p><b>Tabular Report.</b> Use to have a tabular report of data generated.</p>
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4.</a></p>	

## BET SURFACE AREA REPORT OPTIONS



This report is for physical adsorption and chemical adsorption analyses only.

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

### BET Report Options Fields and Buttons Table

Field or Button	Description
<b>Pressures [button]</b>	This option is available when the sample file has a status of <i>Analyzing</i> or <i>Complete</i> . Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.

## BET Report Options Fields and Buttons Table (continued)

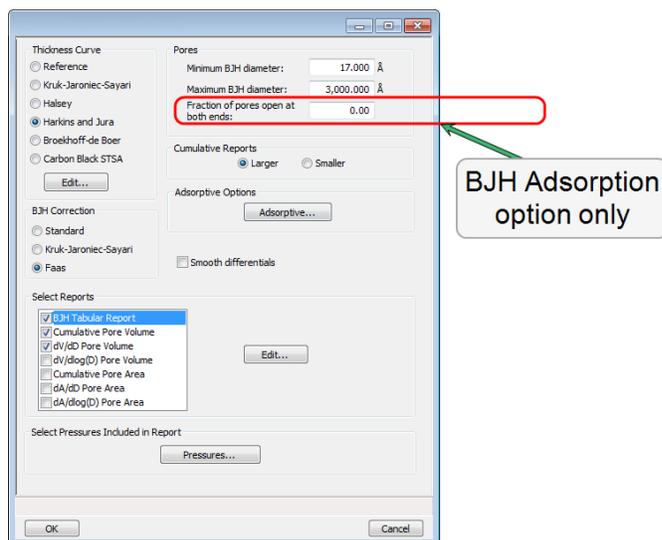
Field or Button	Description
	<p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table if not using the <i>Use Interpolation</i> option.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p> <p><b>Insert Predefined.</b> Click to insert a predefined (default) set of points into the report. <i>Use Interpolation</i> must be selected to enable this button. This button displays for BET reports only.</p> <p><b>Use Interpolation.</b> Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only.</p>
<b>Select Pressure Range for BET fit</b> [text box]	Enter values to indicate the fitted pressure range.
<b>Selected Reports</b> [group box]	<p><b>BET Isotherm plot.</b> Uses BET monolayer volume and constant to produce an isotherm.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET.</li> <li>• <b>Autoscale y-axis.</b> The y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the BET isotherm plot.</li> </ul> <p><b>BET Transform plot.</b> Use to generate a traditional BET surface area plot used to determine monolayer volume and BET C constant.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> Linear x-axes begin at zero. The x-axis field shows the relative pressure for BET.</li> <li>• <b>Autoscale y-axis.</b> The y-axis field shows BET transformation.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the BET transform plot.</li> </ul> <p><b>Tabular report.</b> Use to have a table of measured and calculated values generated.</p>
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

## 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 Report Options Fields and Buttons Table

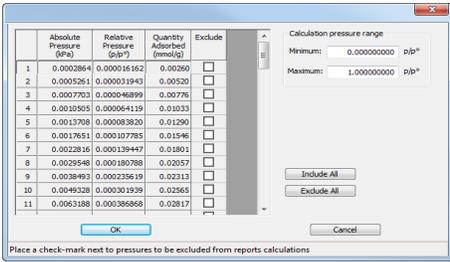
Field or Button	Description
<b>Adsorptive</b> [ <i>button</i> ]	Displays the <i>Adsorptive Options</i> window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified.

Adsorptive	Affinity Coefficient (beta)
1: N2	0.33000
2: Ar	0.26700
3: CO2	0.46100
4:	0.00000
5:	0.00000
6:	0.00000
7:	0.00000
8:	0.00000

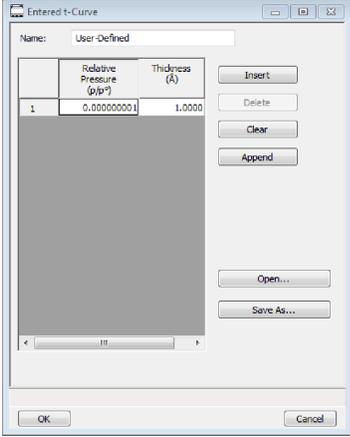
**BJH Adsorption / Desorption Report Options Fields and Buttons Table (continued)**

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

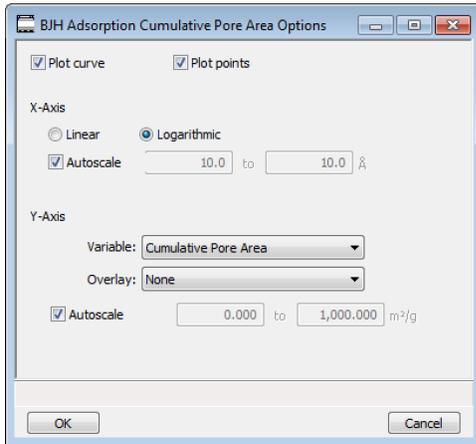
## BJH Adsorption / Desorption Report Options Fields and Buttons Table (continued)

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

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

Field or Button	Description
	 <p>To import values from an existing thickness curve (.THK file), click <b>Open</b>, then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space or a tab.</p>
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

## BJH PLOT OPTIONS

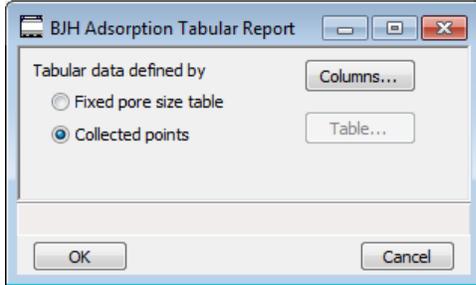


The fields for all plot options are identical for specifying plotting methods and customizing plots. Highlight any plot option in the *Selected Reports* list box in the *BJH Report Options* window, then click [Edit](#).

### BJH Plot Options Fields and Buttons Table

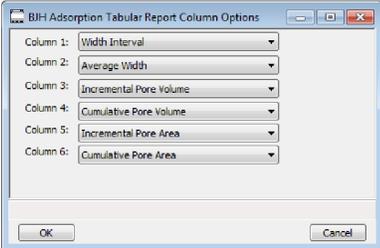
Field or Button	Description
<b>Autoscale</b> [ <i>check box</i> ]	When enabled on the report parameters windows, allows the x- and y- axes to be scaled automatically. <i>Autoscale</i> means that the x- and y- ranges will be set so that all the data is shown. If <i>Autoscale</i> is not selected, the entered range is used.
<b>Plot curve / Plot points</b> [ <i>check box</i> ]	Select to plot points on the graph.
<b>X-Axis</b> [ <i>group box</i> ]	Use to have the x-axis on a logarithmic or linear scale.
<b>Y-Axis</b> [ <i>group box</i> ]	<b>Overlay.</b> Select an option to overlay onto the current report. <b>Variable.</b> Select a variable.
 For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .	

## BJH TABULAR REPORT OPTIONS



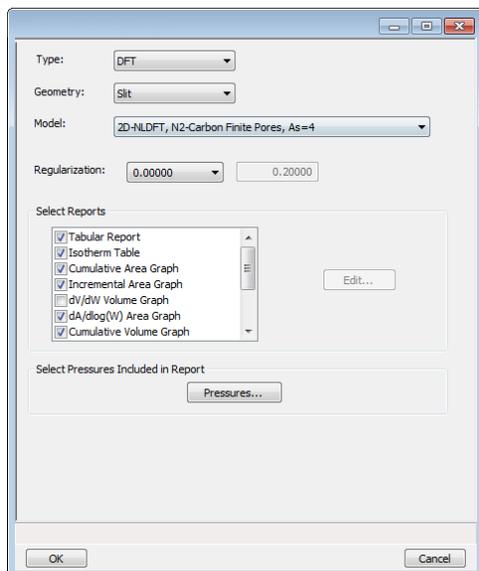
Highlight *BJH Tabular Report* in the *Selected Reports* list box on the *BJH Adsorption Report Options* window, then click **Edit** to specify the method of data reduction.

### BJH Tabular Report Options Fields and Buttons Table

Field or Button	Description
<b>Collected points</b> [selection]	Use to include all relative pressure points collected. Refer to the <b>Columns</b> button below.
<b>Columns</b> [button]	Select the data types to include in the report. <i>Column [n]</i> indicates the column order and data contents for the report. 
<b>Fixed pore size table</b> [selection]	Use to specify exact pore sizes for volume or area data. Click <b>Table</b> to modify the fixed pore size table. Refer to <b>Table</b> and <b>Columns</b> buttons elsewhere in this table.
<b>Table</b> [button]	The fixed pore size table must contain a minimum of two points. The points must be strictly decreasing. Enabled only when <i>Fixed pore size table</i> is selected.
 <b>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</b>	

## 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 Report Options Fields and Buttons Table

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

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

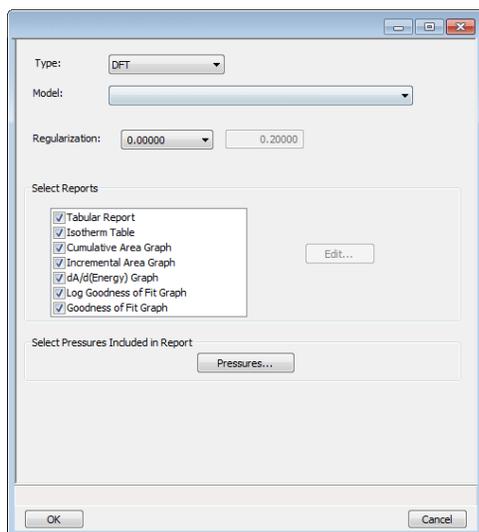
Field or Button	Description
	<p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p>
<p><b>Regularization</b> [drop-down box]</p>	<p>Select the extent of smoothing to apply to the data. If <i>0.20000 (user)</i> is selected, enter a number in the text box giving a relative weight for the smoothing during deconvolution. Larger values produce more smoothing.</p>
<p><b>Select Reports</b> [group box]</p>	<p>Select the reports to generate. To edit graph details, highlight the graph option and click <b>Edit</b>. The <i>Log Goodness of Fit</i> and <i>Goodness of Fit</i> graphs cannot be edited.</p> <div style="border: 1px solid gray; padding: 10px; margin: 10px 0;"> </div> <p><b>Autoscale Options.</b> Use to autoscale the x-axis and/or y-axes.</p> <p><b>Axis Range.</b> <i>From / To</i> fields are enabled when <i>Autoscale</i> options are not selected. Enter the starting and ending values for the x- and/or y-axes.</p> <ul style="list-style-type: none"> <li>• <b>X-axis.</b> Shows the pore size.</li> <li>• <b>Y-axis.</b> Shows the area.</li> </ul> <p><b>Overlay.</b> Select an overlay for the report.</p> <p><b>Plot Type.</b> Select the method for data display.</p>

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

Field or Button	Description
<b>Type</b> [drop-down box]	<p><b>Classical.</b> Model based on the Kelvin equation and thickness for determining the pore size distribution. See <a href="#">DFT Models on page C - 1</a>.</p> <p><b>DFT.</b> Model based on the density functional theory.</p>
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## DFT SURFACE ENERGY REPORT OPTIONS

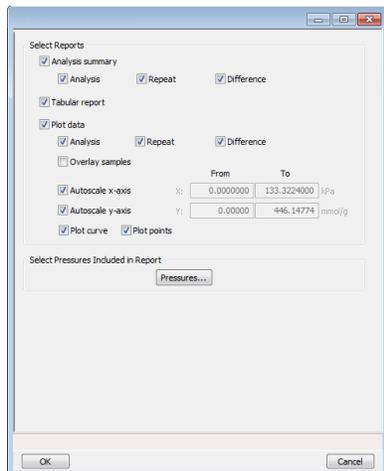
The *DFT Surface Energy* report contains the results of surface energy distribution analyses.



*DFT Surface Energy Report Options* fields and buttons are identical to the *DFT Pore Size Report Options*. See [DFT Pore Size Report Options on page 8 - 15](#).

## DIFFERENCE METHOD REPORT OPTIONS

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



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

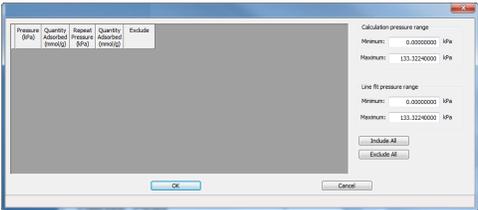
**Difference Method.** The repeat isotherm data are subtracted from the primary isotherm.  $Q_0$  is the y-intercept of a straight line through the difference data.

**Sinfelt Method.** Both the primary and repeat isotherms are fitted to a straight line.  $Q_0$  is the difference between the y-intercepts of the fit lines.

### Difference and Sinfelt Report Options Fields and Buttons Table

Field or Button	Description
<b>Analysis summary</b> [selection]	<p><b>Analysis.</b> Generates a summary of the following for the first analysis:</p> <ul style="list-style-type: none"> <li>• Percent metal dispersion</li> <li>• Metallic surface area</li> <li>• Volume adsorbed</li> <li>• Slope</li> <li>• Correlation coefficient</li> </ul> <p><b>Difference.</b> Generates a summary of the differences between the following information for the first and repeat analyses:</p> <ul style="list-style-type: none"> <li>• Percent metal dispersion</li> </ul>

## Difference and Sinfelt Report Options Fields and Buttons Table (continued)

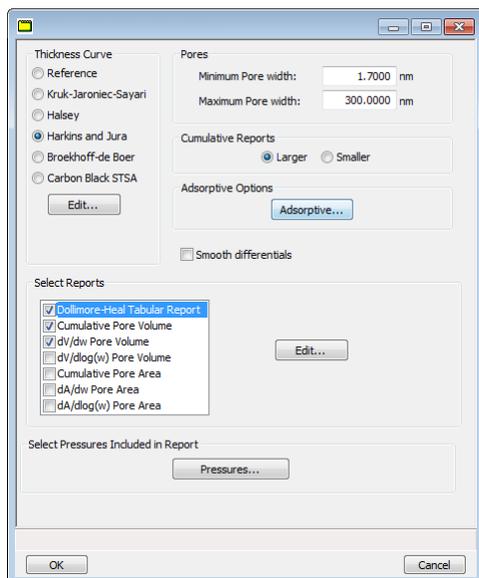
Field or Button	Description
	<ul style="list-style-type: none"> <li>• Metallic surface area</li> <li>• Average difference volume</li> </ul> <p><b>Repeat.</b> (Sinfelt report only). Generates a line fit plot for the secondary analysis.</p>
<b>Pressures</b> [button]	<p>Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.</p>  <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p> <p><b>Line fit pressure range.</b> Enter the minimum and maximum pressures for line fit.</p>

## Difference and Sinfelt Report Options Fields and Buttons Table (continued)

Field or Button	Description
<b>Plot data</b> [ <i>selection</i> ]	<p><b>Analysis.</b> Includes a line fit plot for the primary analysis.</p> <p><b>Autoscale x-axis / Autoscale y-axis.</b> Select to have the X- and/or Y- axes automatically scaled. The application uses the highest values collected during analysis as the ending points for an axis range. X-axis shows the pressure. Y-axis shows the quantity of gas adsorbed.</p> <p><b>Difference.</b> Plots the difference between the analysis and repeat analysis lines.</p> <p><b>Overlay samples.</b> Overlays data from the current sample with that of other samples. Click <b>Overlays</b> on the <i>Report Options</i> window to choose other sample files.</p> <p><b>Plot curve</b> and <b>Plot points.</b> Use to specify how to plot data. Plot data as a curve, points, or both.</p> <p><b>Repeat.</b> Includes a line fit plot for the secondary analysis.</p>
<b>Tabular report</b> [ <i>selection</i> ]	Select to have a report of the pressure points generated.
<div style="border: 1px solid green; padding: 5px;">  <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p> </div>	

## DOLLIMORE-HEAL ADSORPTION / DESORPTION REPORT OPTIONS

The *Dollimore-Heal Adsorption Report Option* and the *Dollimore-Heal Desorption Report Option* generate reports from both adsorption and desorption data. The fields and buttons for these reports are identical to the *BJH Adsorption / Desorption Report Options*. See [BJH Adsorption / Desorption Report Options on page 8 - 9](#).

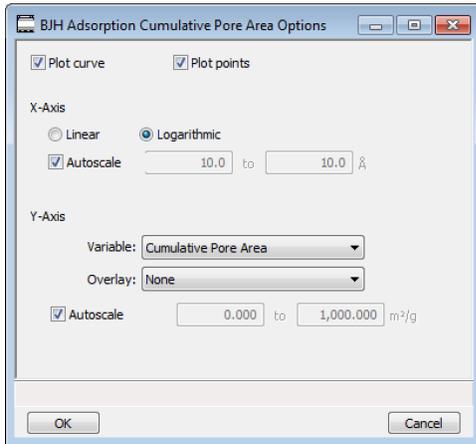


The screenshot shows a software dialog box titled "Dollimore-Heal Adsorption / Desorption Report Options". It contains several sections for configuring report parameters:

- Thickness Curve:** A list of radio buttons for selecting a thickness curve: Reference, Kruk-Jaroniec-Sayari, Halsey, Harkins and Jura (selected), Broelhoff-de Boer, and Carbon Black STSA. An "Edit..." button is located below this list.
- Pores:** Two input fields for pore width: "Minimum Pore width:" set to 1.7000 nm and "Maximum Pore width:" set to 300.0000 nm.
- Cumulative Reports:** Two radio buttons: "Larger" (selected) and "Smaller".
- Adsorptive Options:** An "Adsorptive..." button.
- Smooth differentials:** A checkbox that is currently unchecked.
- Select Reports:** A list of checkboxes for selecting report components:
  - Dollimore-Heal Tabular Report
  - Cumulative Pore Volume
  - dV/dw Pore Volume
  - dV/dlog(w) Pore Volume
  - Cumulative Pore Area
  - dA/dw Pore Area
  - dA/dlog(w) Pore Area
 An "Edit..." button is located to the right of this list.
- Select Pressures Included in Report:** A "Pressures..." button.

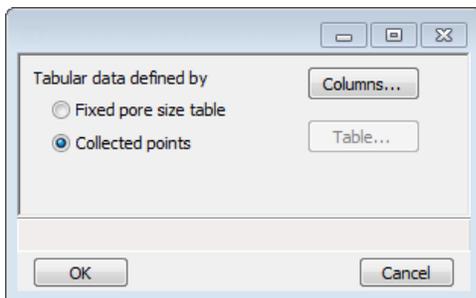
At the bottom of the dialog box are "OK" and "Cancel" buttons.

## DOLLIMORE-HEAL 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**. The fields and buttons for these reports are identical to the *BJH Plot Report Options*. See [BJH Plot Options on page 8 - 13](#).

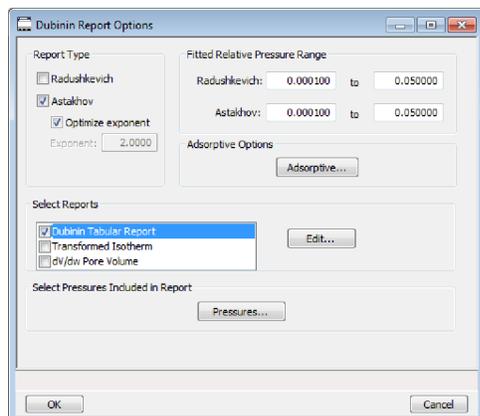
## DOLLIMORE-HEAL TABULAR REPORT OPTIONS



*Dollimore-Heal Tabular Report Options* are identical to the *BJH Tabular Report Options*. See [BJH Tabular Report Options on page 8 - 14](#).

## DUBININ REPORT OPTIONS

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



### Dubinin Report Options Fields and Buttons Table

Field or Button	Description																		
<b>Adsorptive</b> [ <i>button</i> ]	Displays the <i>Adsorptive Options</i> window. The recommended adsorptives and their values are shown. Up to eight adsorptive and adsorbate property factor combinations may be specified. <div data-bbox="555 1050 907 1415" data-label="Image"> <table border="1"> <thead> <tr> <th>Adsorptive</th> <th>Affinity Coefficient (beta)</th> </tr> </thead> <tbody> <tr> <td>1: N2</td> <td>0.33000</td> </tr> <tr> <td>2: Ar</td> <td>0.26700</td> </tr> <tr> <td>3: CO2</td> <td>0.46100</td> </tr> <tr> <td>4:</td> <td>0.00000</td> </tr> <tr> <td>5:</td> <td>0.00000</td> </tr> <tr> <td>6:</td> <td>0.00000</td> </tr> <tr> <td>7:</td> <td>0.00000</td> </tr> <tr> <td>8:</td> <td>0.00000</td> </tr> </tbody> </table> </div>	Adsorptive	Affinity Coefficient (beta)	1: N2	0.33000	2: Ar	0.26700	3: CO2	0.46100	4:	0.00000	5:	0.00000	6:	0.00000	7:	0.00000	8:	0.00000
Adsorptive	Affinity Coefficient (beta)																		
1: N2	0.33000																		
2: Ar	0.26700																		
3: CO2	0.46100																		
4:	0.00000																		
5:	0.00000																		
6:	0.00000																		
7:	0.00000																		
8:	0.00000																		
<b>Fitted Relative Pressure Range</b> [ <i>group box</i> ]	Enter the minimum and maximum limits for Radushkevich or Astakhov relative pressures included in the line fit.																		

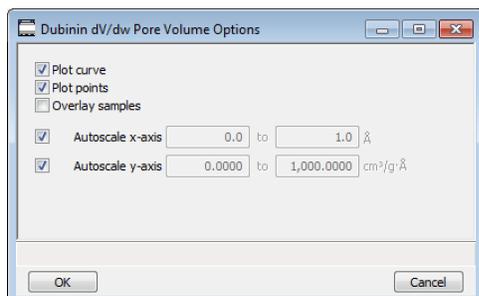
Dubinin Report Options Fields and Buttons Table (continued)

Field or Button	Description
<p><b>Pressures</b> [button]</p>	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p> <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p>
<p><b>Report Type</b> [group box]</p>	<p>Select report types. If <i>Astakhov</i> is selected, either select <i>Optimize exponent</i> or enter an appropriate exponent value in the text box.</p>
<p><b>Select Reports</b> [group box]</p>	<p>Select the reports to generate. Highlight the report, then click <b>Edit</b> to modify report options.</p>
<p><b>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons</a> on page 2 - 4.</b></p>	

## 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.

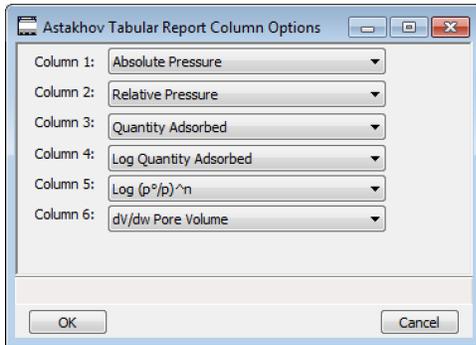


### Dubinin Pore Volume Report Fields and Buttons Table

Field or Button	Description
<b>Autoscale x-axis / Autoscale y-axis</b> [ <i>check box</i> ]	Select an option to have the x- and/or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges.  Enable to enter beginning and ending values manually.
<b>Overlay samples</b> [ <i>selection</i> ]	Use to overlay sample files on the plot.
<b>Plot curve / Plot points</b> [ <i>selection</i> ]	Select to plot points on the graph.
 For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .	

## 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.

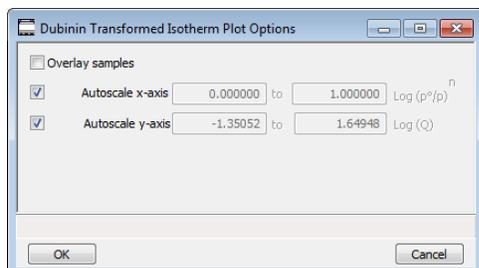


**Log (p°/p)^n.** The value for *[n]* is the optimized exponent if *Optimize exponent* is selected on the *Dubinin Report Options* window. If not, then the value for *[n]* is the entered exponent value.

## 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.

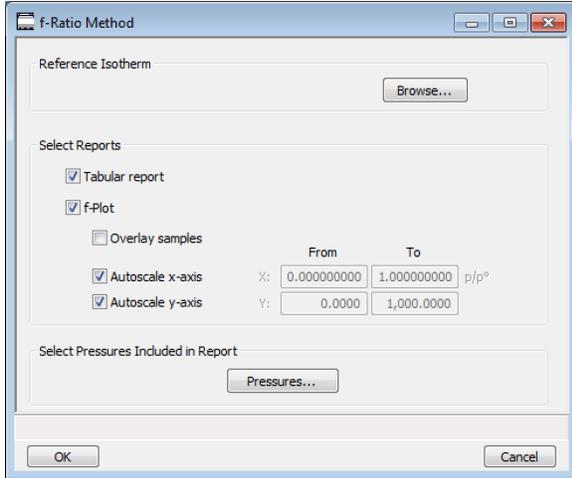


Dubinin Transformed Isotherm Plot Options Fields and Buttons Table

Field or Button	Description
<b>Autoscale x-axis / Autoscale y-axis</b> [ <i>check box</i> ]	<p>Select an option to have the x- and/or y-axes scaled automatically. Both axes begin at 0; the system uses the highest values collected during analysis as the ending points for axis ranges.</p> <p>Deselect to enter beginning and ending values manually.</p> <p><b>Autoscale x-axis.</b> Shows the quantity of gas adsorbed at standard temperature and pressure.</p> <p><b>Autoscale y-axis.</b> Shows the log of relative pressure.</p>
<b>Overlay Samples</b> [ <i>check box</i> ]	Use to overlay sample files on the plot.
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## F-RATIO METHOD REPORT OPTIONS

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



### f-Ratio Fields and Buttons Table

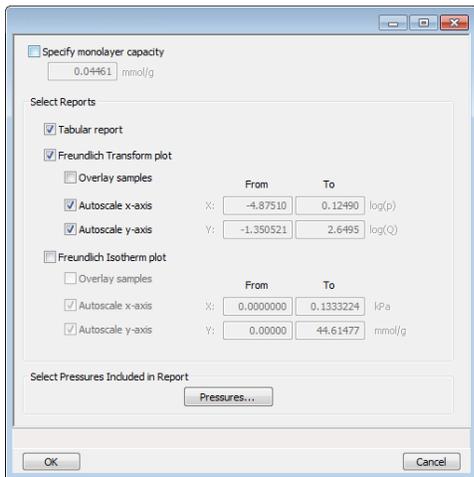
Field or Button	Description
<b>Pressures [button]</b>	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p> <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations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

## f-Ratio Fields and Buttons Table (continued)

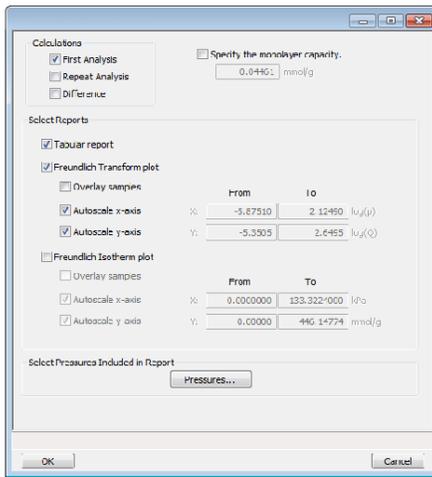
Field or Button	Description
<b>Reference isotherm</b> [group box]	Browse to select a sample file to use as a reference for the isotherm. Select a file containing an isotherm measured from a non-porous sample of the same material as the current sample.
<b>Selected Reports</b> [group box]	<p><b>Tabular Report.</b> Use to have a tabular report of data generated.</p> <p><b>f-Plot.</b> Use to generate a normalized isotherm.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> The X-axis field is dimensionless in units of f-ratio.</li> <li>• <b>Autoscale y-axis.</b> The Y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the f-plot.</li> </ul>
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

## 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**.



**Physical Adsorption**

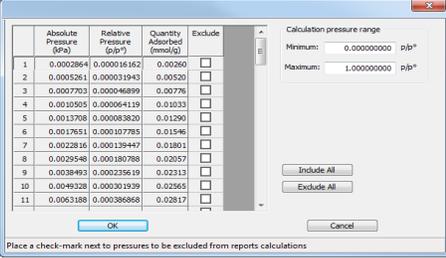
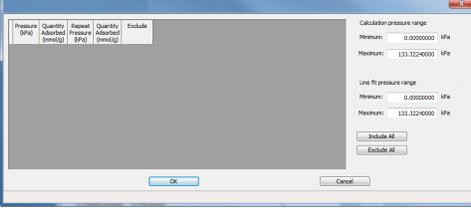


**Chemical Adsorption**

### Freundlich Report Options Fields and Buttons Table

Field or Button	Description
<b>Calculations</b> [group box] 	Select from the various calculation options.  <b>Difference.</b> Plots the difference between the analysis and repeat analysis lines.  <b>First Analysis.</b> Includes a line fit plot for the primary analysis.  <b>Repeat Analysis.</b> Includes a line fit plot for the secondary analysis.

Freundlich Report Options Fields and Buttons Table (continued)

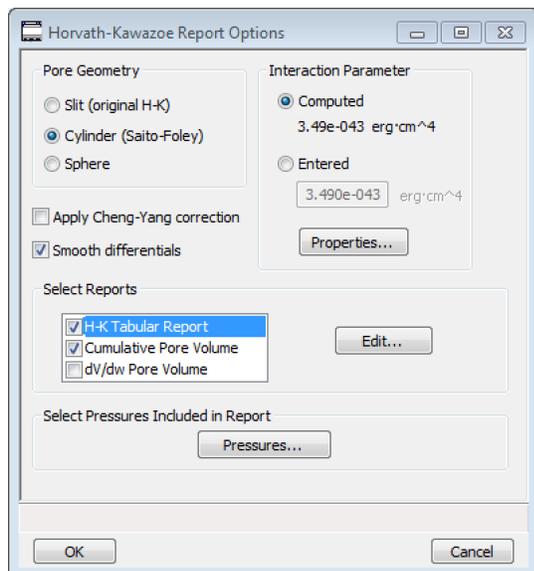
Field or Button	Description
<p><b>Pressures [button] <span style="border: 1px solid black; padding: 0 2px;">P</span></b></p>	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p>
<p><b>Pressures [button] <span style="border: 1px solid black; padding: 0 2px;">C</span></b></p>	<p>Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.</p>  <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p> <p><b>Line fit pressure range.</b> Enter the minimum and maximum pressures for line fit.</p>

Freundlich Report Options Fields and Buttons Table (continued)

Field or Button	Description
<b>Select Reports</b> [group box]	<p><b>Freundlich Isotherm plot.</b> Plots the absolute pressure vs quantity adsorbed. Shows best fit line.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> Linear x-axes begin at zero. The x-axis field shows the absolute pressure.</li> <li>• <b>Autoscale y-axis.</b> Y-axes begin at zero. The y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the Freundlich isotherm plot.</li> </ul> <p><b>Freundlich Transform plot.</b> Plots the log(P) vs log(Q) and the best fit.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> The x-axis field shows the absolute pressure.</li> <li>• <b>Autoscale y-axis.</b> The y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the Freundlich transform plot.</li> </ul> <p><b>Tabular report.</b> Select to include pressure points included in the report.</p>
<b>Specify monolayer capacity</b> [selection]	Select and enter the monolayer capacity of the sample.
<b>Tabular report</b> [selection]	Use to have a report of the pressure points generated.
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4.</a></p>	

## HORVATH-KAWAZOE REPORT OPTIONS

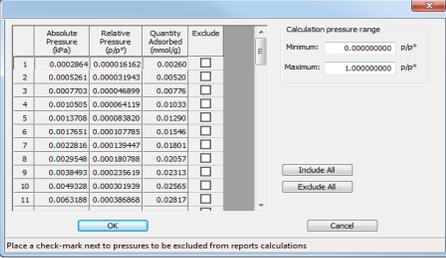
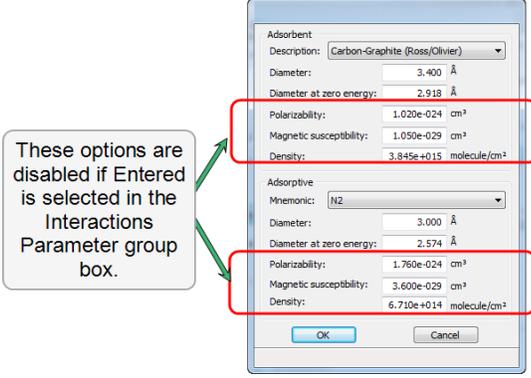
The *Horvath-Kawazoe* method plots individual peaks for different pore sizes even if the difference between one pore size and the next is only one angstrom (0.10 nm) or less.



### Horvath-Kawazoe Report Fields and Buttons Table

Field or Button	Description
<b>Apply Cheng-Yang correction</b> [ <i>selection</i> ]	Use to apply the Cheng-Yang correction to the pore size analysis. This correction substitutes the Langmuir equation of state for Henry's Law in the Horvath-Kawazoe derivation.
<b>Interaction Parameter</b> [ <i>group box</i> ]	Use to determine which interaction parameter will be used in the report. These options are disabled if <i>Sphere</i> is selected in the <i>Pore Geometry</i> group box.  <b>Computed.</b> Use to calculate using the parameters on the <i>Horvath-Kawazoe Physical Properties</i> window (click <b>Properties</b> to display the <i>Physical Properties</i> window). The interaction parameter is recalculated each time a parameter in the <i>Physical Properties</i> window is edited.  <b>Entered.</b> Calculates using the value entered in the text box.
<b>Pore Geometry</b> [ <i>group box</i> ]	Select the option that best represents the physical geometry of the micropores in the sample material. When <i>Sphere</i> is selected, options in the <i>Interaction Parameter</i> group box are disabled.

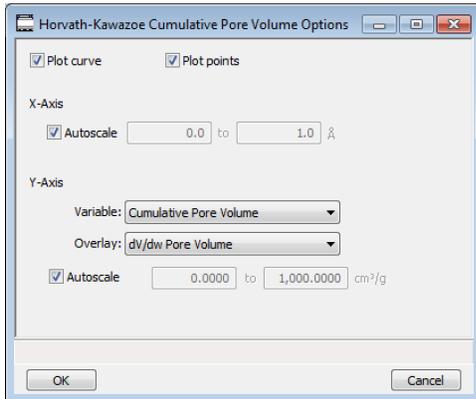
## Horvath-Kawazoe Report Fields and Buttons Table (continued)

Field or Button	Description
<b>Pressures</b> [ <i>button</i> ]	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p>
<b>Properties</b> [ <i>button</i> ]	<p>Click to view or edit the constants describing the physical properties of the adsorbent and adsorptive.</p>  <p><b>Adsorbent.</b> Contains the parameters for the sample. If using <i>Computed</i> for the interaction parameter, all fields are enabled. If using <i>Entered</i>, only the values in the <i>Diameter</i> and <i>Diameter at zero energy</i> text fields may be edited.</p> <ul style="list-style-type: none"> <li>• <b>Density.</b> Enter the density per unit area of the sample.</li> <li>• <b>Description.</b> Select the name of the sample used in the analysis.</li> <li>• <b>Diameter.</b> Enter the diameter of the sample atom.</li> <li>• <b>Diameter at zero energy.</b> Enter the diameter of an atom at zero interaction energy: <math>(2/5)^{1/6} \times \text{diameter}</math>.</li> </ul>

## Horvath-Kawazoe Report Fields and Buttons Table (continued)

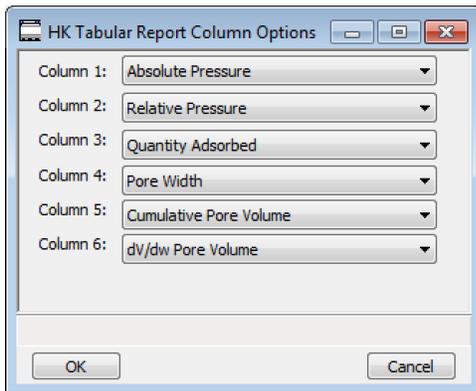
Field or Button	Description
	<ul style="list-style-type: none"> <li>• <b>Magnetic susceptibility.</b> Enter the magnetic susceptibility of the sample.</li> <li>• <b>Polarizability.</b> Enter the polarizability of the sample.</li> </ul> <p><b>Adsorptive.</b> Contains the parameters for the adsorptives. If using <i>Computed</i> for the interaction parameter, all fields are enabled. If using <i>Entered</i>, only the values in the <i>Diameter</i> and <i>Diameter at zero energy</i> text fields may be edited.</p> <ul style="list-style-type: none"> <li>• <b>Density.</b> Enter the density per unit area of the adsorptive.</li> <li>• <b>Diameter.</b> Enter the diameter of the gas phase atom.</li> <li>• <b>Diameter at zero energy.</b> Enter the diameter of an atom at zero interaction energy: <math>(2/5)^{1/6} \times \text{diameter}</math>.</li> <li>• <b>Magnetic susceptibility.</b> Enter the magnetic susceptibility of the adsorptive.</li> <li>• <b>Mnemonic.</b> Select the mnemonic of the adsorptive gas in use.</li> <li>• <b>Polarizability.</b> Enter the polarizability of the adsorptive.</li> </ul>
<b>Select Reports</b> [group box]	Select the types of reports to generate. Highlight the report, then click <b>Edit</b> to modify report parameters.
<b>Smooth Differentials</b> [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 <a href="#">Common Fields and Buttons on page 2 - 4</a> .

## HORVATH-KAWAZOE PLOT OPTIONS



Highlight a plot option in the *Selected Reports* list box in the *Horvath-Kawazoe Report Options* window, then click **Edit** to customize the plotting method. See [BJH Plot Options on page 8 - 13](#) for additional information on fields and buttons for this report.

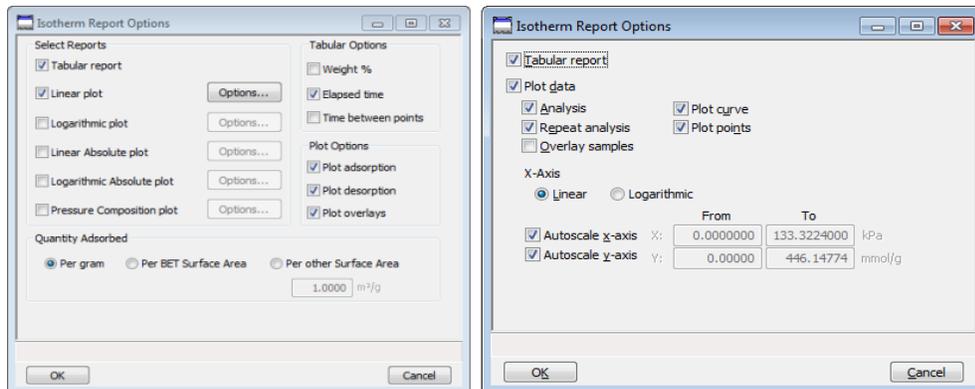
## HORVATH-KAWAZOE TABULAR REPORT OPTIONS



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.



**Physical Adsorption**

**Chemical Adsorption**

### Isotherm Report Options Fields and Buttons Table

Field or Button	Description
<b>Autoscale</b> [ <i>checkbox</i> ] <b>SC</b>	<p><b>Autoscale x-axis.</b> Linear x-axes begin at zero. Logarithmic x-axes begin at an appropriate value. The x-axis field shows the relative or absolute pressure.</p> <p><b>Autoscale y-axis.</b> The y-axis field shows the quantity of gas adsorbed.</p>
<b>Options</b> [ <i>button</i> ] <b>P</b>	<p>Click to display related linear plot options. All plot windows contain identical fields.</p> <p><b>Autoscale x-axis.</b> Linear x-axes begin at zero. Logarithmic x-axes begin at an appropriate value. The x-axis field shows the relative or absolute pressure.</p> <p><b>Autoscale y-axis.</b> The y-axis field shows the quantity of gas adsorbed.</p> <p><b>Plot curve / Plot points.</b> Select to plot points on the graph.</p>
<b>Plot Data</b> [ <i>checkbox</i> ] <b>SC</b>	<p>Select each option to include in the final report.</p>
<b>Plot Options</b> [ <i>group box</i> ] <b>P</b>	<p>Select the types of isotherm to plot.</p>

## Isotherm Report Options Fields and Buttons Table (continued)

Field or Button	Description
<b>Quantity Adsorbed</b> [group box] <b>P</b>	Select how to report the quantity adsorbed. <ul style="list-style-type: none"> <li>per gram (cm<sup>3</sup>/g) STP</li> <li>per BET Surface Area (cm<sup>3</sup>/m<sup>2</sup>) STP or mmol/g</li> <li>per other Surface Area (cm<sup>3</sup>/m<sup>2</sup>) STP or mmol/m<sup>2</sup></li> </ul>
<b>Selected Reports</b> [group box] <b>P</b>	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.
<b>Tabular Options</b> [group box] <b>P</b>	Select the options to include on the report. <p><b>Elapsed time.</b> Time elapsed during the analysis</p> <p><b>Time between points.</b> Time elapsed between points during the analysis</p> <p><b>Weight %.</b> Enter the mass percentage when plotting pressure composition</p>
<b>Tabular Report</b> [group box] <b>SC</b>	Select to include tabular data in the report.
<b>x-axis</b> [group box] <b>SC</b>	Indicate if the x-axis should be in linear or logarithmic format.
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## LANGMUIR REPORT OPTIONS



This report is for physical adsorption and chemical adsorption analyses only.

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.

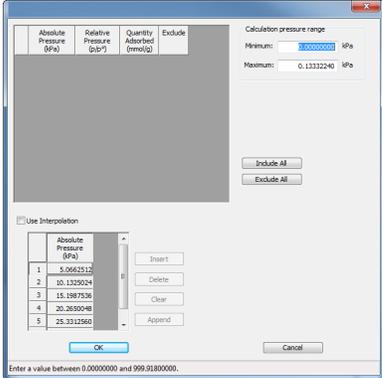
Physical Adsorption

Chemical Adsorption

### Langmuir Report Options Fields and Buttons Table

Field or Button	Description
<b>Calculations</b> [group box]	Select one or more of the calculation options to be used for analysis.
<b>Pressures</b> [button]	Use to enter a range of pressure points to be included in the report or to modify table values for pressure points. <div style="text-align: center;"> </div> <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table.</p>

## Langmuir Report Options Fields and Buttons Table (continued)

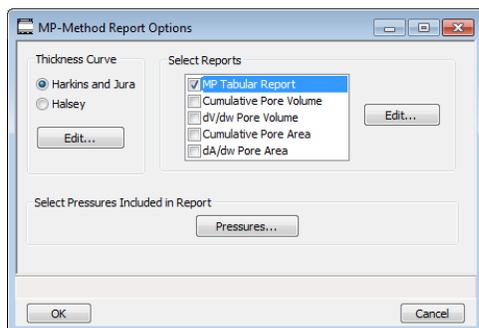
Field or Button	Description
	<p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p> <p><b>Line fit pressure range.</b> Enter the minimum and maximum pressures for line fit.</p>
<p><b>Pressures [button] </b></p>	<p>This option is available when the sample file has a status of <i>Analyzing</i> or <i>Complete</i>. Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.</p>  <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table.</p> <p>To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p> <p><b>Use Interpolation.</b> Use to indicate if the system should use the table or interpolated data. This option is available for BET and Langmuir reports only.</p>
<p><b>Select Pressure Range for Langmuir fit [group box] </b></p>	<p>Enter values to indicate the fitted pressure range.</p>
<p><b>Selected Reports [group box]</b></p>	<p><b>Langmuir Isotherm Plot.</b> Uses the Langmuir monolayer volume and constant to produce an isotherm.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> Linear x-axes begin at zero. The x-axis</li> </ul>

## Langmuir Report Options Fields and Buttons Table (continued)

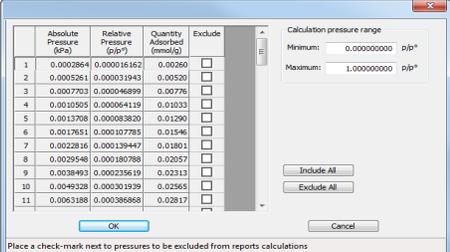
Field or Button	Description
	<p>field shows the absolute pressure for Langmuir.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale y-axis.</b> The y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the Langmuir isotherm plot.</li> </ul> <p><b>Langmuir Transform Plot.</b> Use to generate a traditional Langmuir surface area plot used to determine monolayer volume constant</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> Linear x-axes begin at zero. The x-axis field shows the absolute pressure for Langmuir.</li> <li>• <b>Autoscale y-axis.</b> The y-axis field shows Langmuir transformation.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the Langmuir transform plot.</li> </ul>
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4.</a></p>

## MP-METHOD REPORT OPTIONS

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



### MP-Method Report Options Fields and Buttons Table

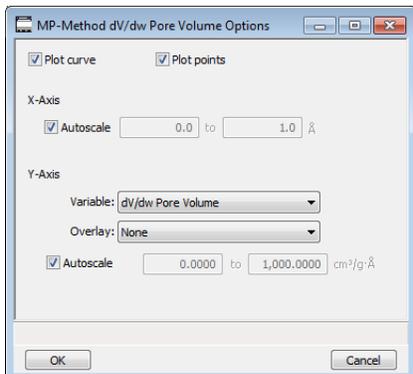
Field or Button	Description
<b>Pressures [button]</b>	<p>Use to select a pressure range for report calculations and points for exclusion from calculations.</p>  <p>Place a check-mark next to pressures to be excluded from reports calculations</p> <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p>

## MP-Method Report Options Fields and Buttons Table (continued)

Field or Button	Description
<b>Select Reports</b> [group box]	Select the reports to generate. Highlight the report, then click <b>Edit</b> to modify report options.
<b>Thickness Curve</b> [group box]	Select the thickness curve, then click <b>Edit</b> to modify the values in the equation for the selected curve.
	For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .

## MP-METHOD PLOT REPORT OPTIONS

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

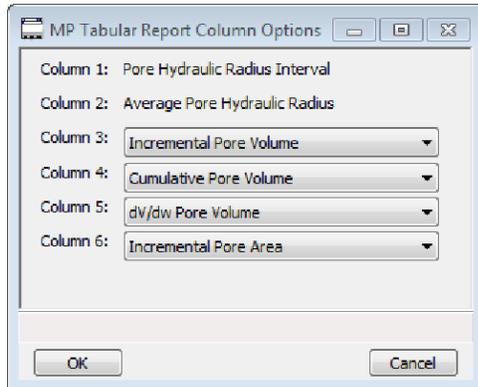


### MP Method Plot Options Fields and Buttons Table

Field or Button	Description
<b>Overlay</b> [ <i>drop-down box</i> ]	Select an option to overlay on the current report.
<b>Plot curve / Plot points</b> [ <i>selection</i> ]	Select to plot points on the graph.
<b>Thickness Curve</b> [ <i>group box</i> ]	Select the thickness curve, then click <b>Edit</b> to modify the values in the equation for the selected curve.
<b>X-Axis</b> [ <i>check box</i> ]	Use to have the x-axis autoscaled or enter beginning and ending values.
<b>Y-Axis</b> [ <i>group box</i> ]	<p><b>Autoscale.</b> Use to have the y-axis autoscaled or enter beginning and ending values.</p> <p><b>Overlay.</b> Select an option to overlay on the current report.</p> <p><b>Variable.</b> Select a variable.</p>
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## MP-METHOD TABULAR REPORT OPTIONS

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

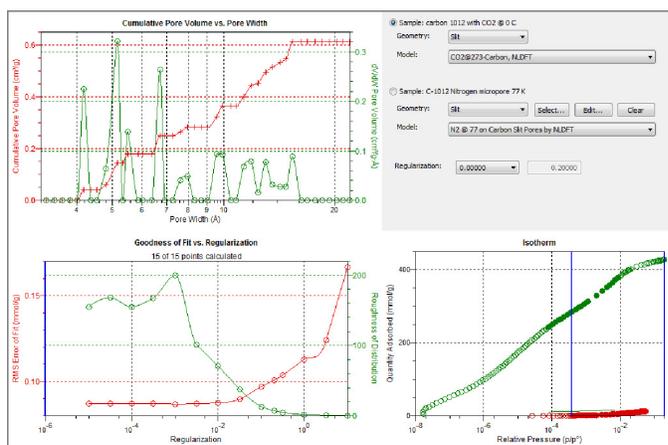


The MP Method reports hydraulic radius only. If Pore size in diameter is selected on the Unit Selection window, pore size in radius will be reports.

## NLDFT ADVANCED PSD REPORT

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 N<sub>2</sub> at 77 degrees Kelvin, then only the N<sub>2</sub> 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.

### NLDFT Advanced PSD Report Fields and Buttons Table

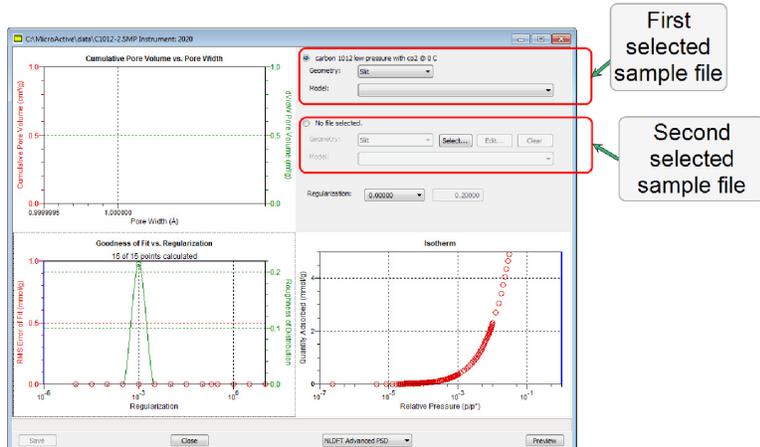
Field or Button	Description
<b>Geometry</b> [drop-down box]	Select the pore shape.
<b>Model</b> [drop-down box]	Lists the models that meet the specified criteria and match the adsorbate and temperature of the sample data. If no models appear, no models meet the selected criteria. One model must be selected.
<b>Regularization</b> [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.
<b>Select Reports</b> [group box]	Use to select the second sample file.



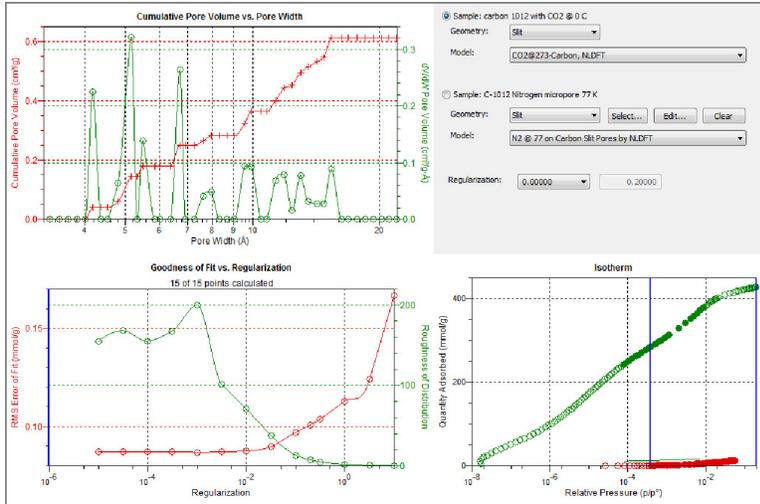
For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 4](#).

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.



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



## OPTIONS REPORT

**Physical adsorption analyses.** **P** 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

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

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

**Dynamic analyses.** **DA** Produces a printed report of predefined collection of sample file parameters



Options reports cannot be edited.

## ***SAMPLE LOG REPORT***

This report provides information on:

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

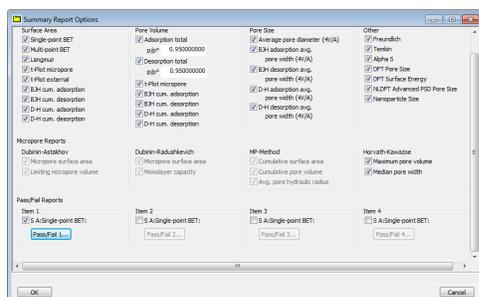
## ***SINFELT AND DIFFERENCE METHODS***

The *Difference Method Report* and the *Sinfelt Method Report* windows are identical unless otherwise specified. See [\*Difference Method Report Options on page 8 - 18.\*](#)

## SUMMARY REPORT OPTIONS

### PHYSICAL ADSORPTION ANALYSES

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  $p/p^0$  field is enabled. Enter the relative pressure used to calculate the total pore volume.

### Summary Report Fields and Buttons Table

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

## Summary Report Fields and Buttons Table (continued)

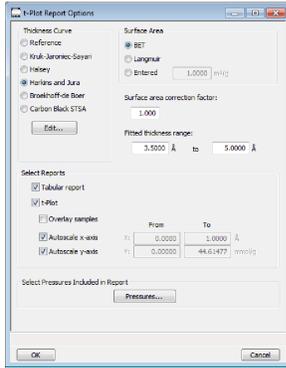
Field or Button	Description
	parameter. A range can be left open by not selecting the limit. In the text box to the right of <i>Upper / Lower</i> , enter operator instructions to be displayed if a failure is encountered.
<b>Select All / Deselect All</b> [button]	Selects (or deselects) all options.
	For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .

## DYNAMIC ANALYSES

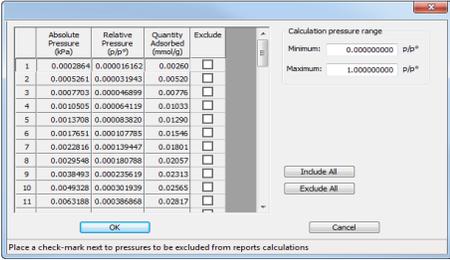
The *Summary Report* for dynamic analyses provides a condensed summary of the peaks and selected analysis parameters.

## T-PLOT REPORT OPTIONS

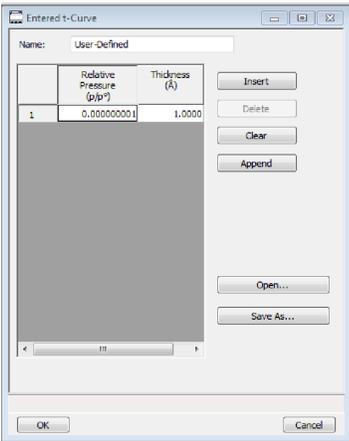
The *t*-Plot calculation allows quantitative analysis of the area and total volume ascribed to micropores. Matrix area (the area external to micropores) is directly determined and often proves to be a valuable way of characterizing complex mixed materials.



t-Plot Report Options Fields and Buttons Table

Field or Button	Description
<b>Fitted thickness range</b> [text box]	Enter the minimum and maximum thicknesses (in angstroms or nanometers) to include in the thickness curve. Go to <b>Options &gt; Units</b> to specify default units.
<b>Pressures</b> [button]	Use to select a pressure range for report calculations and points for exclusion from calculations. <div style="text-align: center;">  </div> <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p>

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

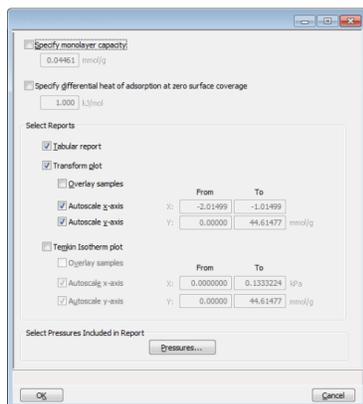
Field or Button	Description
<b>Selected Reports</b> [group box]	<p><b>Tabular Report.</b> Use to have a tabular report of data generated.</p> <p><b>t-Plot.</b> Use to have a graphical representation of data generated.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> The X-axis field shows the statistical thickness of the adsorbed film.</li> <li>• <b>Autoscale y-axis.</b> The Y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the t-plot.</li> </ul>
<b>Surface area correction factor</b> [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.
<b>Surface Area</b> [group box]	Select the surface area value used for thickness calculations. BET is the most commonly used option.
<b>Thickness Curve</b> [group box]	<p>Select the thickness curve, then click <b>Edit</b> to modify the values in the equation for the selected curve. The Frenkel-Halsey-Hill thickness curve can be applied using the Halsey option.</p> <p><b>Kruk-Jaroniec-Sayari / Halsey / Harkins and Jura / Broekhoff-de Boer / Carbon Black STSA.</b> Select the thickness curve option, then click <b>Edit</b>. Modify the equation for the selected curve as needed.</p> <p><b>Reference.</b> Select <i>Reference</i>, then click <b>Edit</b> to define a t-curve by entering both the relative pressure and thickness values. One predefined curve is shipped with the analysis program and is found in the <i>Reference</i> directory.</p> 

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

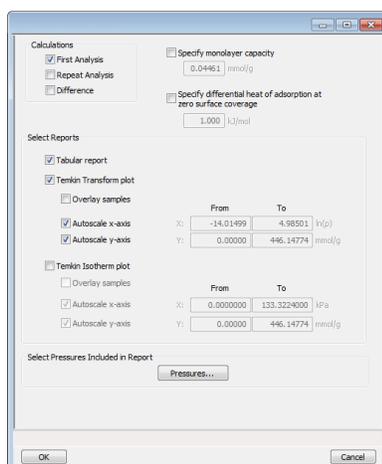
Field or Button	Description
	To import values from an existing thickness curve (.THK file), click <b>Open</b> , then select the file containing the values. The table to be imported must have a .TXT or .THK file extension and have a two-column format with the relative pressures in the first column and the thickness values in the second column. Columns must be separated by a space or a tab.
<b>t-Plot</b> [ <i>check box</i> ]	<p>Use to have a graphical representation of data generated.</p> <p><b>Autoscale x-axis.</b> The X-axis field shows the statistical thickness of the adsorbed film.</p> <p><b>Autoscale y-axis.</b> The Y-axis field shows the quantity of gas adsorbed.</p> <p><b>Overlay samples.</b> Use to overlay sample files on the <i>t</i>-plot.</p>
	<p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>

## TEMKIN REPORT OPTIONS

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

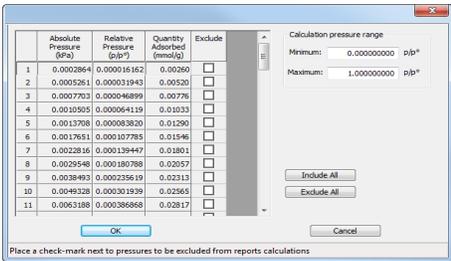


Physical Adsorption

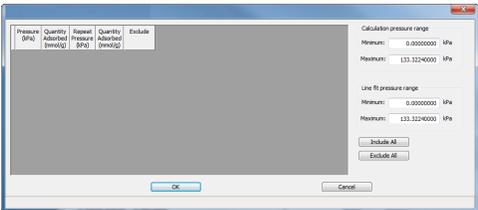


Chemical Adsorption

### Temkin Report Options Fields and Buttons Table

Field or Button	Description
<b>Calculation</b> [ <i>group box</i> ] <b>C</b>	Select one or more of the calculation options to be used for analysis.
<b>Pressures</b> [ <i>button</i> ] <b>P</b>	Use to select a pressure range for report calculations and points for exclusion from calculations.   <p>Place a check-mark next to pressures to be excluded from reports calculations</p> <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table. To exclude a point from the calculations used to generate the report, select <i>Exclude</i>.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p>

## Temkin Report Options Fields and Buttons Table (continued)

Field or Button	Description
<b>Pressures</b> [button] 	<p>Use to enter a range of pressure points to be included in the report or to modify table values for pressure points.</p>  <p><b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table.</p> <p><b>Exclude All.</b> Select to exclude all pressure points in the table.</p> <p><b>Include All.</b> Select to include all pressure points in the table.</p> <p><b>Line fit pressure range.</b> Enter the minimum and maximum pressures for line fit.</p>
<b>Selected Reports</b> [group box]	<p><b>Tabular Report.</b> Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.</p> <p><b>Temkin Isotherm plot.</b> Overlays the Temkin isotherm with the analysis data.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> Linear X-axes begin at zero. The X-axis field shows the absolute pressure.</li> <li>• <b>Autoscale y-axis.</b> Y-axes begin at zero. The Y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the isotherm plot.</li> </ul> <p><b>Temkin Transform plot.</b> Plots a linear form of the Temkin transform plot.</p> <ul style="list-style-type: none"> <li>• <b>Autoscale x-axis.</b> The X-axis field shows the logarithm of pressure (ln).</li> <li>• <b>Autoscale y-axis.</b> The Y-axis field shows the quantity of gas adsorbed.</li> <li>• <b>Overlay samples.</b> Use to overlay sample files on the transform plot.</li> </ul>

## Temkin Report Options Fields and Buttons Table (continued)

Field or Button	Description
Specify differential heat of adsorption [ <i>check box</i> ]	Select and enter the differential heat of adsorption at zero surface coverage. This allows inclusion of all Temkin constants.
Specify monolayer capacity [ <i>check box</i> ]	Select and enter the monolayer capacity of the sample.
 For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .	

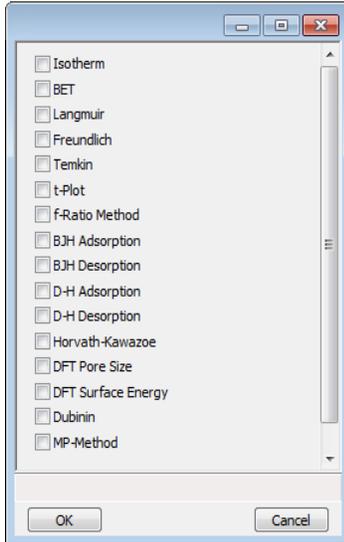
**USER-DEFINED REPORT OPTION**


The *Selected Reports* list box may display a *User-Defined* option rather than an *Advanced* option. These options are the same.

See [Advanced Report Options on page 8 - 3](#)

## ***VALIDATION REPORT OPTIONS***

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

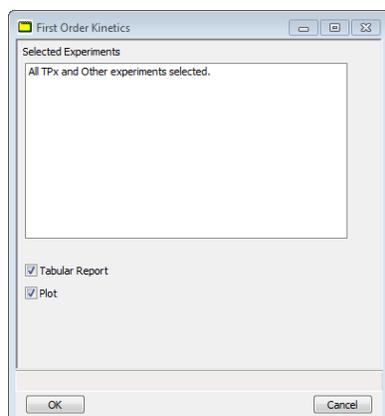


## TCD REPORT OPTIONS FOR DYNAMIC ANALYSIS

### ADVANCED REPORTS

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

### FIRST ORDER KINETICS REPORT OPTIONS



#### First Order Kinetics Report Options Fields and Buttons Table

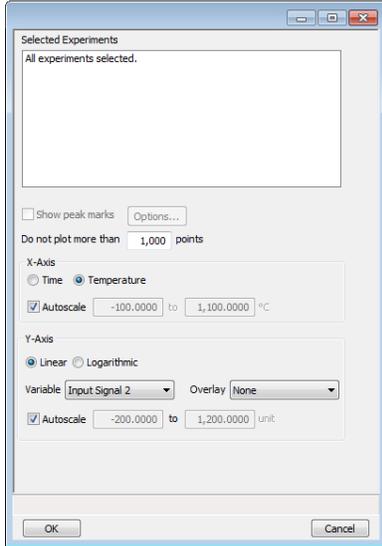
Field or Button	Description
<b>Plot</b> [ <i>check box</i> ]	Select to have the report formatted in a graph format.
<b>Selected Experiments</b> [ <i>group box</i> ]	Displays all related experiments in the current file. Only experiments with collected data are shown.
<b>Tabular Report</b> [ <i>check box</i> ]	Select to have report formatted in table format.



For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 4](#).

## GRAPH REPORT OPTIONS

- TCD Signal vs. Time
- Temperature vs. Time
- TCD Signal vs. Temperature
- TCD Concentration vs. Time
- TCD Concentration vs. Temperature
- TCD Signal and TCD Concentration vs. Time



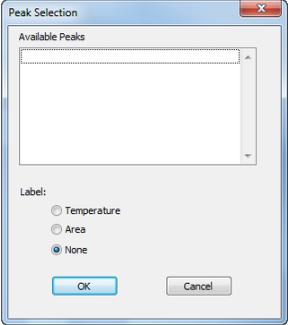
Any signal (the detector signal or the 1 or 2 auxiliary signal) may be plotted against time or temperature. Any signal can be overlaid onto the primary signal.

For color output to a monitor or printer, signals are displayed in different colors. For black and white output, different symbols are used.

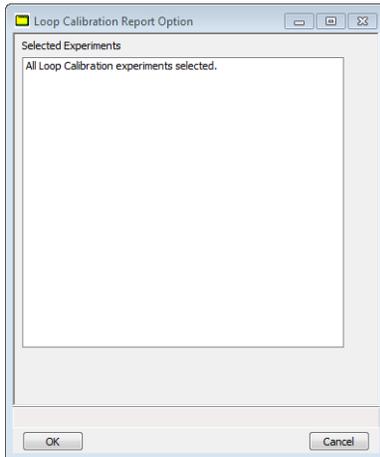
### Graph Report Options Fields and Buttons Table

Field or Button	Description
<b>Do not plot more than [n] points [text box]</b>	Specify a maximum number of points to plot. Plot speed may be affected when a higher resolution is entered.

Graph Report Options Fields and Buttons Table (continued)

Field or Button	Description
<b>Options</b>	<p>Enabled when the <i>Show peak marks</i> option is selected.</p> <p>Lists the peaks for the results of the highlighted experiment. Select any (or all) peaks to include in the graph.</p>  <p>Select the peak labels to display on the graphs.</p>
<b>Selected Experiments</b> [group box]	<p>Displays all related experiments in the current file. Only experiments with collected data are shown.</p> <p>This option is disabled for new files.</p>
<b>Show peak marks</b> [check box]	<ul style="list-style-type: none"> <li>• Enables the <b>Options</b> button to specify the peaks to include in the experiment</li> <li>• Displays the areas and baselines on the graph</li> <li>• Draws a straight baseline between the selected peaks</li> </ul>
<b>X-axis</b> <b>Y-axis</b> [group box]	<p><b>Autoscale.</b> When enabled on the report parameters windows, allows the x- and y- axes to be scaled automatically. <i>Autoscale</i> means that the x- and y- ranges will be set so that all the data is shown. If <i>Autoscale</i> is not selected, the entered range is used.</p> <p><b>Overlay.</b> Select to overlay another variable with the current one.</p> <p><b>Variable.</b> Specify <i>Time</i> or <i>Temperature</i> for the X-axis variable.</p>
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## LOOP CALIBRATION REPORT OPTIONS



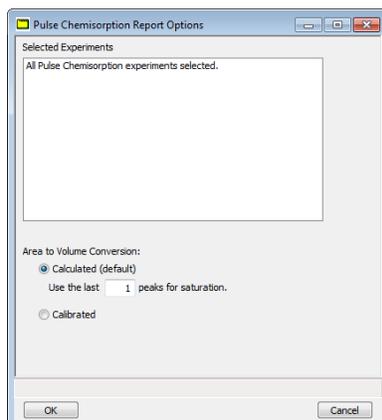
### Loop Calibration Report Options Fields and Buttons Table

Field or Button	Description
<b>Selected Experiments</b>	Displays all related experiments in the current file. Only experiments with collected data are shown.
 For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .	

## OPTIONS REPORT FOR CHEMICAL ADSORPTION

See [Options Report on page 8 - 50](#)

## PULSE CHEMISORPTION REPORT OPTIONS



### Pulse Chemisorption Report Options Fields and Buttons Table

Field or Button	Description
<b>Area to Volume Conversion</b> [group box]	<p><b>Calculated.</b> Uses the standard calculation and the raw signal.</p> $\text{Volume} = \frac{\text{volume injected} \times \text{active concentration}}{\text{area of last peak}}$ <p><b>Calibrated.</b> Uses selected peaks for the conversion. A calibration step must be included in the experiment to use this method.</p>
<b>Selected Experiments</b> [group box]	Displays all related experiments in the current file. Only experiments with collected data are shown.
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## SAMPLE LOG REPORT

Inserts a log of sample operations in the reports. The Sample Log report cannot be edited.

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## 9 DIAGNOSTICS

### Unit [n] > Diagnostics

Use to display diagnostic readings, start and schedule diagnostic tests, and open saved diagnostic reports.

### START DIAGNOSTIC TEST

#### Unit [n] > Diagnostics > Start Diagnostic Test

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



It is recommended to schedule the *Analysis Manifold Leak Test* and the *P<sub>0</sub> Port Leak Test* to run unattended on a weekly basis. These tests check for system leaks and require no operator intervention.



The *P<sub>0</sub> Port Leak Test* should only be run if the Psat tube is attached. If a vapor source is attached, this test should not be run.

#### Start Diagnostic Test Fields and Buttons Table

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.

**Start Diagnostic Test Fields and Buttons Table (continued)**

Field or Button	Description
<b>Next</b> [ <i>button</i> ]	Starts the next test.
<b>Operator</b> [ <i>text box</i> ]	Enter information to identify the person running the service test.
<b>Repeat</b> [ <i>button</i> ]	Repeats the selected diagnostic test.
<b>Report after test</b> [ <i>check box</i> ]	Automatically generates reports to the selected destination when the test is complete.
<b>Sequence</b>	Sequence number assigned to the test.
<b>Start</b> [ <i>button</i> ]	Starts the diagnostic test.
<b>Test</b> [ <i>drop-down box</i> ]	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](#).**

## SCHEDULE DIAGNOSTIC TESTS

### Unit [n] > Diagnostics > Schedule Diagnostic Tests

Allows the specification of one-time or periodic running of a sequence of diagnostic tests. A separate list of tests is saved for each of the possible test frequencies. Tests are categorized and flagged as requiring intervention or not. If tests requiring intervention are scheduled, the operator has the option of omitting the tests if the operator does not respond within a specified time after an initial prompt is displayed and before the test is started. Events are logged in the analyzer log for all starting, ending, and omitted tests.



It is recommended to schedule the *Analysis Manifold Leak Test* and the *P<sub>0</sub> Port Leak Test* to run unattended on a weekly basis. These tests check for system leaks and require no operator intervention.



The *P<sub>0</sub> Port Leak Test* should only be run if the Psat tube is attached. If a vapor source is attached, this test should not be run.

### Schedule Diagnostics Test Frequency Fields and Buttons Table

Field or Button	Description
<b>Available Tests</b> [drop-down box]	Select one or more tests to run unattended.
<b>Insert</b> [button]	Inserts the selected test in the <i>Available Tests</i> drop-down list.

Schedule Diagnostics Test Frequency Fields and Buttons Table (continued)

Field or Button	Description
<b>Skip these tests if the operator does not respond within [n] minutes</b> [ <i>check box</i> ]	Check this option if any test requiring operator intervention should be omitted if the operator does not respond within the specified time.
<b>Start test sequence if instrument is idle any time between 00:00:00 and 00:00:00</b> [ <i>text box</i> ]	Enter a from and to time for an unattended test to begin if the instrument is idle at any time during the entered time frame.
<b>Test Frequency</b> [ <i>selection</i> ]	Select how often the test is to run unattended.
<b>Test Sequence</b> [ <i>group box</i> ]	<p>Provides the test file identification and estimated run time. A checkmark in the <i>Intervention Required</i> column indicates that operator intervention is required.</p> <p>To remove a test from the sequence, select the test, then click <b>Delete</b>.</p> <p>To add a test to the test sequence, highlight a row in the <i>Test Sequence</i> box, select a test from the <i>Available Tests</i> list, then click <b>Insert</b>.</p>
 <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p>	

## DIAGNOSTIC TEST REPORT

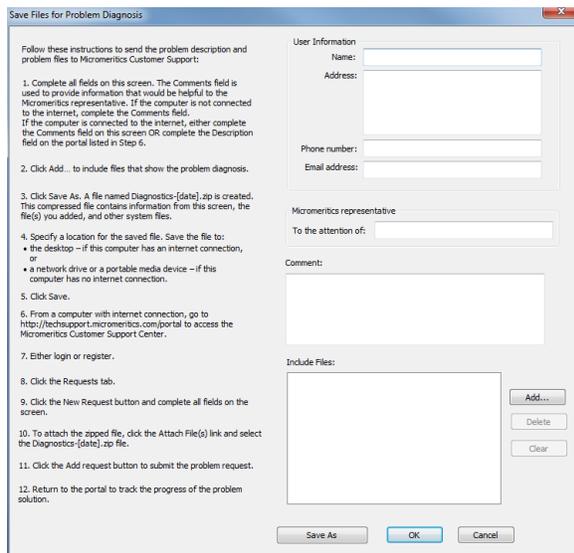
### Unit [n] > Diagnostics > Diagnostic Test Report

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

## SAVE FILES FOR PROBLEM DIAGNOSIS

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

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



1. Complete the form. A default file named *Diagnostics-[date].zip* is created unless another file name is specified.
2. When the file is saved, go to the [Micromeritics Customer Portal](http://techsupport.micromeritics.com/portal) (<http://techsupport.micromeritics.com/portal>) to either log in or register.
3. Click the *Requests* tab.
4. Click **New Request**, then complete all fields.
5. To attach the zipped file, click the *Attach File(s)* link, then select the *Diagnostics-[date].zip* file.
6. Click **Add Request** to submit the problem request.
7. Return to the portal to track the progress of the problem solution.

### Save Files for Problem Diagnostic Fields and Buttons Table

Field or Button	Description
<b>Comment [text box]</b>	Enter information that would be helpful to the Micromeritics representative. If the computer is not connected to the internet, complete this field. If the computer is connected to the internet, this information can be completed on the Micromeritics Customer Support portal.

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

Field or Button	Description
<b>Include Files</b>	<p><b>Add.</b> Click to select additional files to send with this problem diagnosis.</p> <p><b>Delete.</b> Select the file in the <i>Include Files</i> box, then click <b>Delete</b> to remove the file from the list.</p> <p><b>Clear.</b> Click to clear all files from the <i>Include Files</i> box.</p>
<b>Save As</b> [ <i>button</i> ]	<p>Click to specify the name and location of the compressed file. Make a note of the file name and location. This file will need to be sent to your Micromeritics representative for problem resolution.</p>
<b>Micromeritics representative</b> [ <i>text box</i> ]	<p>Enter the name of your Micromeritics representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).</p>
<b>User Information</b> [ <i>text box</i> ]	<p>Enter information for the person to be contacted by a Micromeritics representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).</p>
<div style="border: 1px solid green; padding: 5px;">  <p>For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a>.</p> </div>	

## 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.

All Readings			
<b>Manifold Pressure</b>		<b>Temperatures</b>	
Signal	Nominal	Signal	Nominal
1000 mmHg: 75.520	75.520 kPa	Manifold: 23.5	23.5 °C
10 mmHg: 0.59328	0.59328 kPa	Ports: 24.5	24.5 °C
Piran: 2.84e-001	2.00e-001 kPa	Heater: 28.5	28.5 °C
Cold Cathode: 4.13e-001	4.67e-001 kPa	Ambient: 25.5	25.5 °C
		Upper Cabinet: 29.5	29.5 °C
<b>High-Speed Pressure</b>		<b>Mantle</b>	
Signal	Nominal	Signal	Nominal
1000 mmHg: 75.787	75.787 kPa	Mantle: 26.5	26.5 °C
10 mmHg: 0.72661	0.72661 kPa	Target: 30.6	°C
		Ramp Rate: 2.0	°C/min
<b>Port 1</b>		<b>Port 2</b>	
Signal	Nominal	Signal	Nominal
1000 mmHg: 75.121	75.121 kPa	1000 mmHg: 75.254	75.254 kPa
10 mmHg: 0.19332	0.19332 kPa	10 mmHg: 0.32654	0.32654 kPa
0.1 mmHg: 0.0016459	0.0016459 kPa	0.1 mmHg: 0.1349683	0.1349683 kPa
<b>Port 3</b>		<b>Pi</b>	
Signal	Nominal	Signal	Nominal
1000 mmHg: 75.387	75.387 kPa	1000 mmHg: 75.654	75.654 kPa
10 mmHg: 0.45996	0.45996 kPa		
0.1 mmHg: 0.2682907	0.2682907 kPa		
<b>Electronics</b>			
<b>ADC1</b>			
Temperature:	30.5 °C	Analog 5 V:	0.000 V +15 V: 0.000 V
		Digital 5 V:	0.000 V -15 V: 0.000 V
<b>ADC2</b>			
Temperature:	31.5 °C	Analog 5 V:	0.000 V +15 V: 0.000 V
		Digital 5 V:	0.000 V -15 V: 0.000 V
<b>Mantle Interface</b>			
Temperature:	32.5 °C	Local 5 V:	0.000 V +15 V: 0.000 V
Heater and Mantle:	0.000 Vrms	5 V:	0.000 V -15 V: 0.000 V
<b>Mantle Resistance</b>			
Manifold Heater:	0.0 Ohms	Mantle:	0.0 Ohms

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## 10 CALIBRATION

### Unit [n] > Calibration

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.

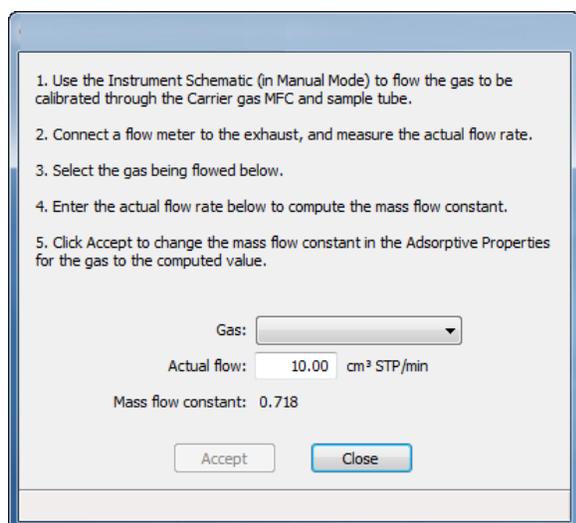
Use to perform system calibrations. Disabled calibration menu options can be accessed only with the assistance of an authorized Micromeritics service representative. Calibrations can be saved to a file and reloaded later.

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

## GAS FLOW CALIBRATION

### Unit [n] > Gas Flow Calibration

See [Gas Charts on page E - 1](#)



1. Use the Instrument Schematic (in Manual Mode) to flow the gas to be calibrated through the Carrier gas MFC and sample tube.

2. Connect a flow meter to the exhaust, and measure the actual flow rate.

3. Select the gas being flowed below.

4. Enter the actual flow rate below to compute the mass flow constant.

5. Click Accept to change the mass flow constant in the Adsorptive Properties for the gas to the computed value.

Gas:

Actual flow:  cm<sup>3</sup> STP/min

Mass flow constant:

Use the *Gas Flow Constant Calibration* option to determine a constant used by the analyzer to ensure accurate gas flows through each Mass Flow Controller (MFC). The Mass Flow Controllers were calibrated before shipping and calibration is typically not necessary.

1. Flow the gas to be calibrated through the Carrier gas MFC and sample tube. Go to **Unit [n] > Enable Manual Control**. Ensure a checkmark displays to the left of the menu item. If the instrument schematic does not display, go to **Unit [n] > Show Instrument Schematic**.
2. Connect a flow meter to the exhaust and measure the actual flow rate.
3. Select the gas being flowed.
4. Enter the actual flow rate to compute the mass flow constant.

5. Click **Accept** to change the mass flow constant in the Adsorptive Properties for the gas to be computed value.

## **MASS FLOW CONTROLLER CALIBRATION**

See [Gas Charts on page E - 1](#)

The analyzer uses Mass Flow Controllers (MFCs) to control the flow of gases. These MFCs require a conversion constant for each gas or gas mixture, to compensate for variations in gas flows resulting from variations in the gases' properties.

In most cases, the default MFC conversion constant yields accurate data. A new conversion constant may be used if:

- A unique gas mixture is used
- The gas to be used is not included in the table
- A higher precision calibration of the MFCs for a given gas is required
- Unexpected analysis data lead you to believe that the default value is in error

## **FLOW MEASUREMENT**

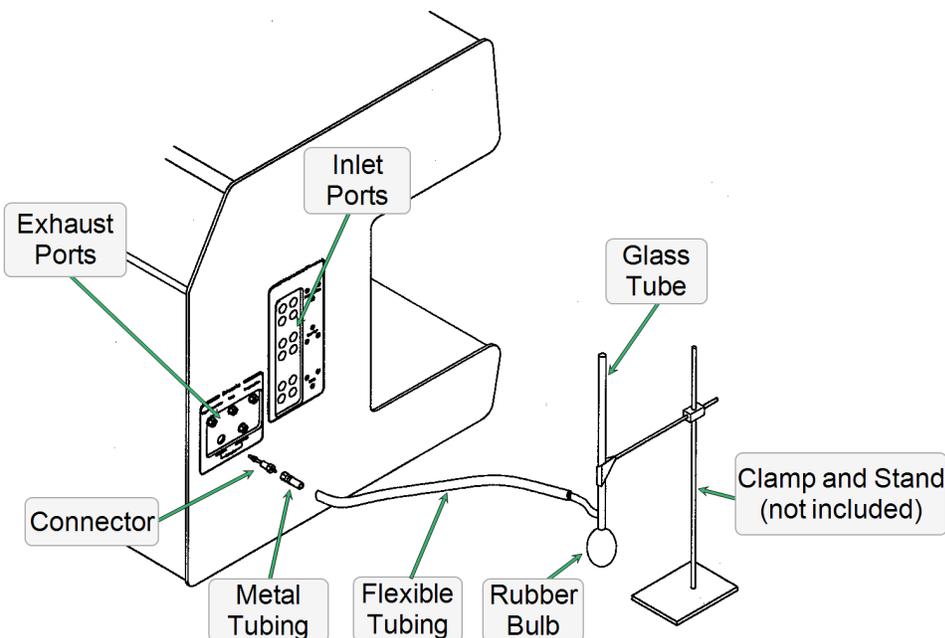
An external device that measures the flow of gas from the analyzer's exhaust is used during MFC calibration. A soap bubble burette is shipped with the analyzer. For even more precise calibration, use another type of flow meter and follow these instructions for obtaining a new conversion constant, substituting your flow meter for the bubble burette.

## **INSTALL THE SOAP BUBBLE BURETTE**



These instructions are for assembling and using the burette supplied by Micromeritics. If using another type of flow meter, follow the meter manufacturer's assembly and operation instructions.

1. Carefully unwrap the glass tube and the rubber bulb.
2. Attach one end of the flexible tubing to the side arm of the glass tube.
3. Attach the metal tubing provided with the bubble burette to the other end of the flexible tubing.



4. Locate the exhaust port that corresponds to the MFC to be used for this calibration. Remove external plumbing from that port and attach the bubble burette using the connector provided with the burette. For example, if using the Carrier Flow Controller, attach the bubble burette to the carrier gas exhaust port.
5. Fill the rubber bulb with the contents of the 8 oz. bottle of the leak detector fluid included in the accessory kit.
6. Attach the bulb to the bottom of the glass tube.



The soap bubble burette must be held in an upright position.

## ***DETERMINE THE CONSTANT FOR INDIVIDUAL GASES***

### ***Unit [n] > Gas Flow Calibration***

1. Go to ***Unit [n] > Enable Manual Control***. Ensure a checkmark displays to the left of the menu item. If the instrument schematic does not display, go to ***Unit [n] > Show Instrument Schematic***. Flow the gas to be calibrated through the Carrier gas MFC and sample tube.
2. Connect a flow meter to the exhaust and measure the actual flow rate. If using the bubble burette shipped with the analyzer, observe a bubble as it rises through the glass tube. The lines on the glass tube indicate the beginning and ending points for measuring the progress of a bubble through the tube. Use a stopwatch to measure the amount of time that elapses from the moment the bubble passes the lower mark on the tube until the moment it passes the higher mark on the tube.
3. Go to ***Unit [n] > Gas Flow Calibration*** and select the gas being flowed.

1. Use the Instrument Schematic (in Manual Mode) to flow the gas to be calibrated through the Carrier gas MFC and sample tube.

2. Connect a flow meter to the exhaust, and measure the actual flow rate.

3. Select the gas being flowed below.

4. Enter the actual flow rate below to compute the mass flow constant.

5. Click Accept to change the mass flow constant in the Adsorptive Properties for the gas to the computed value.

Gas:

Actual flow:  cm<sup>3</sup> STP/min

Mass flow constant: 0.718

4. Enter the actual flow rate in the *Actual flow* field to compute the mass flow constant.
5. Click **Accept** to change the *Mass flow constant* field in the *Adsorptive Properties* for the gas to the computed value.
6. Use the attach the gas to be calibrated to an inlet port for the MFC to be used during calibration.
7. On the instrument schematic, right click the *Flow Controller* icon. Select *Set* then enter the *Set flow rate*. Click **OK**.

Automatically set isolation valves

Open

Close

Set flow rate:  sccm

Mass flow controller constant: 1.000

Enter a value between 0.0 and 200.0.



The added gas can now be selected for any port in **Unit [n] > Unit Configuration**. If planning to leave this gas connected to the port, change the gas selection for that port to the correct name.

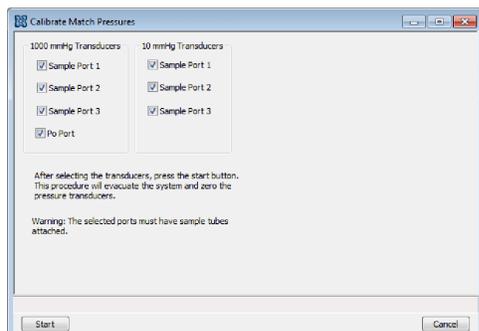
## MATCH TRANSDUCERS

### 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 **Cancel** to stop the calibration process.

### Match Transducers Fields and Buttons Table

Field or Button	Description
<b>1000 mmHg Transducers</b>	Select the ports.
<b>10 mmHg Transducers</b>	Select the ports. Enabled only for ports with 10 mmHg transducers present.

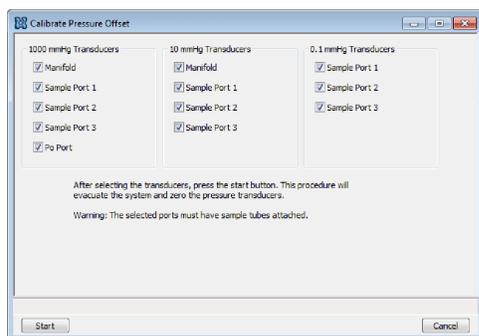


For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 4](#).

## 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**. The window closes when the operation is complete. Click **Cancel** to stop the calibration process.

### Pressure Offset Fields and Buttons Table

Field or Button	Description
<b>1000 mmHg Transducers</b>	Select the manifold and/or ports.
<b>10 mmHg Transducers</b>	Select the manifold and/or ports. Enabled only for the manifold and ports with 10 mmHg transducers present.
<b>0.1 mmHg Transducers</b>	Select the ports. Enabled only for ports with 0.1 mmHg transducers present.

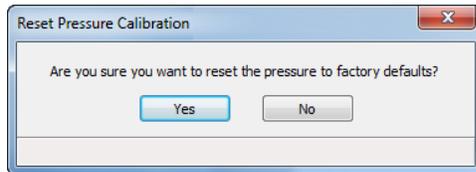


For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 4](#).

## RESET PRESSURE CALIBRATION

### Unit [n] > Calibration > Reset Pressure Calibration

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



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

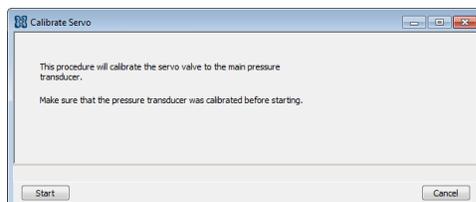
## SERVO VALVE

### 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 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.

## **TCD CALIBRATION**

Analyses yield data on signal reading, peak area, temperature, and time. These data are sufficient for many applications, however volume data may also be needed.

It is not necessary to perform a calibration if volume data are not needed. If volume data are needed, calibration may be performed either before or after the analysis. **NOTE:** This does **not** apply to Loop Calibration, Ambient Temperature, and Atmospheric Pressure. The correct values for these quantities must be entered under **Unit [n] > Unit Configuration** and **Options > Environment Defaults** before starting an analysis that uses them to determine the quantity of gas in an injection.

A group of automatic calibration routines are provided in the form of specialized experiment steps. A calibration run is an analysis using one of these experiment steps. The calibration run can be performed before or after the sample analysis and can be included as a step within the analysis, or it can be performed as a separate analysis.

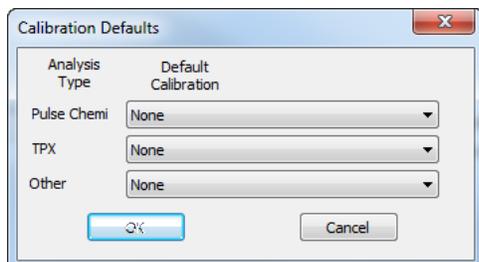
After the sample data and calibration data are collected, the calibration file is associated with the sample file, and the sample data are converted to volume. A single calibration run can be associated with an unlimited number of sample data files. For example — TPR yields peak area and the temperature(s) at which maximum reduction occurs. To obtain the volume of gas uptake, a calibration file must be associated with the analysis file, then reports are created in which the area data are converted to volume data.

A calibration file can be associated with a sample file by:

- going to **Unit [n] > TCD Calibration > Defaults** and selecting a default calibration file for each experiment type,
- using the default file or choose a different file in the *Start Analysis* window.
- clicking **Set Calibration** on the *Peak Editor* window and select from a list of calibration files that were created after the sample file was used in an analysis.

## TCD CALIBRATION DEFAULTS

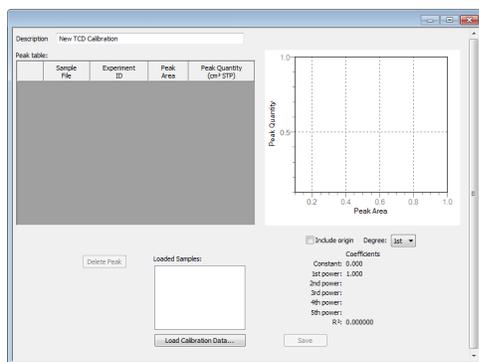
Unit [n] > TCD Calibration > Defaults



Use to specify default calibration files for each type of analysis. Use the *Other* drop-down list to select defaults for custom analyses.

## CREATE A NEW TCD CALIBRATION FILE

Unit [n] > TCD Calibration > New



Repeat this process for each gas concentration in the calibration. For example — if gas concentrations of 10%, 20%, and 30% are used, mark the calibration peaks and create calibration files for each concentration.

Calculation used for peak volume:

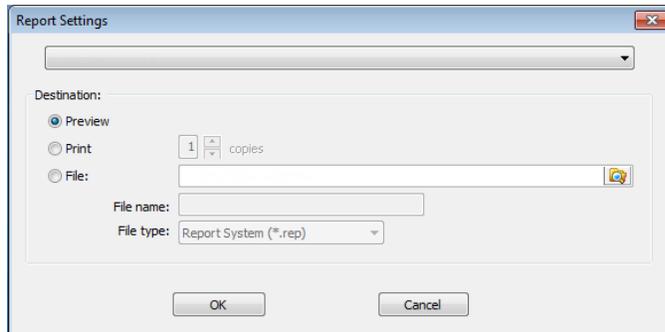
$$V_{syrinj} \times \frac{273.15}{T_{amb} + 273.15} \times \frac{P_{amb}}{760 \text{ mmHg}} = V_{Peak}$$

For example, calculations for 1 cm<sup>3</sup> injection, ambient temperature of 22 °C, ambient pressure of 740 mmHg:

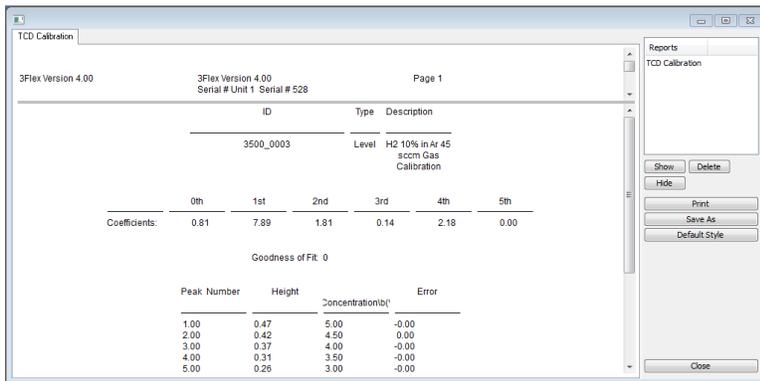
$$1 \times \frac{273.15}{22 + 273.15} \times \frac{740}{760 \text{ mmHg}} = 0.901 \text{ cm}^3 \text{ STP}$$

## CREATE A TCD CALIBRATION REPORT

**Unit [n] > TCD Calibration > Report**



Select a previously defined TCD calibration file from the drop-down list.



ID	Type	Description
3500_0003	Level	H2 10% in Ar 45 sccm Gas Calibration

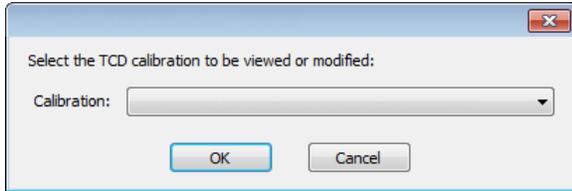
	0th	1st	2nd	3rd	4th	5th
Coefficients:	0.81	7.89	1.81	0.14	2.18	0.00

Goodness of Fit: 0

Peak Number	Height	Concentration(b)	Error
1.00	0.47	5.00	-0.00
2.00	0.42	4.50	0.00
3.00	0.27	4.00	-0.00
4.00	0.31	3.50	-0.00
5.00	0.26	3.00	-0.00

## OPEN A TCD CALIBRATION FILE

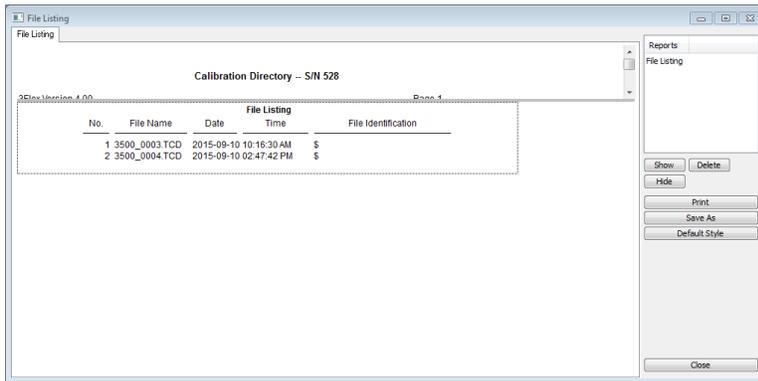
**Unit [n] > TCD Calibration > Open > .Cal File]**



Select a previously defined TCD calibration file from the drop-down list.

## LIST TCD CALIBRATION FILES

**Unit [n] > TCD Calibration > List**



Displays a list view of previously defined TCD calibrations.

## LOOP CALIBRATION FOR TCD ANALYZERS

See [Proper Use of the Septum on page 11-9](#)

Each calibration loop must be calibrated prior to its first use to determine its precise volume under local conditions. Calibration consists of:

- Determining the average area of a series of peaks generated by injections of a known volume of gas through the analyzer septum using a syringe.
- Determining the average area of a series of peaks generated by injections of the same gas using the analyzer's internal loop.
- Calculating the volume of the loop by comparing the average peak area generated by the loop injections with that generated by the syringe injections.
- Entering the calculated loop volume under **Unit [n] > Unit Configuration**.

### STEP 1: UPDATE AMBIENT PRESSURE AND TEMPERATURE

#### **Options > Environmental Defaults**

See [Environmental Defaults for TCD Analyzers on page 2 - 20](#)

Ambient pressure and temperature at the time of the analysis affect the results of a loop calibration. In calculations, the analyzer uses the ambient pressure and temperature recorded in *Environmental Defaults*. Check and update the *Environmental Defaults* before beginning a loop calibration.

### STEP 2: CREATE A SAMPLE FILE

Create the sample file and insert a *Loop Calibration* experiment on the *Analysis Conditions* tab.



A loop calibration must be created for each loop. For Loop Calibration, select a carrier gas, flow rate, and loop gas that will commonly be used for sample analyses.

### STEP 3: PERFORM AN ANALYSIS

1. Install the correct injection loop.
2. Go to **Unit > Start Analysis** and select the sample file for the loop calibration.
3. Click **Next** to accept the default values, then click **Start**.
4. Follow the prompts to make the selected number of injections. Use a volume that is close to the volume of the loop being calibrated. For example, if calibrating the 1 cc loop, use a 1 cc syringe and inject as close to 1 cc of gas as possible.



Pay close attention to the instructions provided in each prompt and perform the steps in the order given. Most accurate data results from keeping injection size as consistent as possible. Injection errors may be evident in the data and may make it necessary to repeat the experiment.

After the last manual injection, the analyzer automatically makes the same number of injections using the loop.

#### **STEP 4: GENERATE THE REPORT**

See [Peak Editor for Dynamic Analysis on page 7 - 11](#)

When properly performed, each manual and automatic injection results in a peak. When the *Results* view of the *Start Analysis* window is selected, each peak can be viewed as it is collected. The area under the peak corresponds to the amount of gas injected.

1. In the *Peak Editor*, open the sample file to ensure the peaks are properly marked.
2. Go to **File > Open > [.SMP]** and open the .SMP file containing the calibration experiment and verify (or correct) the defined peaks using the *MicroActive Peak Editor*.
3. On the *Reports* tab, select only the *Loop Calibration* report option.
4. Go to **Unit > Unit Configuration**. In the *Loop volume* field, enter the *Loop Volume*.



Verify that the *Loop Volume* and *Environmental Default* values are correct prior to starting an analysis with injections.

## ***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]ast.cal*. The backup file is overwritten each time a new one is created.



Changing the calibration may affect the analyzer's performance.

## ***SAVE CALIBRATION TO FILE***

### ***Unit [n] > Calibration > Save to File***

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

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

<b>0217</b>	is the analyzer serial number
<b>2016-04-25</b>	is the date the calibration file was saved
<b>.CAL</b>	is the file name extension

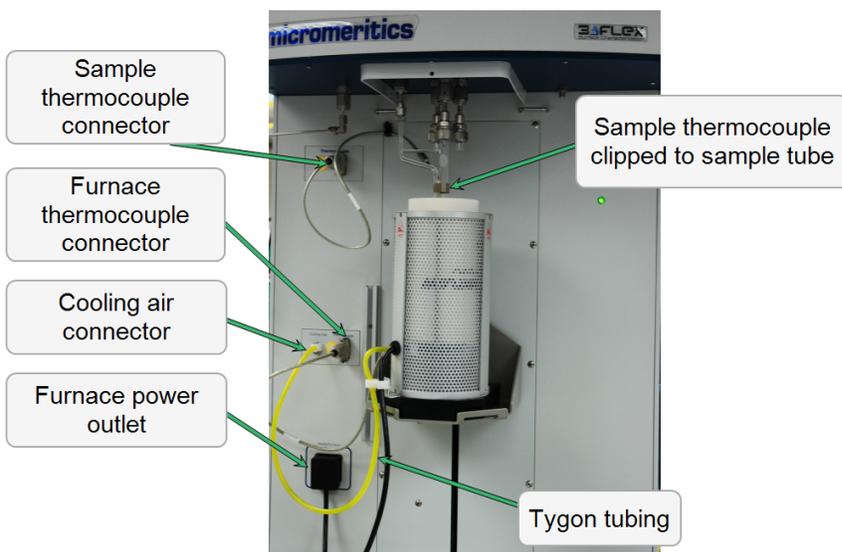
## 11 HARDWARE

### FURNACE INSTALLATION



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

The furnace uses same supply of compressed air as the instrument. There is an internal regulator that has been set at the factory for the correct flow rate of cooling air to the furnace.



NOTE: This photo is shown without the safety shield. The safety shield should always be installed prior to running an analysis.

1. Place and center the furnace on the elevator.
2. Insert the furnace power cable into the power connector.
3. Connect the furnace thermocouple plug into the *Thermocouple* connector on the front of the analyzer.
4. Connect the cooling air tube into the *Cooling Air* connector on the front of the analyzer.

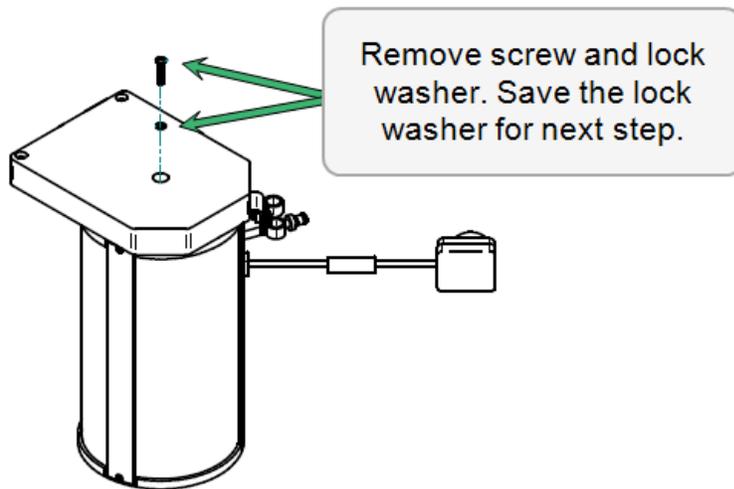
## SECURE FURNACE TO ELEVATOR TRAY



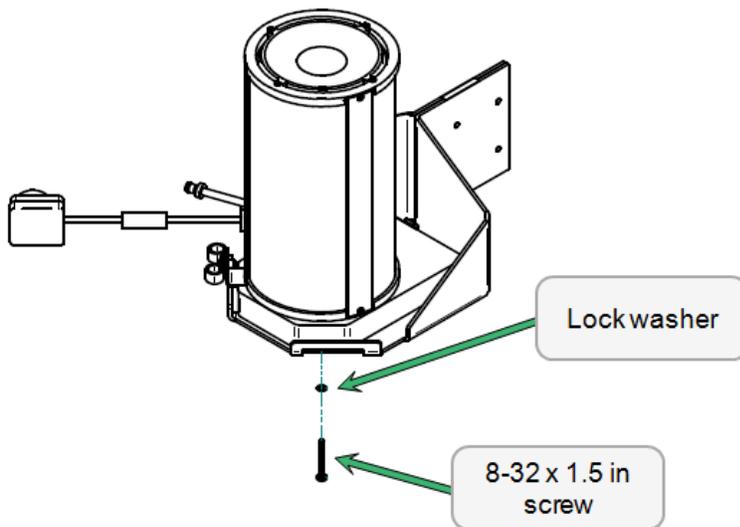
This is an optional procedure for securing the furnace to the elevator tray in areas prone to earthquakes.

Replace the screw that holds the black plastic base to the furnace and replace it with a 8-32 × 1.5 in. screw (part #004-28622-00).

1. Invert the furnace and remove the screw that holds the black plastic base to the bottom of the furnace. Save the lock washer and use it when inserting the longer screw.



2. Place the black plastic base onto the elevator aligning the hole in the base with the hole in the elevator.
3. Position the furnace on top of the black plastic base aligning the hole in the furnace bottom with the hole in the furnace base.



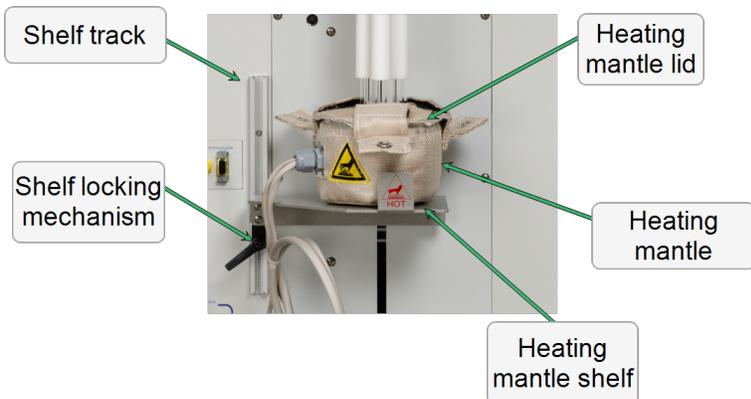
4. Raise the elevator. Position the lock washer in the hole in the elevator tray. Insert the 8-32 × 1.5 in. screw through the lock washer, the hole in the elevator, the black plastic base, and into the furnace.

## HEATING MANTLE INSTALLATION

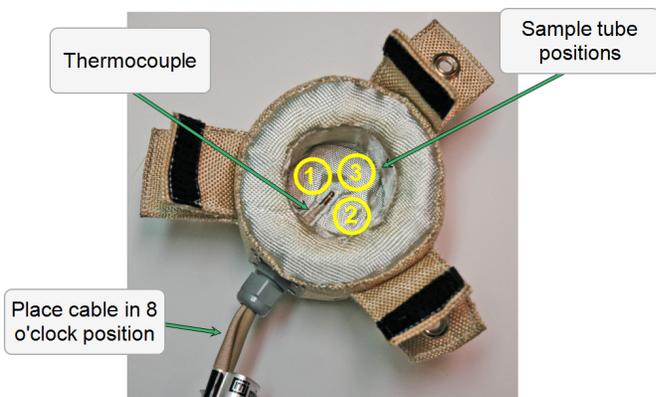
See [Pivot Shelf on page 11-8](#)



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



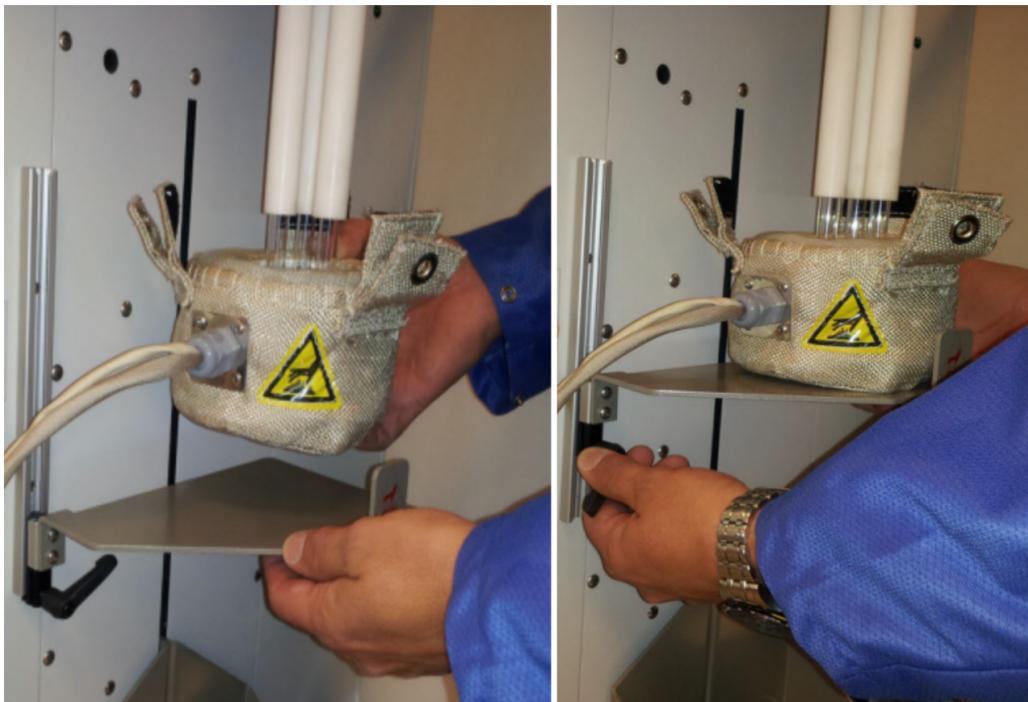
If using less than three sample tubes, position the heating mantle cable in the 8 o'clock position when resting on the shelf surface. Ensure that the bottom of all sample tubes are in full contact with the bottom of the heating mantle interior surface only and **DO NOT** touch the thermocouple located on the bottom surface of the mantle's interior. A single sample tube must be installed on port 2.



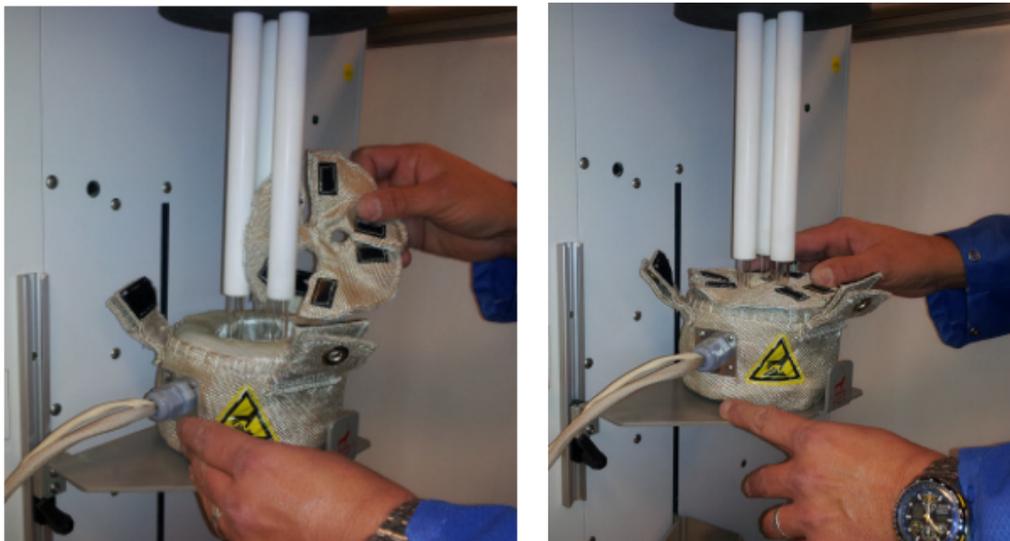
1. Place the mantle around the sample tube bulbs. Ensure that the isothermal jackets are pushed up against the dewar lid to avoid damage to the jackets.



2. While supporting the heating mantle with one hand, slide the shelf locking mechanism into the shelf track. Raise the shelf on the track until the heating mantle rests securely on the shelf and the sample tubes touch the bottom of the inside of the heating mantle. Turn the locking mechanism clockwise to secure the shelf.

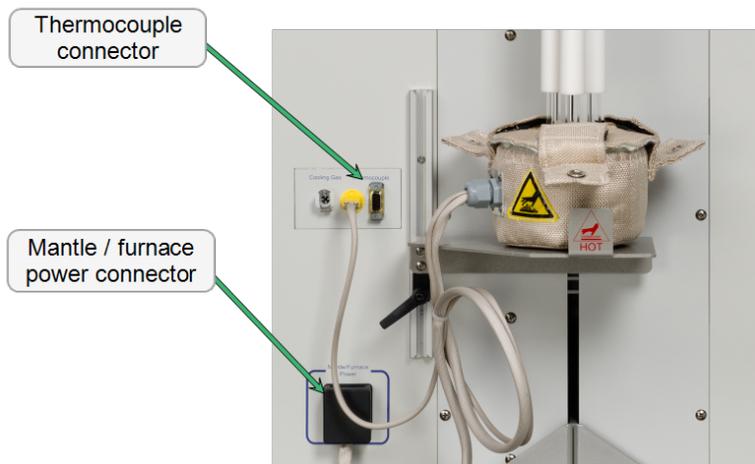


3. Slide the heating mantle cover between the sample tube bulbs and the bottom of the isothermal jackets so that the sample tubes fit within the slots of the mantle cover.



Do not to apply force to the tubes while installing the lid.

4. Secure the heating mantle tabs onto the hook and loop fasteners of the heating mantle cover. Ensure there is at least a 1/2 in. (12 mm) gap between the top of the mantle cover and the bottom of the isothermal jackets. This will prevent damage to the jackets. Replace any damaged jackets.
5. Insert the mantle thermocouple and the mantle power plug into the analyzer's front panel.



6. Acknowledge the prompt on the *Sample Analysis* window. The degas will proceed. When the degas is completed and the mantle has cooled below 45 °C, the *Sample Analysis* window will submit a prompt to remove the degas heating mantle and shelf, properly position the isothermal jackets and dewar lid, and install the dewar.



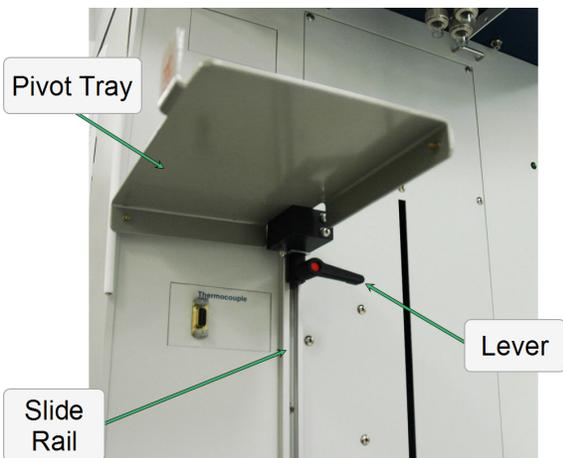
To prevent potential burns, do not touch the sample tube or the heating mantle until they have cooled.

---

7. To remove the heating mantle, take off the heating mantle cover, support the bottom of the heating mantle, then lower the shelf. The shelf must be removed prior to installing the dewar.

## ***PIVOT SHELF***

A pivot shelf is used to support the vapor source container or the heating mantle. It can be pivoted for installation, removal, or height adjustment.



### ***INSTALL THE PIVOT SHELF***

1. Hold the pivot shelf in your left hand and the pivot block and lever (underneath the shelf) with your right hand.
2. Pivot the shelf to the left to expose the block and lever.
3. Insert the slide block into the slide rail on the front of the analyzer.
4. To tighten the shelf into position, start with the lever to the right, push and turn the lever clockwise. Release the lever, then move the lever back to the right position and again push and turn the lever clockwise. Continue this process until the pivot shelf is snug in the slide rail.

### ***REMOVE OR ADJUST THE PIVOT SHELF***

1. Pivot the pivot shelf to the left to expose the pivot block and lever underneath the shelf.
2. Hold the pivot shelf in your left hand and the pivot block lever with your right hand.
3. To loosen the tension on the pivot shelf, start with the lever to the left, push and turn the lever counterclockwise. Release the lever, then move the lever back to the left position and again push and turn the lever counterclockwise. Continue this process until the pivot shelf can move on the slide rail.

## PROPER USE OF THE SEPTUM

See [Replace a Septum on page 12 - 30](#)

The septum is used to inject quantities of gas into the analyzer. The septum is located underneath the top panel of the instrument.



Accuracy of data is diminished when poor techniques are used for injecting gas through the septum.

Injecting the gas through the septum causes a peak to appear, but it also causes a perturbation in the flow of gas through the analyzer. This perturbation is visible in the peak data. To minimize this perturbation, inject the gas more slowly into the septum. Prolonging the injection causes the peak to spread.

An injection method should be developed to balance the need to minimize the perturbation with the need for sharper peaks. See [Peak Editor for Dynamic Analysis on page 7 - 11](#) to adjust peak data to reduce the effects of perturbation.

- Always hold the syringe by its metal parts away from the needle. Holding the syringe by the glass allows body heat to affect the volume of gas in the syringe.
- After filling the syringe, to ensure that the syringe and its contents are at room temperature, allow the syringe to lie on a room temperature surface for about a minute.
- If the gas used is lighter than air, do not allow the filled syringe to remain in a vertical position (needle up) — the gas will diffuse out and the total volume will be reduced.
- To fill the syringe, first empty it completely, then insert it into a septum accessory installed on the gas regulator. Draw the syringe plunger back until the syringe is completely filled with gas.

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Remove the syringe from the septum and allow it to return to room temperature. Press the plunger into the syringe until the correct amount of gas is contained in the syringe.

- Ensure the needle is inserted fully into the septum on the analyzer.
- Press the plunger into the syringe completely to ensure that the entire quantity of gas is injected from the syringe.

## VAPOR SOURCE CONTAINER INSTALLATION

See [Vapor Purification on page J - 1](#)

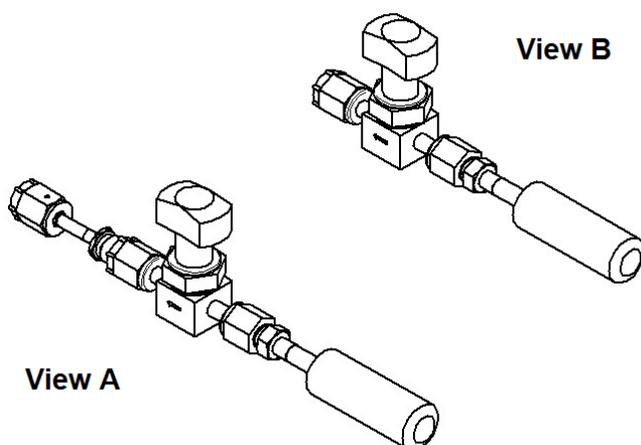


This device has been designed to be used for sample analysis via the analyzer control panel. Any other use may damage this device or the analyzer. Each time the Psat tube or vapor source container is replaced, a new gasket is required. Do not touch the sealing surfaces or the port fitting or gasket with bare hands.

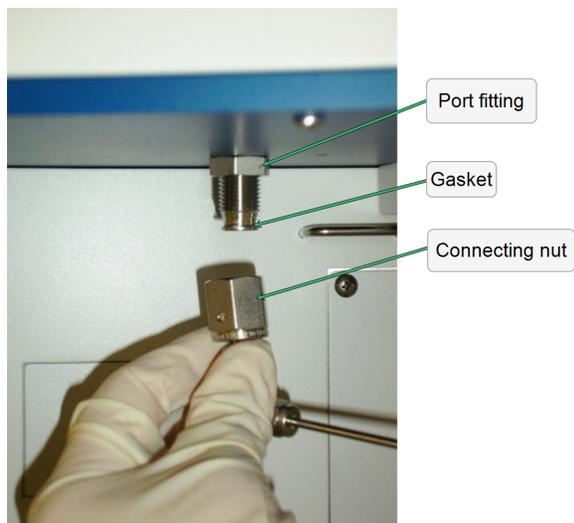
For chemical adsorption only the vapor mantle or the sample furnace can be connected at the time of analysis.



Your equipment may differ slightly. The photos in this section use View A, however, View B is installed in the same manner as View A. If using View B, the Vapor Source Mantle will be slightly shorter than shown in this section.



1. Use an appropriate wrench to loosen the connecting nut from the port fitting by turning the connecting nut counter-clockwise while using a second wrench to hold the port fitting stationary.

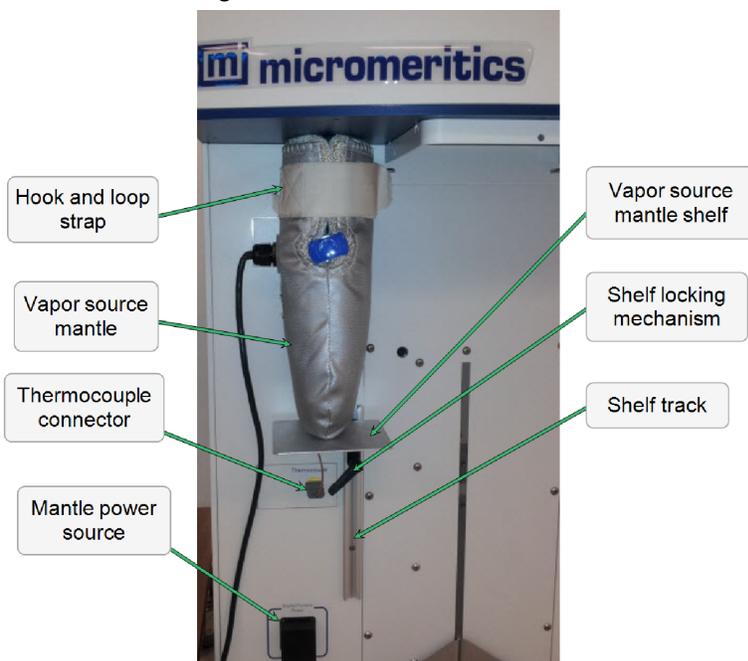


2. Remove the connecting nut and the attached assembly. After removal, the existing seal or a tight-fitting plastic cap can be used to protect the sealing surface assembly from scratches. Prior to reassembly, remove the existing seal or cap, then insert a new seal.
3. Install the vapor source container with a new seal by attaching the connecting nut to the port fitting. Hand tighten the connecting nut by turning clockwise. Use an appropriate size wrench to tighten the assembly an additional 1/8 to 1/4 turn beyond finger tight, while using a second wrench to hold the port fitting stationary on the analyzer.



Turn the vapor source isolation valve to adjust the vapor flow

- Use the manual controls on the analyzer schematic, evacuate the space above the vapor source by opening valves 4 and 6 with all other valves closed. Then close valve 4 before turning the vapor source isolation valve to the vertical (open) position. The Po / vapor port pressure reading on the instrument schematic will show the vapor pressure.



- Slide the vapor source mantle over the vapor source container. Extend the blue knob through the circular hole. Secure the hook and loop strap.
- Insert the thermocouple plug into the connector labeled *Thermocouple*. Insert the power plug into the outlet labeled *Mantle/Furnace Power*.
- If using a support shelf, slide the shelf locking mechanism of the vapor source mantle shelf into the shelf track on the front of the analyzer. Raise the shelf until the vapor mantle is pushed as close as possible to the underside of the upper cabinet. To tighten the shelf, turn the locking mechanism clockwise.

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## 12 TROUBLESHOOTING AND MAINTENANCE

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The instrument has been designed to provide efficient and continuous service; however, certain maintenance procedures should be followed to obtain the best results over the longest period of time. When unexpected results occur, some common operational problems not indicated on the window and their respective causes and solutions are provided:

See [3Flex Links on page iv](#) for a link to error messages.

Most operational problems are caused by:

- Leaks (commonly found at the sample tube O-ring at the analysis port)
- Sample weighing errors
- Use of too much analysis bath fluid in the dewar at the start of an analysis
- Entry of incorrect system volume for analysis
- Impure gas supply

When unexpected analysis results occur, check the above first. Some common operational problems not indicated on the window and their respective causes and solutions are provided below:

### **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.

### **Sample is not within specifications.**

*Cause A:* There may be a manifold leak.

*Action A:* See [Start Diagnostic Test on page 9 - 1](#).

*Cause B:* Gas may be contaminated.

*Action B:*

- Perform a blank analysis. If results are good, perform a reference material analysis.
- Replace tank.
- Check for line leak, which could cause contamination.
- Flush the lines occasionally to help prevent contamination.

*Cause C:* Incorrect type of gas line.

*Action C:* Ensure the gas line is all metal. It is best to use the one shipped with the analyzer. Do not use polymer gas lines or flexible gas lines that may be coated internally with a polymer.

### **High vacuum pump indicator light does not come on.**

*Cause:* No power to the high vacuum pump.

*Action:* Remove the lower panel on the front of the instrument. Check the power supply plug to the pump. Power off the high vacuum pump, then power it back on.

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

*Cause A:* Port filter is dirty.

*Action A:* Replace the port filter.

*Cause B:* No power to the vacuum pump.

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

#### **For oil based pumps:**

*Cause A:* Port filter is dirty.

*Action A:* Replace the port filter.

*Cause B:* Vacuum pump oil is low causing ineffective evacuation

*Action B:* Add or change vacuum pump oil. Add oil to proper level according to the pump's indicator window.

*Cause C:* Alumina in the oil vapor trap is holding moisture because it was not sufficiently dried before being added to the alumina trap.

*Action C:* Replace or dry the alumina.

#### **For oil free pumps:**

*Cause A:* High vacuum pump may have timed out.

*Action A:* Remove the lower front panel from the analyzer. Power off the high vacuum pump, then power it back on.

*Cause B:* The pump diaphragm is worn or damaged.

*Action B:* Contact your Micromeritics service representative.

### **Vacuum pump is noisy.**

*Cause A:* Sample tube connector is loose.

*Action A:* Tighten fitting. Replace O-ring.

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

*Action B:* Replace O-ring. See [Replace the Sample Port Frit on page 12 - 25](#).

*Cause C:* Sample tube is cracked.

*Action C:* Replace with new sample tube.

*Cause D:* No sample tube loaded on a selected port.

*Action D:* Install plug or empty sample tube.

*Cause E:* Gas inlet valve open while vacuum valve open.

*Action E:* With manual control enabled, use the instrument schematic to close gas inlet valve.

### **Vacuum pump system makes loud continuous noise.**

*Cause A:* Sample tube connector nut is loose.

*Action A:* Turn the connector nut clockwise to tighten.

*Cause B:* Sample tube fitting is loose.

*Action B:* Tighten the fitting using a wrench.

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

*Action C:* Replace sample tube O-ring.

*Cause D:* Sample tube is cracked.

*Action D:* Replace the sample tube.

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

*Action E:* Ensure 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 [Show Instrument Schematic on page 2 - 21](#).

### **Valves cannot be operated.**

*Cause A:* Cable from computer to the instrument is loose.

*Action A:* Reconnect the cable.

*Cause B:* Circuit was opened by the circuit breaker.

*Action B:* Press the **Breaker** button located on the side of the analyzer near the power entrance. Contact your Micromeritics service representative if the button will not stay depressed.

**A stable TCD baseline cannot be maintained.**

*Cause A:* TCD filaments are contaminated or need to be replaced.

*Action A:* See [TCD Assembly on page 12 - 31](#) to clean the TCD filaments. Contact your Micromeritics service representative if necessary.

*Cause B:* Possible leak in reference/carrier path.

*Action B:* Perform leak test in the reference/carrier path. See [Perform a Leak Test on page 12 - 17](#).

**Data collection results in very high or very low peaks that are inconsistent with previous experience.**

*Cause A:* TCD filaments are contaminated or need to be replaced.

*Action A:* See [TCD Assembly on page 12 - 31](#) to clean the TCD filaments. Contact your Micromeritics service representative if necessary.

*Cause B:* A different (lower) TCD filament temperature is being used.

*Action B:* Repeat the analysis using a higher filament temperature.

**During data collection, a TCD signal of zero is recorded, or no peaks are seen.**

*Cause A:* TCD filaments are contaminated or need to be replaced.

*Action A:* See [TCD Assembly on page 12 - 31](#) to clean the TCD filaments. Contact your Micromeritics service representative if necessary.

*Cause B:* TCD temperature was not reset after a run automatically suspended.

*Action B:* Re-enable the TCD after all run suspensions.

## ***CLEAN THE POWER SUPPLY AIR FILTER***

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

1. Use a flat blade screwdriver to pry the air filter cover from the base. Do not remove the screws. Use caution when removing the cover to avoid breakage.

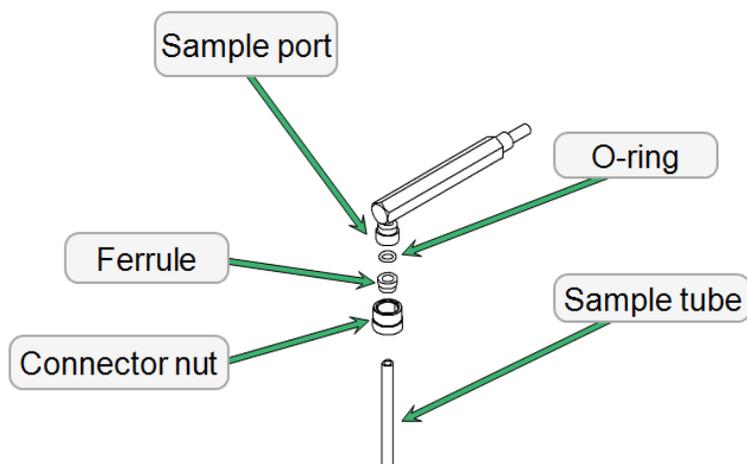


2. Use an air compressor to remove the dust, or rinse with tap water and dry thoroughly.
3. Replace the filter and cover by pressing the cover back into the base.

## ***COLD TRAP TUBE***

A Cold Trap option is available. See [3Flex Links on page iv](#) for links to parts and accessories.

### ***CHANGE THE COLD TRAP TUBE***



#### **To remove the cold trap tube**

1. Loosen the connector nut on each stem of the cold trap tube.
2. Gently pull down on the cold trap tube to remove it from the ports. The connector nut and ferrule will remain on the cold trap tube stems. If the O-ring remains on the cold trap tube stem, examine the O-ring to ensure it is still in good condition prior to re-inserting it into the port. Replace the O-ring, if necessary. If the O-ring remains in the port, it may be gently pried out for replacement.

#### **To Install the cold trap tube**

1. Reassemble the cold trap tube. On each stem, assemble the connector nut, a ferrule (large side pointing toward the port), and an O-ring.
2. Slide each stem fully into the port, then tighten the connector nuts.

## ***PREPARE THE COLD TRAP FOR IDLE PERIODS***

If the analyzer is to be inoperative or unattended long enough for the liquid nitrogen in the cold trap dewar to evaporate (or the dry ice to evaporate from the dry ice acetone slurry), remove the dewar and U-tube so that impurities are not released into the system. Install a clean, dry U-tube. If additional U-tubes are unavailable, clean the current tube and allow to dry; then reinstall at the cold trap port.

If the analyzer is to be inoperative or unattended for a few days, reduce the gas flow to approximately one-quarter of its normal value and leave analyzer powered ON. The gas loss and power drain are very low and the analyzer will be immediately ready for use.



Regardless of how long the analyzer is to be idle, keep a sample tube installed at both sample ports. This ensures the integrity of the system for gas flow and prevents infusion of water and other vapors.

## GUIDELINES FOR CONNECTING GASES

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



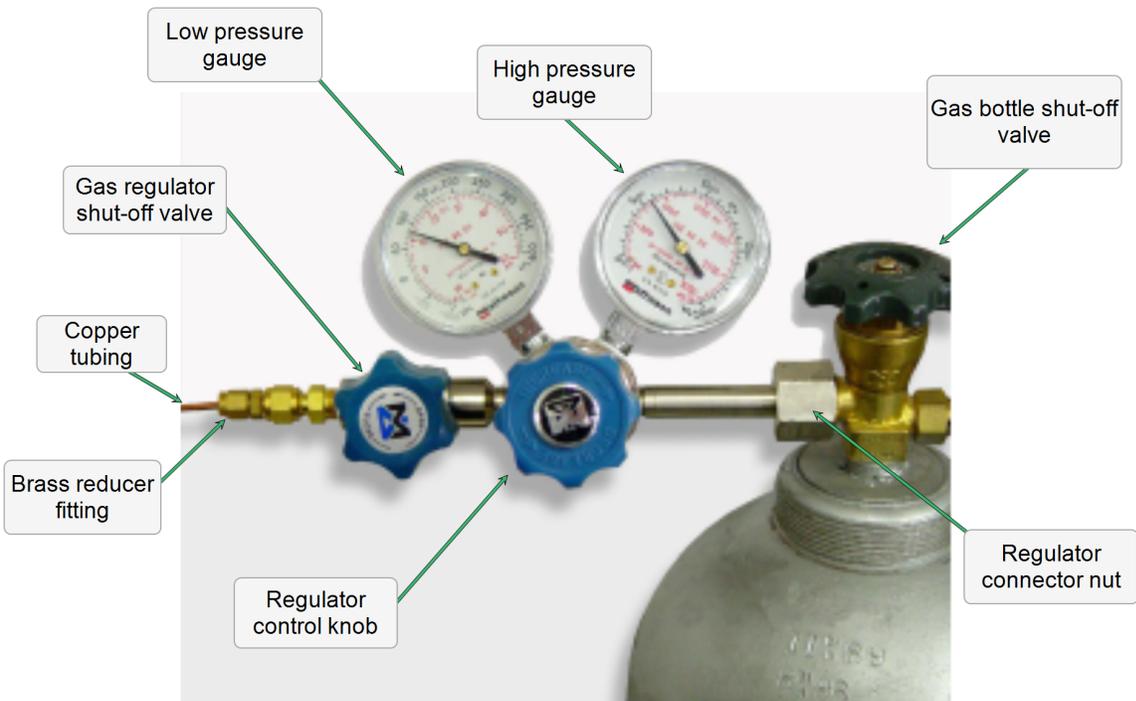
To use oxygen, the analyzer must be equipped with an oxygen-compatible vacuum pump that uses Fomblin<sup>®</sup> (or a suitable equivalent) pump oil or a dry pump. Failure to use the proper vacuum system could result in hazardous conditions, including fire and personal injury.

## CLEAN AND VERIFY THE GAS LINE

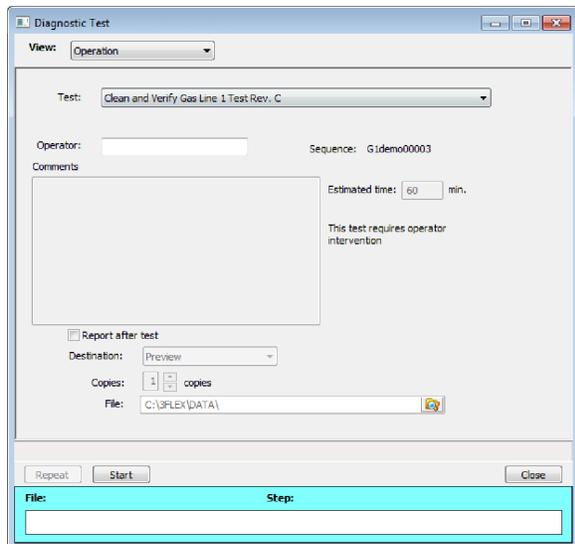
### Unit [n] > Diagnostics > Start Diagnostic Test

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.

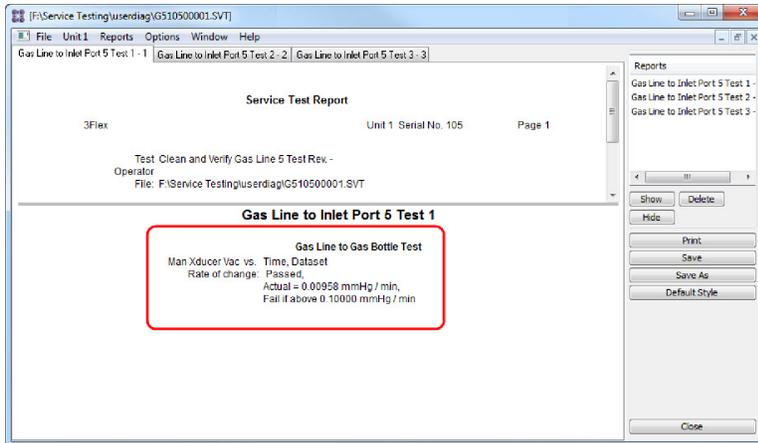
Before beginning, confirm that the state for valves and the low-pressure gauge are as follows:



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. Resize the window (if necessary) to display the *Report after test*, then select *Preview* as the destination. Click **Start**.
3. From the *View* drop-down list, select either *Operation*, *Instrument Log*, or *Instrument Schematic*.
4. 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 instrument and the regulator.
  - b. The test starts with a manual leak check (requires Snoop or equivalent, and IPA), then the line and regulator are evacuated for 20 minutes for cleaning. Next, the leak rate of the gas line is determined.
  - c. With the regulator set to 15 psig, open the 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.
5. A popup window indicates the test is complete. Click **OK**. The reports display.



6. Click each tab across the top portion 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 <i>Failed</i> status, then...
<b>Gas Line to Inlet Port [n] Test 1</b>	Gas Line to Gas Cylinder Test	This test will show a reading of <i>Failed</i> if any of the other tabs has a <i>Failed</i> reading. Correct the failed connection and rerun the test.
<b>Gas Line to Inlet Port [n] Test 2</b>	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.
<b>Gas Line to Inlet Port [n] Test 3</b>	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.

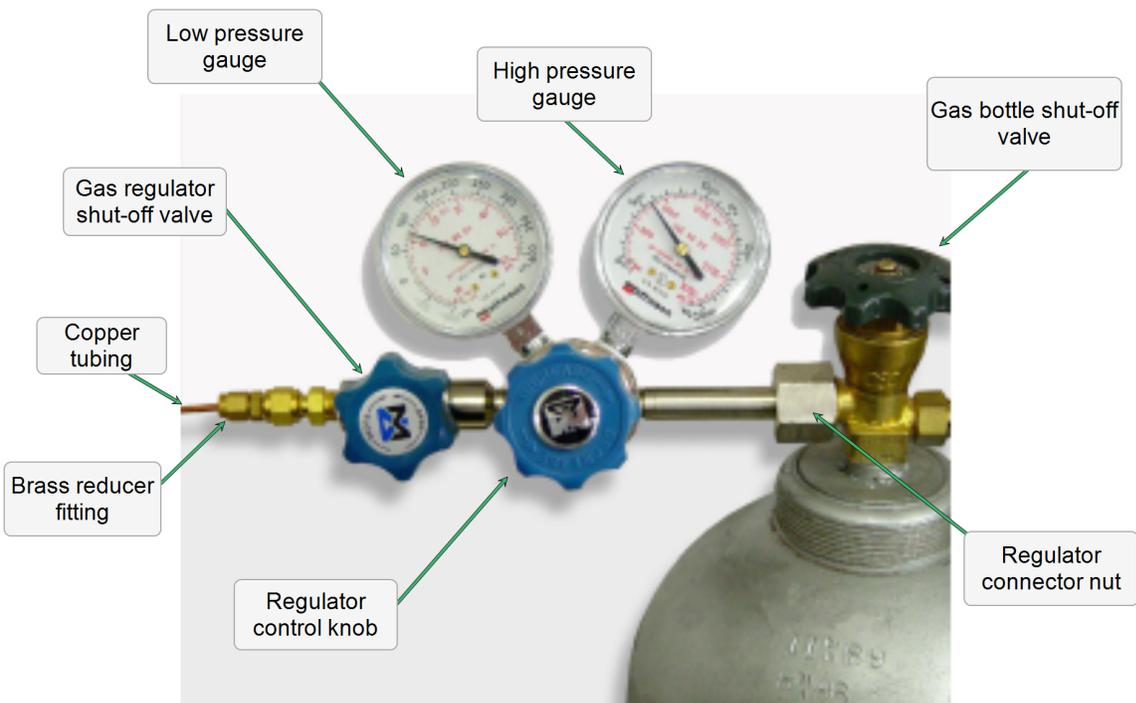
## REPLACE A GAS CYLINDER



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.



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## ***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	15 psig (103 kPag)
ASAP	15 psig (103 kPag)
AutoChem	5 psig (35 kPag)
AutoPore	45 - 50 psig (310 - 345 kPag)
Galaxy	15 psig (103 kPag)  Degas Unit — The internal regulator is pre-set at 5 psig, which provides a steady gas flow of approximately 10sccm to each gas delivery tube.
Gemini	19.5 psig (134.4 kPag)
TriStar	15 psig (103 kPag)

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

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



Over-tightening the fitting may cause a leak.

3. Check for leaks at the high-pressure side of the regulator and in the connector.
  - a. Turn the regulator control knob fully counter-clockwise.
  - b. Slowly open the gas cylinder shut-off valve, then quickly close it.
  - c. Observe the pressure on the high-pressure gauge for approximately one minute:
    - If the pressure is stable, proceed with the next step.
    - If the pressure decreases, tighten the regulator connector nut until it becomes stable. If the pressure does not remain stable, remove the regulator and clean all contacts at the regulator connection, then reinstall the regulator.
4. 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.
  - c. Turn the regulator *Shut-off* valve counter-clockwise to open. Allow gas to flow until both gauges ready 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. One wrench fitting steady and the other is used 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.

## **SPECIFY GAS PORTS**

See [Specify Gas Ports on page 2 - 15](#)

## **ENABLE MANUAL CONTROL**

### ***Unit [n] > Enable Manual Control***

Use to enable the manual control of certain system valves and elevator components. When this option is enabled, a checkmark appears to the left of ***Unit [n] > Enable Manual Control***.

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

See [Show Instrument Schematic on page 2 - 21](#)

## ***O-RING COMPATIBILITY***

O-ring selection for chemical adsorption measurements is based on temperature, time, and chemical compatibility. Chemical compatibility should be the first consideration when selecting an appropriate O-ring. The time at which the furnace, and subsequently the sample cell, is at elevated temperature can also affect the performance of the O-rings and should be a secondary consideration. Common O-ring materials include Buna-N (nitrile), Viton (fluoroelastomer), and Kalrez (perfluoroelastomer). Kalrez has historically been used extensively for chemical adsorption measurements due to compatibility with a wide range of chemicals and temperatures and should be suitable for all applications of the chemical adsorption option. Viton or Buna-N may also be suitable for analyses similar to the reference material example file. The ability to re-use Buna-N or Viton O-rings may be limited, while the re-use of Kalrez O-rings should be more broad. Frequency of use and, potentially, several other factors affect the duration of O-ring use, so rigid rules cannot be specified for these materials. Leak rate and ultimate vacuum levels may be used as indicators for O-ring performance.

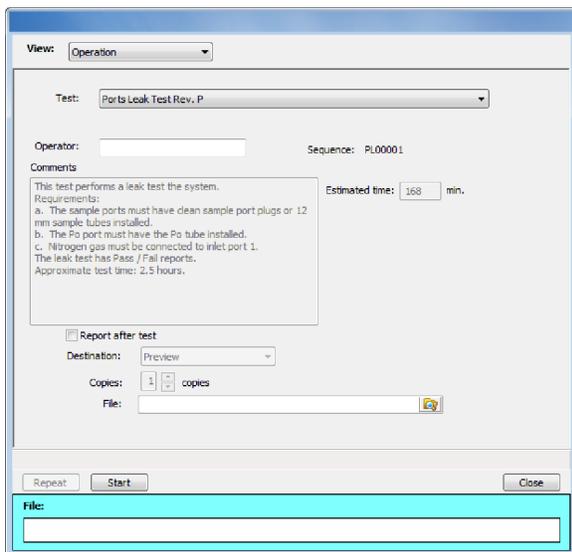
## PERFORM A LEAK TEST

### Unit [n] > Diagnostics > Start Diagnostic Test

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

The test provides:

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



View: Operation

Test: Ports Leak Test Rev. P

Operator:  Sequence: PL00001

Comments

This test performs a leak test the system.  
 Requirements:  
 a. The sample ports must have clean sample port plugs or 12 mm sample tubes installed.  
 b. The Po port must have the Po tube installed.  
 c. Nitrogen gas must be connected to inlet port 1.  
 The leak test has Pass / Fail reports.  
 Approximate test time: 2.5 hours.

Estimated time: 168 min.

Report after test

Destination: Preview

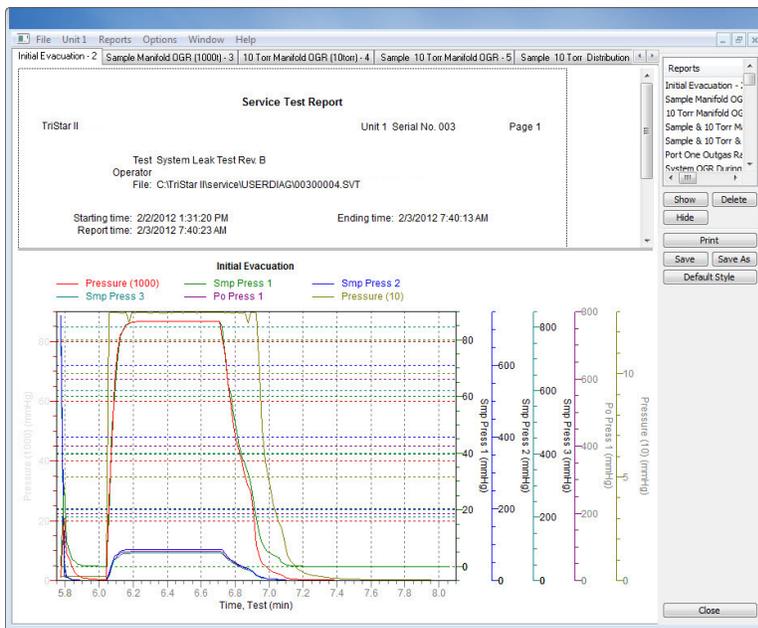
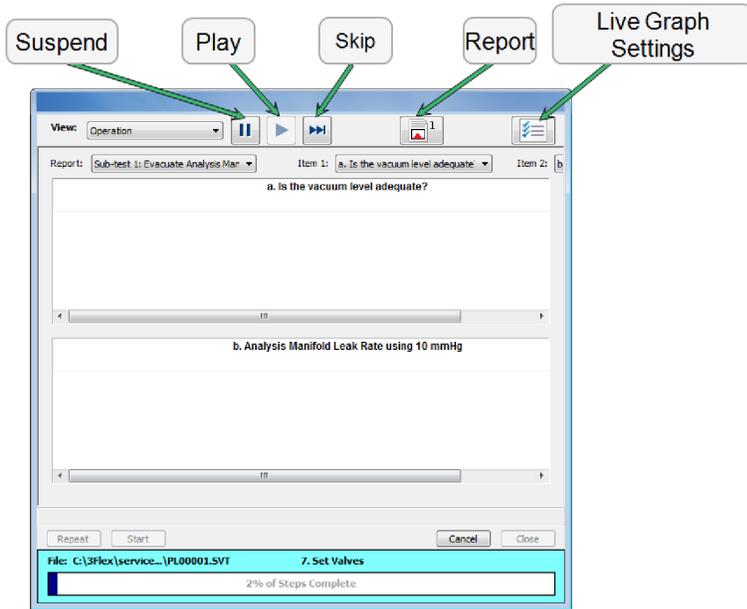
Copies: 1 copies

File:

Repeat Start Close

File:

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.



## ***PERFORM REFERENCE ANALYSIS***

See [Perform a Reference Analysis on page 6 - 25](#)

### ***PNEUMATIC PRESSURE***

The compressed air supply for the pneumatically actuated, hard seal valves and furnace cooling must be set to 100 psi  $\pm$  20 psi for proper operation. Ensure that the regulator for the compressed air supply is correctly set. If a portable air compressor is used, perform maintenance as instructed in the air compressor manual.

## PREVENTIVE MAINTENANCE

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

Maintenance Required	Frequency
Dewar	Check and clean weekly.
Power supply air filters	Clean and replace every 30 days (more often in environments with increased levels of dust).
Analyzer exterior	Clean as needed or every 6 months.
Vacuum pump diaphragm	Replace every 12 months.
Port gasket	Replace every 3 to 6 months (depending on the types of analyses that were run).
Sample tube O-ring	Replace as required or every 3 to 6 months.
Test analyzer for leaks	As required or every 12 months.
Septum	Inspect and replace every 6 months or after every 150 runs. Replace every 12 months or 300 runs.
TCD	Inspect and clean every 3 months or after every 75 runs. Replace every 12 months or 300 runs.
CryoStat	See <a href="#">Preventive Maintenance for the CryoStat on page B - 9</a> .

## CLEAN THE ANALYZER

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



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

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## ***CLEAN THE DEWAR***



When handling dewars, follow the precautions outlined in [Dewar Precautions on page 6 - 1](#).

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Ice and suspended frost particles may accumulate in the bottom of an analysis port dewar. Particles or deposits exceeding 1/4 in. (0.64 cm) in depth may jam between the bottom of the sample tubes and the bottom of the dewar, causing the dewar not to raise fully. Accumulations of fine particles impede liquid nitrogen circulation around the bottom of the sample tubes. This causes the sample temperature to be slightly higher, which 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, causing a sample tube breakage.

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



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

---

See [Enable Manual Control on page 12 - 15](#)

1. Use the schematic to lower the elevator. Remove the dewar from the elevator.
2. Pour the liquid nitrogen from the dewar into an appropriate cryogenic container.
3. Rinse the dewar with warm water to melt any remaining ice accumulation, then dry thoroughly.

## ***LUBRICATE THE ELEVATOR DRIVE ASSEMBLY***

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

Should lubrication become necessary, apply a light coat of TRIGEL™ grease (Micromeritics part number 004-16166-00) to the elevator screw, accessed from the rear of the instrument, as needed.

## ***POWER ANALYZER ON AND OFF***

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. External vacuum pump
3. Analyzer
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 dry roughing pump
5. Smart VacPrep

## REPLACE THE PSAT FITTING GASKET

A gasket is attached to the Psat fitting. Instructions for replacing the Psat fitting gasket are located in the following two links.



Each time the Psat tube or vapor source container is replaced, a new gasket is required. To avoid degassing problems, do not touch the sealing surfaces of the port fitting or gasket with bare hands.

## REPLACE THE PSAT TUBE FERRULES



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



Over an extended period of time, pivoting the Psat tube may cause wear on the nylon and teflon ferrules housed in the Psat tube nuts. If the recommended weekly scheduled *P<sub>o</sub> Port Leak Test* detects a leak by reporting *Failed* on the *Evacuated* or *Pressured* report, the first time a leak is detected, tighten the Psat nuts 1/2 turn and rerun the test. If the leak is still present, replace the nylon and teflon ferrules.

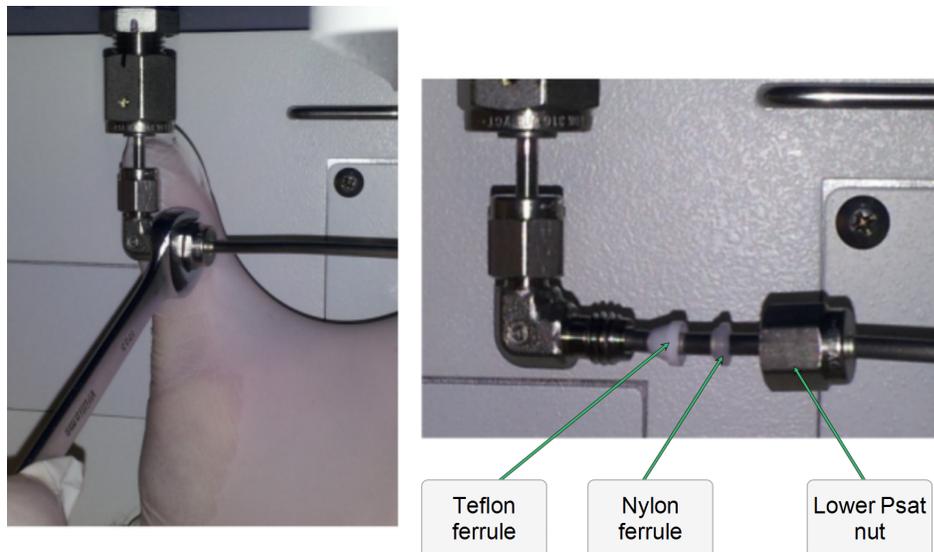


It is recommended that the VCR connector not be removed from the port fitting for this process.

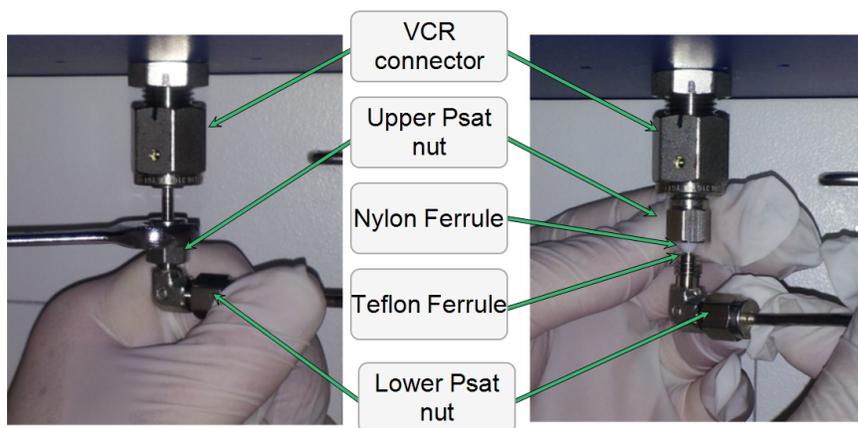


Two ferrule sets are located in the upper and lower Psat nuts. Both sets should be replaced. Additional ferrule sets were included in the analyzer's accessory kit.

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



3. Remove the Psat tube from the Psat elbow.
4. Remove the nut from the Psat tube and remove the set of teflon and nylon ferrules from inside the nut. Orient the ferrules as shown with the cone pointed out of the nut.
5. Insert the Psat nut onto the Psat tube, followed by a nylon ferrule, then a teflon ferrule.
6. Insert the Psat tube into the Psat elbow.
7. Hand tighten the Psat nut by turning the nut clockwise. Then use an appropriate size wrench to tighten the nut an additional 3/4 turn while holding the elbow so it does not move.
8. To remove the upper Psat nut, use a 7/16 in. (11 mm) wrench to loosen the Psat nut by turning the nut counter-clockwise.



9. Remove the Psat tube from the VCR connector.
10. Remove the nut from the Psat tube and remove the set of teflon and nylon ferrules from inside the nut.
11. Reinsert the Psat nut onto the VCR connector, followed by a nylon ferrule, then a teflon ferrule. Orient the ferrules as shown with the cone pointed out of the nut.

12. Insert the Psat elbow into the upper Psat nut.
13. Hand tighten the upper Psat nut by turning the nut clockwise. Then use a 7/16 in. (11 mm) wrench to tighten the nut an additional 3/4 turn while holding the elbow so it does not move.

### REPLACE THE SAMPLE PORT FRIT



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



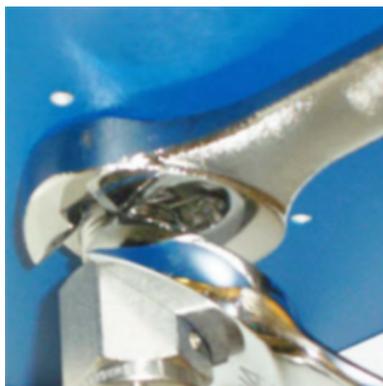
The chemical adsorption port frit is changed in the same manner as the sample port frit.

A frit is located in the connecting nut attached to each analysis port. If the frit becomes contaminated, the contaminant may adsorb or desorb during analysis, affecting the results. A contaminated frit on the analysis port may be indicated as a leak or a free space reading much lower than normal.



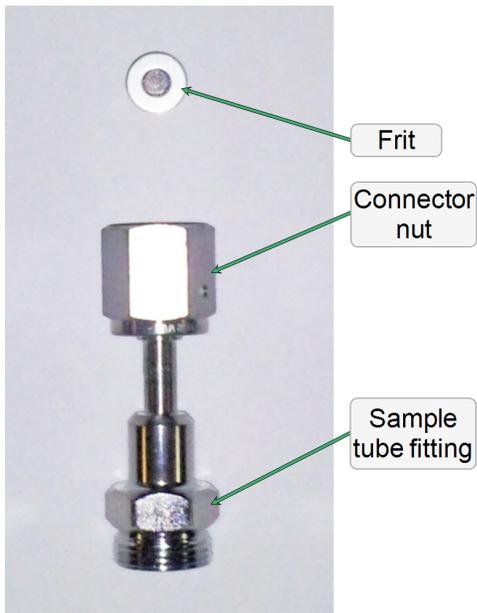
Use a 20 µm frit. The analyzer will not operate properly if an incorrect size is used.

1. Go to **Unit [n] > Enable Manual Control**. Ensure a checkmark displays to the left of the menu item. If the instrument schematic does not display, go to **Unit [n] > Show Instrument Schematic**.
2. Right click on the valve of the appropriate port. If the valve is open, click **Close** to close the valve.
3. Use a wrench to remove the connecting nut from the sample port while using a second wrench to hold the port fitting stationary. Remove and discard the used frit.



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

4. Place a new frit into the connecting nut.



5. Attach the connecting nut to the sample port fitting and finger tighten. Use a wrench to tighten the nut 1/8 to 1/4 turn past finger tight while using a second wrench to hold the port fitting stationary.

## REPLACE THE SAMPLE TUBE O-RING



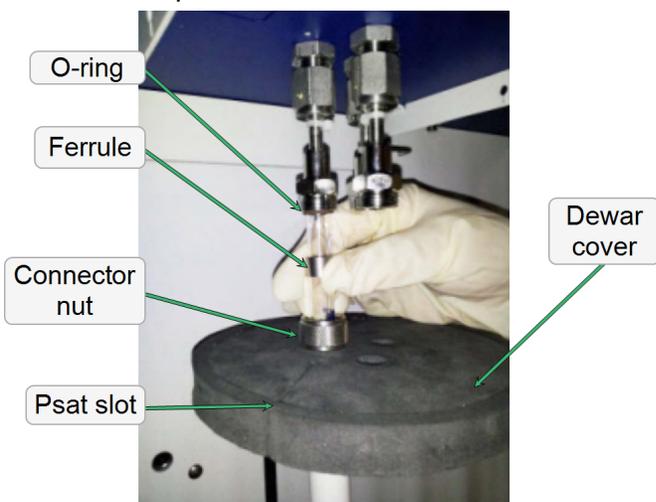
The equipment shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

It is important to maintain a vacuum-tight seal near the top of the sample tube stem. If an O-ring becomes worn or cracked, it does not provide a good seal and will need to be replaced.



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

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



4. Remove the O-ring from the top of the sample tube and replace it with a new one.



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

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

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

## ***PURGE THE SYSTEM***

### ***PURGE AIR OR GAS***

See [Guidelines for Connecting Gases on page 12 - 8](#) when connecting a new gas cylinder to one of the 12 primary gas inlets on the back of the instrument for use as a carrier gas. After connecting a new gas cylinder to one of the four loop gas inlets on the side of the instrument, open the gas inlet valve and allow gas to flow for a period of time to purge the gas line before using that gas in an analysis.

### ***Changing the Gas Flow During an Analysis***

When gases are changed during an analysis, allow the new gas to flow for a period of time before creating conditions that cause the experiment to begin (such as elevating the temperature). To avoid undesirable combinations of gases within the analyzer, purge one gas (by flowing an inert gas for a period of time) before starting to flow another gas.

To flow an inert gas between incompatible gases, insert a *Gas Flow* step (in which an inert gas is flowed for a period of time) and a *Wait* step (to wait for the inert gas to purge the analyzer) between other steps that involve incompatible gases.

When gas flows are changed while the analyzer is recording data, the gas flow is briefly disturbed. This may result in a brief period of noise or other visible disturbances on the peak data. Either disregard the disturbance or insert a *Wait for stable baseline* step immediately after changing gas flows.

## ***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.

## ***REPLACE A SEPTUM***



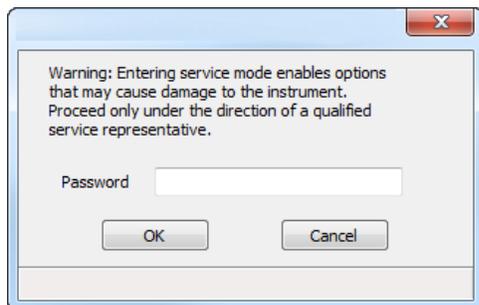
The septum usually requires replacing after approximately 100 injections when using the 1 ml syringe.

1. Turn the knurled nut counterclockwise and remove it from the injection port.
2. Tap the nut into the palm of your hand to remove the septum and discard the used septum.
3. If the washer came out when the septum was removed, place the washer back into the knurled nut first. Place a new septum into the knurled nut.
4. Place the knurled nut back onto the injection port; turn the nut clockwise to finger-tighten.

## ***SERVICE TEST MODE***

### ***Options > Service Test Mode***

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



## ***TCD ASSEMBLY***

The filaments in the thermal conductivity detector assembly are heated to high temperatures and may be exposed to corrosive gases, therefore they may eventually need to be replaced. Symptoms that the TCD filaments may need replacing:

- A stable TCD baseline cannot be maintained.
- Data collection results in very high or very low peaks that are inconsistent with previous experience.
- During a data collection, no TCD signal is displayed on the main display, or the software indicates that no TCD signal is present.

## ***CLEAN THE TCD ASSEMBLY***

In some instances, contamination of the thermal conductivity detector filaments will produce the symptoms described above. To clean the filaments, set the filament zone to 150 °C and flow an inert gas through the analyzer for several hours. Contact your Micromeritics service representative if necessary.

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## A ATOMIC WEIGHTS AND CROSS SECTIONAL AREAS

### Atomic Weights and Cross-sectional Areas for Selected Metals

<b>Metal</b>	<b>Symbol</b>	<b>Atomic Weight (g/mole)</b>	<b>Cross-sectional Area (sq nm)</b>	<b>Density (g/ml)</b>
chromium	Cr	51.996	0.0635	7.19
cobalt	Co	58.933	0.0662	8.9
copper	Cu	63.54	0.0680	8.96
gold	Au	196.967	0.08696	18.9
hafnium	Hf	178.490	0.0862	13.3
iridium	Ir	192.220	0.0769	22.4
iron	Fe	55.847	0.0613	7.89
manganese	Mn	54.938	0.0714	7.43
molybdenum	Mo	95.940	0.0730	10.22
nickel	Ni	58.710	0.0649	8.9
niobium	Nb	92.906	0.0806	8.57
osmium	Os	190.220	0.0629	22.6
palladium	Pd	106.400	0.0787	12.02
platinum	Pt	195.090	0.0800	21.45
rhenium	Re	186.2	0.0649	21.02
rhodium	Rh	102.905	0.0752	12.1
ruthenium	Ru	101.070	0.0613	12.4
silver	Ag	107.868	0.0869	10.5
tantalum	Ta	180.947	0.0800	16.6
thorium	Th	232.038	0.1350	11.7
tin	Sn	118.710	0.1082	4.54
tungsten	W	183.850	0.0741	19.3
vanadium	V	50.942	0.0680	6.11
zirconium	Zr	91.220	0.0877	6.51

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## ***B CRYOSTAT***

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The cryostat elevator is manually operated, however the analyzer elevator must first be disabled to prevent damage to the analyzer.

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### ***Unit [n] > Enable CryoStat***

When selected:

- ***Unit [n] > Disable Elevator*** is automatically selected
- applicable cryostat fields within the application will be enabled

### ***Unit [n] > Disable Elevator***

When selected:

- the analyzer elevator will not move until this option is deselected

## CRYOSTAT OPERATING INSTRUCTIONS

The cryostat provides automatic analysis temperature control over a wide range of sub-ambient temperatures.



- Read and fully understand this entire section prior to carrying out operations on the system.
- All system components must be powered ON in the sequence provided in this section.
- Pay attention to all local safety systems and requirements and consider the specifics of the experiment when operating the system.

1. Prior to starting any analysis on the system, ensure the following:
  - Go to **Unit [n] > Enable CryoStat** and ensure a checkmark displays to the left of the menu option. A checkmark indicates the item has been selected and disables the elevator during an analysis when using the cryostat.
  - The cryostat is correctly mounted and held firmly in place by the metal retaining bands.
  - Release the brake on the cryostat cradle and move the system upwards to check that the sample well and the analyzer sample port is correctly aligned.
  - Check that the vacuum connection, helium connection, and wired connections to the compressor and temperature controller are in place and correctly fitted.
2. Prepare the samples:
  - Samples can be prepared in-situ using the analyzer heating mantle (if the heating mantle fits with the cryostat installed). See [Degas in Situ on page 5 - 4](#). This will depend on the type of heating mantle used.
  - The cryostat can be prepared to run analyses in the DOWN position while the samples are being prepared. Leave the insulating cover for the cryostat sample well in place while doing this.
3. Turn on the helium supply and adjust the rotameter to scale position 55. This corresponds to a flow rate of approximately 5 ml/min. See [Rotameter Scale Readings on page B - 5](#).
4. Allow the helium to purge the sample well for fifteen minutes. This purge procedure removes water vapor from the sample well and prevents water vapor from freezing when the system is in use at sub-ambient/cryogenic temperatures.
5. Power ON the compressor — first switch the circuit breaker to the UP position. The compressor will now start to pump.
6. Press the **ALL OFF** button to power ON the temperature controller. Set the required temperature on the temperature controller or in the analysis application. If using the temperature controller, refer to the temperature controller manual supplied by the manufacturer.

7. Create a sample file (see [Sample Files on page 3 - 1](#)). There are settings specific to physisorption analyses using a cryostat in [Analysis Conditions for Physical Adsorption and Chemical Adsorption on page 4 - 5](#).
8. With the sample tubes in place, preparation completed, and the tubes cooled to ambient temperature; slide the insulating cover up the sample tubes so that the cover fits snug against the mouth of the raised cryostat.
9. Remove the insulating cover from the top of the cryostat sample well. When prompted by the analyzer application, raise the cryostat.
10. Allow the samples to thermally equilibrate before starting the analysis.
11. When prompted by the analyzer application, lower the cryostat and place the insulating cover over the sample well of the cryostat.
12. If the sample tubes are to be removed, use **Unit [n] > Backfill Ports** or use the manual control option and pressurize the sample tubes to 760 mmHg at ambient temperature.
13. Weigh the samples after analysis and enter the sample weight into the sample file.
14. The cryostat can now be used to analyze more samples at the same temperature or change the set point temperature.
15. Power OFF the compressor, vacuum pump, helium supply, and temperature controller. The cryostat should remain powered ON unless it will be unused for a long period of time. If it is necessary to power OFF the cryostat, set it to ambient temperature and allow it to come up to room temperature.

## ***INSTALL SAMPLE TUBES FOR THE CRYOSTAT***

Sample tubes for the cryostat are installed in the same manner as the sample tubes for the analyzer.

## ***REMOVE THE CRYOSTAT FROM THE ANALYZER***

To run a normal analysis, the cryostat must be removed.

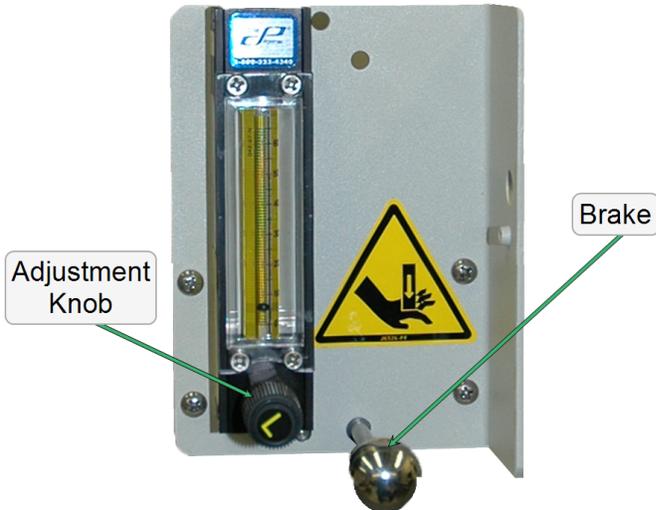
1. Move the cryostat to the DOWN position.
2. Place the brake in the bottom position.
3. Remove the 1/8 in. nylon helium tube.
4. Close vacuum valve.
5. Remove the 1/4 in. flexible stainless steel evacuation hose at the Swagelok valve.
6. Remove the large hose clamps and lift the entire cryostat off the cradle.
7. Lay the assembly on an adjacent bench so that other hoses do not need to be disconnected.
8. Close the helium purge supply, close the vacuum valve, power OFF the vacuum pump and compressor, turn OFF the temperature controller.



It is not necessary to remove the carriage assembly from the analyzer, however, the special safety shield (supplied with the cryostat kit) should be used. This shield has been modified to reach over the cryostat panel and hook onto the analyzer.

## ROTAMETER SCALE READINGS

The rotameter scale corresponds approximately to flow rates:



### Rotameter Scale to Flow Rate

Scale Reading	Flow Rate (ml/min)
65	5.8
55	5
45	4.3
35	3.3
25	2.5
15	1.8
5	1.3

## ***TROUBLESHOOTING THE CRYOSTAT***

### **Frost or ice accumulates around the outside of the cryostat in the area of the sample well**

*Cause:* The internal vacuum is degrading and must be re-evacuated.

*Action:* Effective re-evacuation can be done only when the cryostat is warm, such as after an analysis. It is recommended that the vacuum pump always remain powered ON.

### **Elevator tries to rise**

*Cause:* Analysis program is not set to enable the cryostat.

*Action:* In the analysis program, go to **Unit [n] > Enable CryoStat** and ensure that this option is selected. Failure to do so will cause the P<sub>0</sub> port to be evacuated and the elevator will try to rise.

### **Valve motor in cryostat cold head does not start when the compressor starts**

*Cause A:* Cold head cable is not connected.

*Action A:* Stop the compressor. Connect the cable.

*Cause B:* Open circuit in the cold head cable.

*Action B:* Disconnect the cold head cable. Check each conductor for continuity. Replace the cable if necessary.

*Cause C:* Defective valve motor.

*Action C:* Contact your Micromeritics service representative.

*Cause D:* Blown fuse in the compressor's electrical box.

*Action D:* See the manual supplied by the compressor manufacturer.

### **Valve motor in cryostat cold head hums but does not start**

*Cause A:* Valve disk has stalled.

*Action A:* Check the operating pressures on the compressor.  
Contact your Micromeritics service representative.

*Cause B:* Defective valve motor.

*Action B:* Contact your Micromeritics service representative.

*Cause C:* Open circuit in the cold head cable.

*Action C:* Disconnect the cold head cable. Check each conductor for continuity. Replace the cable if necessary.

### **Valve motor on the cryostat cold head runs but there is no cooldown**

*Cause A:* No insulating vacuum.

*Action A:* Check the vacuum system for operation and leaks.

*Cause B:* Gas line couplings are not fully engaged.

*Action B:* Ensure all couplings are fully engaged and torqued.

*Cause D:* Gas lines are connected incorrectly.

*Action D:* Reconnect the gas lines. See the manual supplied by the compressor manufacturer.

*Cause E:* Compressor output is inadequate.

*Action E:* Troubleshoot the compressor. See the manual supplied by the compressor manufacturer.

### **Shroud is sweating or abnormally cold**

*Cause:* Loss of insulating vacuum.

*Action:* Check the vacuum system for operation and leaks.

### **Abnormally noisy operation after a sustained period of five to fifteen minutes**

*Cause A:* Incorrect compressor pressures.

*Action A:* Troubleshoot the compressor. See the manual supplied by the compressor manufacturer.

*Cause B:* Contaminants in the gas.

*Action B:* Perform *Gas Cleanup and Recharging* procedure on the cold head, compressor, and the gas lines. See the manual supplied by the compressor manufacturer. Contact your Micromeritics service representative.

### **Intermittent operation**

*Cause:* Compressor is cycling on and off.

*Action:* Troubleshoot the compressor. See the manual supplied by the compressor manufacturer.

### **Temperature is cycling**

*Cause:* Contaminated gas is causing a cold head freezing-thawing cycle.

*Action:* Perform *Gas Cleanup and Recharging* procedure on the cold head, compressor, and the gas lines. See the manual supplied by the compressor manufacturer. Contact your Micromeritics service representative.

### **Sudden loss of refrigeration capacity**

*Cause A:* Loss of insulating vacuum.

*Action A:* Check the vacuum system for operation and leaks.

*Cause B:* Compressor malfunction.

*Action B:* Troubleshoot the compressor. See the manual supplied by the compressor manufacturer.

### **Slow loss of refrigeration capacity**

*Cause A:* Small insulating vacuum leak.

*Action A:* Leak check and repair the vacuum system.

*Cause B:* Worn seals in the cold head.

*Action B:* Contact your Micromeritics service representative.

*Cause C:* Cold head is leaking.

*Action C:* Contact your Micromeritics service representative.

**PREVENTIVE MAINTENANCE FOR THE CRYOSTAT**

The preventive maintenance procedures can be located in the documents supplied by the cryostat manufacturer.

**If frost accumulates outside the sample well of the cryostat.**

*Cause:* Internal vacuum is degrading.

*Action:* Ensure the high vacuum system is running and the vacuum isolation valve on the carriage is open.

Maintenance Required	Frequency
Cold head	Every 13,000 hours
Compressor (absorber)	Every 30,000 hours

## **CRYOSTAT CALIBRATION**

The cryostat temperature reading does not necessarily reflect the temperature at the sample. The discrepancy is large enough to cause significant differences between data taken with a cryogenic bath and data taken with the cryostat set to the temperature of the bath.

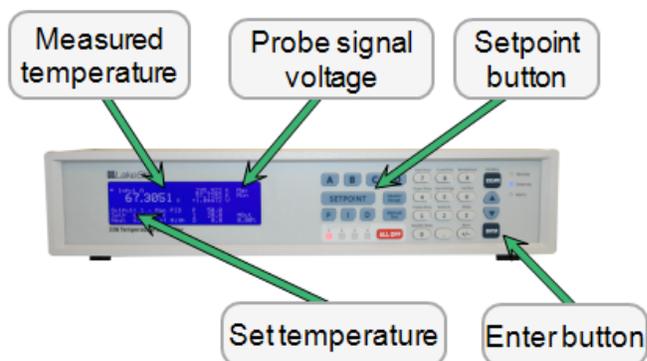
One solution is to modify the voltage-to-temperature curve stored in the temperature controller so that the reading accurately reflects the temperature of the sample. This option requires additional interaction with the temperature controller to download, upload, and select the voltage-to-temperature curves. However, this is straightforward with the Lakeshore controller and their Curve Handler software.

### **REQUIRED ITEMS**

- A 3Flex analyzer equipped with an installed Micromeritics cryostat unit.
- A selection of adsorptives to cover the temperature range to be calibrated — such as N<sub>2</sub>, Ar, methane, ethane, and propane. Choose adsorptives whose saturation pressure is less than 800 mmHg at the measurement temperatures.
- The Lakeshore Curve Handler software (free download): <http://www.lakeshore.com/products/Pages/curvehandler.aspx>.
- An empty sample tube for the Micromeritics gas adsorption analyzer being used.
- A spreadsheet application — such as Microsoft Excel. Knowledge of how to load a .TXT file containing tabular data into a spreadsheet application is beneficial.

### **CALIBRATION PROCEDURE**

1. In the analysis application, go to the *Unit* menu. Ensure there is a checkmark to the left of *Disable Elevator* and no checkmark to the left of *Enable CryoStat*. The cryostat will be adjusted manually.
2. Install a blank sample tube into the gas adsorption analyzer. Note that only one tube is required for the 3Flex.
3. Raise the cryostat and lock it in place following the general cryostat procedure.
4. Set the cryostat to the required temperature. On the LakeShore temperature controller, press **SETPOINT**, enter the temperature (in kelvins), then press **ENTER**.



5. Wait until the cryostat reaches the entered temperature. The time varies depending on how big the temperature jump is. For example, going from 200 K to 215 K may take 15 to 20 minutes; going from 298 K to 77 K might take 90 minutes. Once the cryostat has reached the operating temperature, record the set temperature, the measured temperature, and the cryostat signal probe voltage.
6. Create a sample file to measure saturation pressure.

The analysis is to run in the absolute pressure dosing mode with an upper pressure limit of 950 mmHg. Increment volume dosing is to be employed using a dose volume of 20 cm<sup>3</sup>/g STP with a 5 second equilibration time. In Analysis Conditions:

- a. Select the *Absolute pressure dosing* option.
- b. Select the adsorptive to be used.
- c. Enter the pressure settings shown in the *Pressure Settings for 3Flex* table below.

#### Pressure Settings for 3Flex

	Starting Pressure (mmHg)	Pressure Increment (mmHg)	Dose amount (cm <sup>3</sup> /g STP)	Equilibration Interval(s)	Ending Pressure (mmHg)
Line 1	0.000000			5	100.000000
Line 2	100.000000		20.0000	5	900.000000

7. Once the analyzer is set up and the temperature for the first data point has reached equilibrium, start the analysis defined by the sample file. The analyzer will dose the sample tube with 20 cm<sup>3</sup>/g STP of adsorptive. Once the tube pressure reaches the adsorptive saturation pressure, the generated isotherm will climb straight up. Take several points at this saturation pressure, then pause the analysis and set the cryostat to the next temperature.

Once the cryostat has reached the set temperature and has equilibrated a few minutes, record the cryostat temperature and the probe signal, then resume the analysis. Perform this cycle for the number of temperature points required for the adsorptive being used. Typical isotherms are shown below. Note that the temperature labels are for clarity and are not a part of the normal report.

Additional adsorptives will allow the calibration to be extended. A set of local fits may be needed to cover a wide temperature change.



Depending on the selected adsorptive, a warning message may be given indicating that 950 mmHg is not the saturation pressure for the temperature being used. This is a normal message and will not interfere with the experiment. Click **OK** and continue with the analysis.



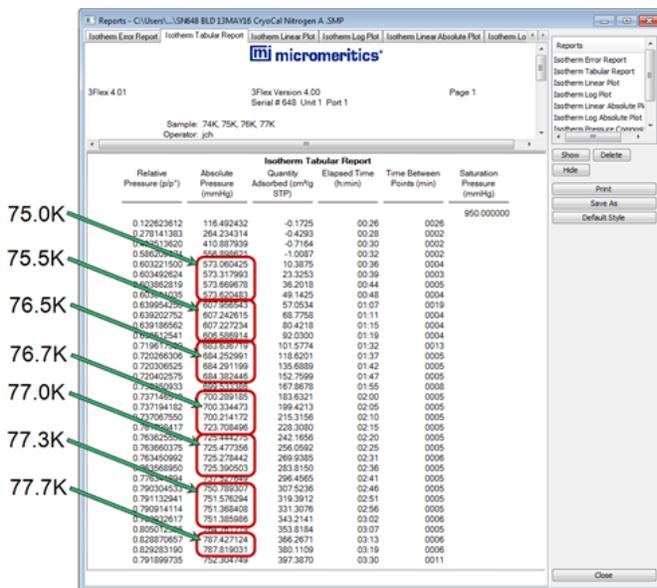
These analyses will not end and will have to be terminated manually by stopping the analysis window. To terminate the analysis, click **Cancel**, then wait a few seconds and click **Cancel** again (do not double-click the **Cancel** button).

The analysis will end abruptly with adsorptive in the sample tube. The sample tube must be manually evacuated before the cryostat is lowered.

Manually close all valves. go to **Unit > Evacuate Ports** to evacuate the sample tube(s) used for the measurement.

The cryostat can be lowered once the sample tube has been completely evacuated.

This saturation pressure data can either be read from the graph or an isotherm report can be made and the saturation pressure read from the report:



8. Place the data in table format using a spreadsheet application such as Microsoft Excel.

## Temperature and Saturation Pressure Values

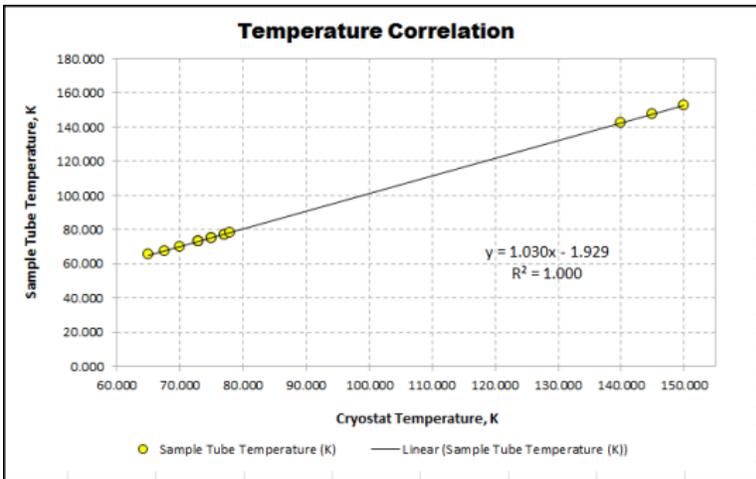
	Set Temperature (K)	CryoStat Temperature (K)	Signal Probe Voltage	Measured Saturation Pressure (mmHg)	Sample Tube Temperature (K)
Nitrogen	65	64.999	1.04878	137.833	65.317
	67.5	67.498	1.04474	206.334	67.778
	70	69.999	1.04066	301.792	70.294
	73	72.996	1.03572	454.217	73.239
	73	72.996	1.03572	456.148	73.271
	73	72.998	1.03571	452.139	73.239
	75	75.002	1.03240	584.063	75.189
	75	75.002	1.03245	587.793	75.240
	77	76.993	1.02896	743.094	77.165
	77	76.995	1.02897	742.343	77.156
	78	78.002	1.02737	832.533	78.165
Ethylene	140	139.999	0.91437	111.175	142.470
	145	144.999	0.90456	168.125	147.470
	150	150.000	0.89477	245.617	152.350

<b>Set Temp</b>	Cryostat temperature setting, in kelvins.
<b>CryoStat Temperature</b>	Temperature reported by the temperature controller.
<b>Signal Probe Voltage</b>	Signal probe voltage reading.
<b>Measured Saturation Pressure</b>	Equilibrium pressure measured by the instrument when the isotherm is vertical.
<b>Sample Tube Temperature</b>	Sample tube temperature calculated from the measure saturation pressure using NIST's REFPROP program (bundled with the Micromeritics applications).

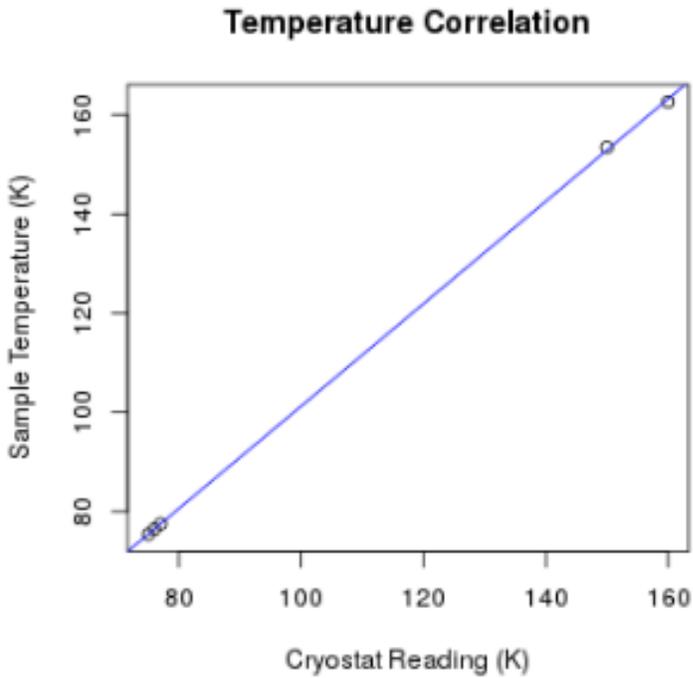
	Temperature (K)	Pressure (mmHg)	Liquid Density (kg/m <sup>3</sup> )	Vapor Density (kg/m <sup>3</sup> )	Liquid Enthalpy (kJ/kg)	Vapor Enthalpy (kJ/kg)	Liquid Entropy (kJ/kg-K)	Vapor Entropy (kJ/kg-K)
1	75.037	573.00	816.51	3.5555	-126.76	75.345	2.7724	5.4658
2	75.497	607.00	814.45	3.7492	-125.82	75.713	2.7848	5.4542
3	76.472	684.00	810.08	4.1852	-123.83	76.479	2.8108	5.4302
4	76.664	700.00	809.21	4.2753	-123.43	76.628	2.8159	5.4255
5	76.957	725.00	807.89	4.4159	-122.83	76.854	2.8237	5.4185
6	77.254	751.00	806.54	4.5617	-122.23	77.081	2.8315	5.4114
7	77.663	788.00	804.68	4.7687	-121.39	77.391	2.8423	5.4017
8								

Screenshot from REFPROP software

The spreadsheet data can be graphed in the REFPROP spreadsheet program. Plot the REPROP to determine temperature versus the Cryostat reading. Fit the data points:

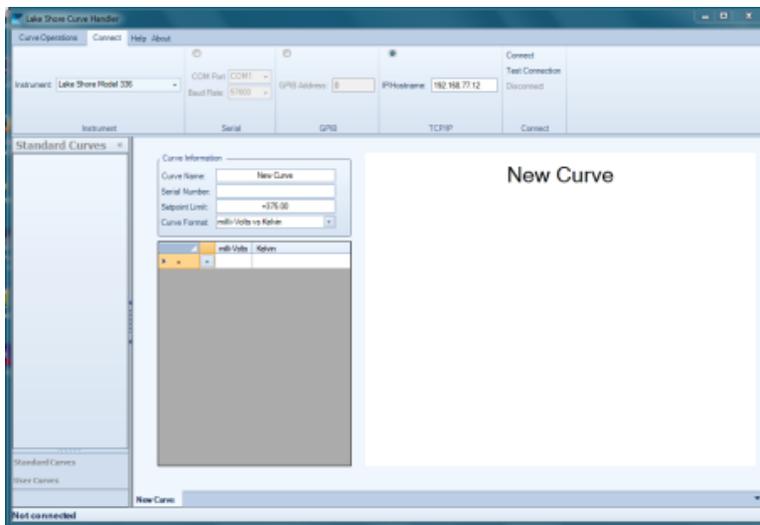


Additional adsorptives will allow the calibration to be extended. The following graph contains data for nitrogen combined with data from ethane:

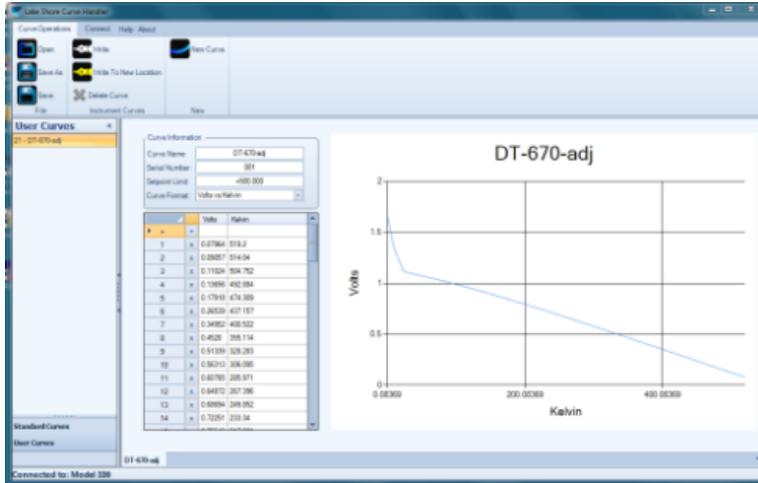


- The REPROP determined sample tube temperatures, combined with the signal probe voltages read from the LakeShore controller are then used to construct a cryostat calibration curve.

The LakeShore Curve Handler program (shown below) is used for this operation. (See the LakeShore Curve Handler manual for communications information.) Connect the cryostat to the computer via an Ethernet connection.



- Click **Connect** to establish communication with the LakeShore Temperature Controller. Once communication is established, all the calibration curves in the LakeShore Temperature Controller will be listed.



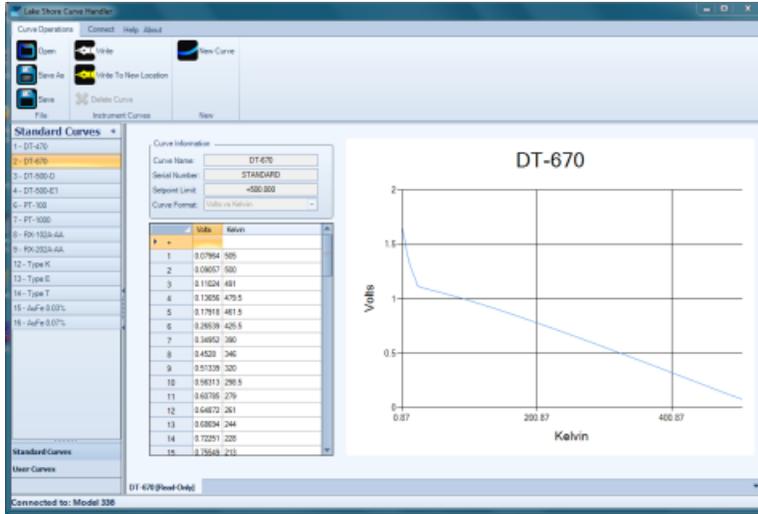
- The ColdEdge Cryostat/LakeShore Model 336 Temperature Controller uses the DT-670 silicon diode as the temperature sensor according to ColdEdge Micromeritics Interface Owner's Manual. Select the curve for the DT-670 temperature probe. Click **Save As** and save the DT-670 curve with a different name (i.e., *DT-670-xx*). Note that all calibration curves are stored as ASCII files in the Documents library with the file extension *.curve*. There will be a *DT-670.curve* file and a *DT-670-xx.curve* file.

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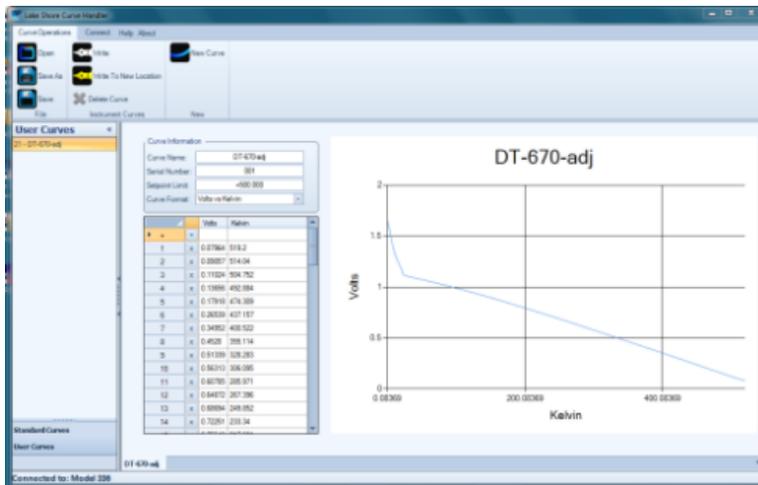
Sensor Model: DT-670
Serial Number: STANDARD
Data Format: 2 (volts/kelvin)
SetPoint Limit: +500.000

Measurement (volts)    Temp (K)
7.964000E-02          5.050000E+02
9.057000E-02          5.000000E+02
1.102400E-01          4.910000E+02
1.365600E-01          4.795000E+02
1.791800E-01          4.615000E+02
2.653900E-01          4.255000E+02
3.495200E-01          3.900000E+02
4.528000E-01          3.460000E+02
5.133900E-01          3.200000E+02
5.631300E-01          2.985000E+02
6.078500E-01          2.790000E+02
6.487200E-01          2.610000E+02
6.869400E-01          2.440000E+02
7.225100E-01          2.280000E+02
7.554900E-01          2.130000E+02
7.869900E-01          1.985000E+02
8.170200E-01          1.845000E+02
8.445400E-01          1.715000E+02
8.695800E-01          1.595000E+02
8.932300E-01          1.480000E+02
9.144700E-01          1.375000E+02
9.343600E-01          1.275000E+02
9.529000E-01          1.180000E+02
9.701300E-01          1.090000E+02
9.860700E-01          1.005000E+02
9.989200E-01          9.350000E+01
1.010640E+00          8.700000E+01
1.021250E+00          8.100000E+01
1.031670E+00          7.500000E+01
1.041890E+00          6.900000E+01
1.051920E+00          6.300000E+01
1.062770E+00          5.640000E+01
1.074720E+00          4.900000E+01
1.091100E+00          3.870000E+01
1.096020E+00          3.570000E+01
1.100140E+00          3.330000E+01
1.103930E+00          3.120000E+01
    
```

12. The LakeShore Curve Handler instruction manual provides several methods of entering user created calibration. The following process uses a file copy of a modified version of the DT-670 curve previously saved.
13. Produce a modified calibration curve by applying the fit from step 8 to the temperatures in DT-760 curve.
14. Start the LakeShore Curve Handler program and establish communication with the LakeShore Temperature Controller.



15. Click **Open** and select the modified *DT-670-xx.curve*. Place this curve in the first empty bin. User curve bins start at Number 21.
16. The new curve should now be able to be loaded into the LakeShore Curve Handler program. Click the *User's Curve* tab in the lower left side of the screen.



17. Note in this example the modified curve *DT-670-xx.curve* is in bin 22 (left column) and the curve is displayed on the right. The values should be reviewed and any changes made. Click **Save** if changes have been made. Click **Write** to load the new curve into the LakeShore Temperature Controller. Exit the LakeShore program.
18. To select the new curve, on the temperature controller, press the *Input* command (key 7), select the *Input* channel (usually *A*), use the arrows to navigate to the curve menu, press **Enter**, then select the curve. Use curve 22 in this example. Press **Enter** once more, then press **Escape**. The new, modified curve is now in place. More details can be found on page 52 of the LakeShore Model 336 Temperature Controller User's Manual.

The table below shows the effect of the new curve:

**Temperature and Saturation Pressure Values**

<b>Set Temp (K)</b>	<b>Cryostat Temperature (K)</b>	<b>Measured Saturation Pressure (mmHg)</b>	<b>Uncalibrated Sample Tube Temperature (K)</b>	<b>Calibrated Sample Tube Temperature (K)</b>	<b>% Error (Calculated vs Uncalibrated)</b>
<b>73</b>	72.997	473.450	73.553	73.250	-0.414
<b>75</b>	74.996	615.020	75.603	75.215	-0.517
<b>77.3</b>	77.002	786.618	77.648	77.029	-0.804

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## C DFT MODELS

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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

$\epsilon$  = a characteristic energy of the adsorptive,  
 $\sigma$  = the diameter of the adsorptive molecule, and  
 $s$  = the separation distance.

## ***MOLECULAR SIMULATION METHODS***

Two simulation techniques are commonly used to determine the distribution of gas molecules in a system in equilibrium: the molecular dynamics method and the Monte Carlo method. Both of these are used as reference methods because their results are considered exact.

### ***MOLECULAR DYNAMICS METHOD***

In the molecular dynamics method, the position and velocity of individual gas particles are calculated over time at very short intervals. This method takes into account both the forces acting between the gas particles themselves and those acting between the gas particles and the atoms of the simulated surface. As the simulated particles collide with each other and with the surface, the average concentration of particles in the space near the surface is calculated; this calculation yields the amount of gas adsorbed.

This method can be thought of as a way to determine the chronological record of the movement of each particle in the system using time steps of 10-14 seconds. Although the mathematics are simple, the number of calculations required for a system of even a few hundred particles is astronomical and challenges even the fastest computers.

## **MONTE CARLO METHOD**

In the Monte Carlo method, determination of the system equilibrium distribution begins with an assumption (which may be only approximate) about the initial configuration of particles in the system. The system is “equilibrated” through a process of randomly selecting one particle and conditionally moving it a random distance in a random direction.

If the move results in a configuration of *lower total energy*, then the move is completed and another particle is randomly selected to be moved.

If the move results in a configuration of *higher energy*, a probability for that event is calculated, and a random number between zero and one is generated. If the generated number is smaller than the probability of the event, then the move is accepted; otherwise, another particle is selected and the process is repeated. This process continues until the average total energy of the system no longer decreases; at this point, average configuration data are accumulated to yield the mean density distribution of particles in the system.

Monte Carlo simulations require considerably less computation time than molecular dynamic simulations and can yield the same results; however, neither method provides a really practical way to calculate complete isotherms.

## **DENSITY FUNCTIONAL FORMULATION**

*Density functional theory* offers a practical alternative to both molecular dynamic and Monte Carlo simulations. When compared to reference methods based on molecular simulation, this theory provides an accurate method of describing inhomogeneous systems yet requires fewer calculations. Because the density functional theory provides accuracy and a reduced number of calculations, it is the basis embodied in the DFT models.

The system being modeled consists of a single pore represented by two parallel walls separated by a distance  $H$ . The pore is open and immersed in a single component fluid (adsorptive) at a fixed temperature and pressure. Under such conditions, the fluid responds to the walls and reaches an equilibrium distribution. In this condition (by the definition of equilibrium), the chemical potential at every point equals the chemical potential of the bulk fluid. The bulk fluid is a homogenous system of constant density; its chemical potential<sup>1)</sup> 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.

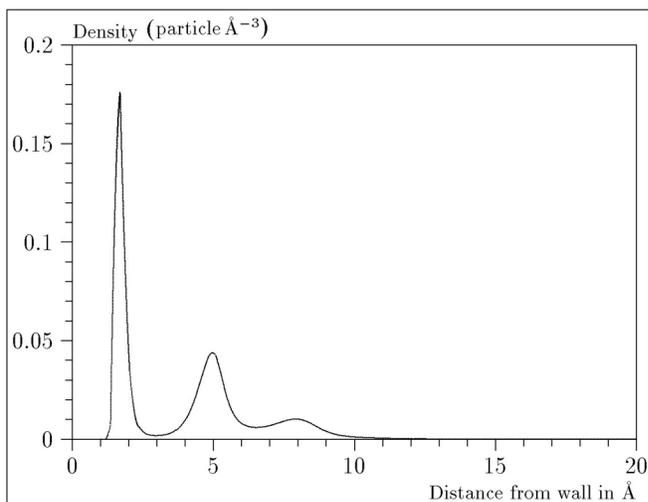
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<sup>1)</sup> Chemical potential may be thought of as the energy change felt by a probe particle when it is inserted into the system from a reference point outside the system. It can also be defined as the partial derivative of the grand potential energy with respect to density (or concentration).

As noted previously, at equilibrium, the whole system has a minimum (Helmholtz) free energy, known thermodynamically as the grand potential energy (GPE). Density functional theory describes the thermodynamic grand potential as a functional of the single-particle density distribution; therefore, calculating the density profile that minimizes the GPE yields the equilibrium density profile. The calculation method requires the solution of a system of complex integral equations that are implicit functions of the density vector. Since analytic solutions are not possible, the problem must be solved using iterative numerical methods. Although calculation using these methods still requires supercomputing speed, the calculation of many isotherm pressure points for a wide range of pore sizes is a feasible task. The complete details of the theory and the mathematics can be found in the papers listed under [DFT Model References on page C - 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.

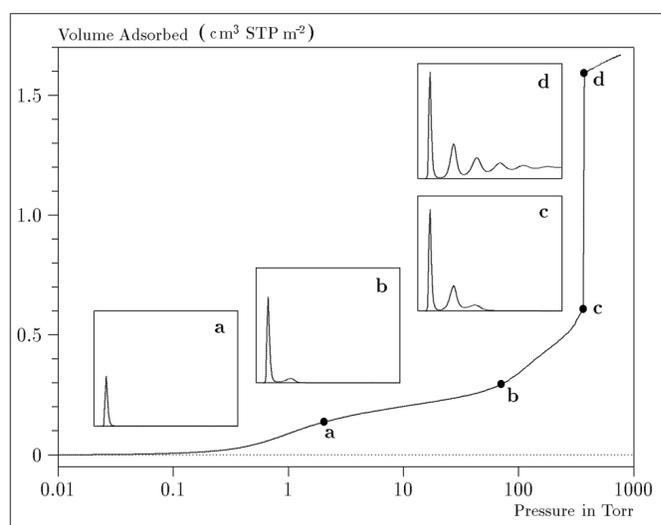


**Figure 1. Density Profile for Argon on Carbon at 87.3 K and a Relative Pressure of 0.5**

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.



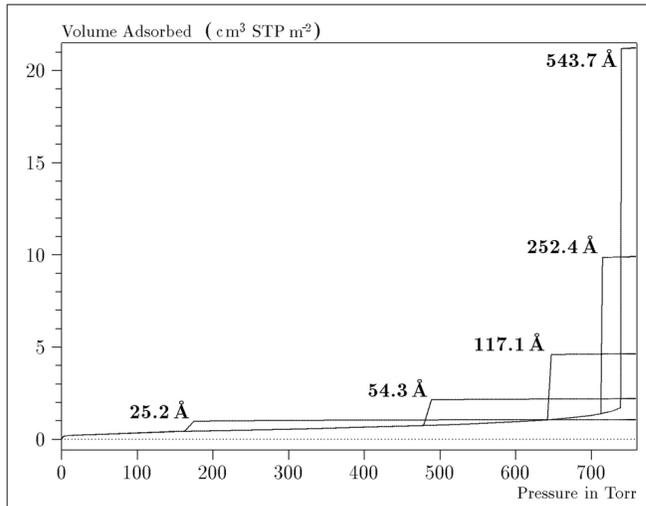
**Figure 2. Model Isotherm for Argon at 87.3 K in a 40 Å Slit in a Carbon Substrate**

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.

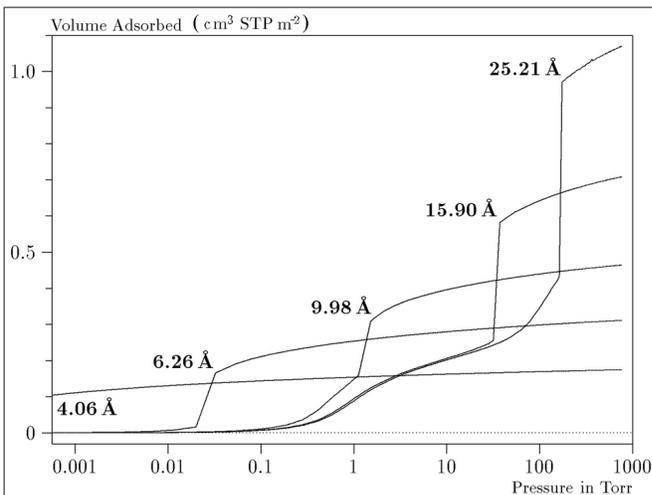
- Inset **a** shows the profile corresponding to point a on the isotherm where the surface is about half covered.
- At point **b**, the first layer is so full that it is more favorable for atoms to start a new layer.
- At point **c**, a third layer is forming. Point **c**, for this size slit, is the 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.

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**

<b>Geometry:</b>	Slit
<b>Substrate:</b>	Carbon (graphite)
<b>Category:</b>	Porosity
<b>Method:</b>	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  $1 \times 10^{-6}$  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**

<b>Geometry:</b>	Free surface
<b>Substrate:</b>	Surface energy
<b>Method:</b>	Nitrogen at 77K

Using the modified Tarazona prescription described by Olivier (see [DFT Model References on page C - 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, ie, for the adsorptive / adsorbent pair divided by Boltzmann constant. The units are therefore Kelvin.

**N2 - Cylindrical Pores - Oxide Surface****AR - Cylindrical Pores - Oxide Surface**

<b>Geometry:</b>	Cylinder
<b>Substrate:</b>	Oxide
<b>Category:</b>	Porosity
<b>Method:</b>	Nitrogen at 77K; Argon at 87K

Model isotherms were calculated using a combination of statistical mechanical calculations and experimental observations for macroporous silicas and MCM-41 mesoporous silicas as well as zeolites. The pore-filling pressures were determined as a function of the pore size from adsorption isotherms on MCM-41 materials characterized by X-ray and other techniques. The variation of the pore fluid density with pressure and pore size has been accounted for by density functional theory calculations. The N2 model reports pore sizes ranging from 3.8 to 387 Å and the AR model from 3.8 to over 500 angstroms.

**References:** M. Jaroniec, M. Kruk, J.P. Olivier, and S. Koch, "A New Method for the Accurate Pore Size Analysis of MCM-41 and Other Silica-Based Mesoporous Materials," Proceedings of COPS-V, Heidelberg, Germany (1999).

**N2 – Cylindrical Pores – Pillared Clay Surface (Montmorillonite)**

<b>Geometry:</b>	Cylinder
<b>Substrate:</b>	Crystalline Silicate
<b>Category:</b>	Porosity
<b>Method:</b>	Nitrogen at 77K

Model isotherms were calculated using a combination of statistical thermodynamic Non-Local Density Functional Theory (NLDFE) calculations and experimental isotherms for reference samples of montmorillonite. The construction method for the hybrid models was analogous to that described in the first reference below (Jaroniec et al, 1999). The additional references add additional theoretical details as well as examples of the application of the model to pillared clay catalysts. This model reports pore widths from 3.8 to 387 angstroms.

**References:** Mietec Jaroniec, Michal Kruk, James P. Olivier and Stefan Koch, "A New Method for the Characterization of Mesoporous Silicas," Proceedings of COPS-V, 1999, Studies in Surface Science, Vol 128, *Characterization of porous Solids V*, Unger, et al, Eds, Elsevier, Amsterdam, 2000.

James P. Olivier and Mario L. Occelli, "Surface Area and Microporosity of a Pillared Interlayered Clay (PILC) from a Hybrid Density Functional Theory

(DFT) Method," *The Journal of Physical Chemistry B*; 2001, 105(3), 623-629.

M. L. Occelli, J. P. Olivier, J. A. Perdigon-Melon, and A. Auroux, "Surface Area, Pore Volume Distribution, and Acidity in Mesoporous Expanded Clay Catalysts from Hybrid Density Functional Theory (DFT) and Adsorption Microcalorimetry Methods," *Langmuir* 2002, 18, 9816-9823.9b.

James P. Olivier, "The Importance of Surface Heterogeneity in Developing Characterization Methods." *6<sup>th</sup> 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**

<b>Geometry:</b>	Cylinder
<b>Substrate:</b>	Carbon
<b>Category:</b>	Porosity
<b>Method:</b>	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.

<b>References:</b>	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)
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**Ar - Zeolites H-Form by NLDFT**

<b>Geometry:</b>	Cylinder
<b>Substrate:</b>	Zeolite
<b>Category:</b>	Porosity
<b>Method:</b>	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<sup>+</sup> and (NH<sub>4</sub>)<sup>+</sup> exchanged zeolites. The reported pore size range is from 3.5 to 300 angstroms.

**Ar - Zeolites Me-Form by NLDFT**

<b>Geometry:</b>	Cylinder
<b>Substrate:</b>	Zeolite
<b>Category:</b>	Porosity
<b>Method:</b>	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 (see the calculations section in [3Flex Links on page iv](#)). 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**

<b>Geometry:</b>	Slit
<b>Substrate:</b>	Average
<b>Category:</b>	Porosity
<b>Method:</b>	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:

<b>Surface tension =</b>	8.88 dynes cm <sup>-1</sup>
<b>Molar density =</b>	0.02887 g cm <sup>-3</sup>

#### **N2 - Halsey Thickness Curve**

<b>Geometry:</b>	Cylinder
<b>Substrate:</b>	Average
<b>Category:</b>	Porosity
<b>Method:</b>	Nitrogen 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

**Reference:** G. Halsey, J. Chem. Phys 16, 931 (1948).

### Kelvin Equation with Harkins and Jura Thickness Curve

#### **N2 - Harkins and Jura Thickness Curve**

**Geometry:** Slit  
**Substrate:** Average  
**Category:** Porosity  
**Method:** Nitrogen 77 K

The kernel function is calculated using the Harkins and Jura equation with standard parameters:

$$t = \left( \frac{13.99}{0.034 - \log(P/P_0)} \right)^{1/2}$$

The nitrogen properties used in the Kelvin equation are:

**Surface tension =** 8.88 dynes cm<sup>-1</sup>  
**Molar density =** 0.02887 g cm<sup>-3</sup>

#### **N2 - Harkins and Jura Thickness Curve**

**Geometry:** Cylinder  
**Substrate:** Average  
**Category:** Porosity  
**Method:** Nitrogen 77 K

The calculation is the same as above except that cylindrical geometry is assumed.

**References:** W. D. Harkins and G. Jura, J.A.C.S. 66, 1366 (1944).  
 J. H. DeBoer et al., J. Colloid and Interface Sci. 21, 405 (1966).

### Kelvin Equation with Broekhoff-de Boer Thickness Curve

#### **N2 - Broekhoff-de Boer Model**

**Geometry:** Cylinder  
**Substrate:** Average  
**Category:** Porosity  
**Method:** Nitrogen 77 K

The kernel function is calculated using the Broekhoff-de Boer equation with standard parameters:

$$\log\left(p/p^0\right) = \frac{-16.11}{t^2} + 0.1682^{-0.1137t}$$

The nitrogen properties used in the Kelvin equation are:

<b>Surface tension =</b>	8.88 dynes cm <sup>-1</sup>
<b>Molar density =</b>	0.02887g cm <sup>-3</sup>

## N2 - Broekhoff-de Boer Model

<b>Geometry:</b>	Cylinder
<b>Substrate:</b>	Average
<b>Category:</b>	Porosity
<b>Method:</b>	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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## ***D FREE SPACE CORRECTION***

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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<sub>2</sub> at approximately 77 K) is situated so that it can be raised to immerse most of the sample tube. Two temperature zones exist within the sample tube when immersed in the cryogenic bath: a warm zone (the volume above the liquid level and near ambient temperature) and a cold zone (the volume below the liquid level at cryogenic temperature). Not only must the total free space volume be determined, but it also is necessary to determine the quantity of gas residing within the “cold” zone since a nonideality correction must be applied to only that quantity of gas.

The total quantity of gas in the partly immersed sample holder cannot simply be determined using  $n = PV/RT$  because temperature is not constant over the total volume, but instead is distributed as two temperature zones with a steep temperature gradient between them. A convenient method for resolving this problem is to derive two factors which, for the existing temperature profile, can be multiplied by the prevailing pressure to reveal the molar volume of gas contained in the cold zone and the total quantity residing in the free volume of the immersed sample holder (the cold free space).

The 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. Warm and cold free spaces are calculated from (1) system volume, (2) system, ambient, and bath temperatures, and (3) measured pressures. From these, the value of the portion of cold free space at cryogenic temperature which requires correction for nonideality can be determined.

This method may be undesirable if:

- Helium is unavailable; free space determination 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 warm and cold free spaces. The values to enter may be obtained in one of two ways:

- A pre-analysis free space calibration of the sample tube containing sample
- The total free space of an empty sample tube is measured and the displacement of the sample calculated from its mass and density and subtracted from the total free space

In either procedure, ensure that the level (or, in cases where the Isothermal Jacket is used, the effective level) of the cryogen bath on the sample tube is the same when the analysis is performed as it was when gathering data for free space calculations.

## E GAS CHARTS

### RELATIVE THERMAL CONDUCTIVITY OF GASES

Name	Chemical Formula	Conductivity (Relative to Air)
Air		1.00
Ammonia	NH <sub>3</sub>	0.92
Argon	Ar	0.68
Butane	C <sub>4</sub> H <sub>10</sub>	0.60
Carbon Dioxide	CO <sub>2</sub>	0.62
Carbon Monoxide	CO	0.97
Ethane	C <sub>2</sub> H <sub>6</sub>	0.79
Helium	He	5.84
Hydrogen	H <sub>2</sub>	7.07
Krypton	Kr	0.37
Methane	CH <sub>4</sub>	1.29
Neon	Ne	1.87
Nitric Oxide	NO	0.99
Nitrogen	N <sub>2</sub>	1.00
Nitrogen Dioxide	NO <sub>2</sub> or N <sub>2</sub> O <sub>4</sub>	1.51
Nitrous Oxide	N <sub>2</sub> O	0.65
Oxygen	O <sub>2</sub>	1.02
Sulfur Dioxide	SO <sub>2</sub>	0.38
Water Vapor	H <sub>2</sub> O	0.67

## TYPICAL GASES USED

Flow Rate of 50 cm <sup>3</sup> /min				
Test	Gases			Other
	Preparation	Carrier	Loop	
<b>TPR Experiment</b>	Argon	10% H <sub>2</sub> in Argon	N/A	
Calibration	N/A		Argon	TCD Level Calibration
<b>TPD Ammonia</b>	Helium or 15% NH <sub>3</sub> in Helium	Helium	N/A	
Calibration	N/A		NH <sub>3</sub> in Helium	TCD Level Calibration
<b>TPD Pyridine</b>	Helium	Helium	Helium	Pyridine in Vapor Generator
Calibration	N/A			User-defined Pyridine in Vapor Generator
<b>TPD Hydrogen</b>	10% H <sub>2</sub> in Argon	Argon	N/A	Calibration
Calibration	N/A		10% H <sub>2</sub> in Argon	TCD Level Calibration
<b>TPD Oxygen</b>	10% O <sub>2</sub> in Helium	Helium	N/A	
Calibration	N/A		10% O <sub>2</sub> in Helium	TCD Level Calibration
<b>TPO Experiment</b>	Helium	10% O <sub>2</sub> in Helium	N/A	
Calibration	N/A		Helium	TCD Level Calibration
<b>H<sub>2</sub> Pulse Chemisorption</b>	10% H <sub>2</sub> in Argon	Argon	10% H <sub>2</sub> in Argon	
<b>CO Pulse Chemisorption</b>		Helium	10% CO in Helium	
Calibration	Not Required			
<b>BET Surface Area</b>	Helium	30% N <sub>2</sub> in Helium	N/A	
Calibration	N/A			User-defined manual injections of N <sub>2</sub> (0.5, 1.0, 1.5, and 2.0 cm <sup>3</sup> )

## GAS CONVERSION CONSTANTS

The ChemiSorb analyzer uses Mass Flow Controllers (MFCs) to control the flow of gases. These MFCs require a conversion constant for each gas or gas mixture, to compensate for variations in gas flows resulting from variations in the properties of gases. A default gas table containing MFC conversion constants is included on the *Options* menu. The following table provides a more complete list of gases and their conversion constants.

### Gas Conversion Constants for the MFCs

Gas	Symbol	MFC Conversion Constant (H <sub>2</sub> = 1.000)
Acetylene	C <sub>2</sub> H <sub>2</sub>	0.6535
Air (mixture)		0.9901
Allene	C <sub>3</sub> H <sub>4</sub>	0.4752
Ammonia	NH <sub>3</sub>	0.7822
Argon	Ar	1.3861
Arsine	AsH <sub>3</sub>	0.7525
Boron Trichloride	BCl <sub>3</sub>	0.4356
Boron Trifluoride	BF <sub>3</sub>	0.5743
Bromine Pentafluoride	BrF <sub>5</sub>	0.2871
Bromine Trifluoride	BrF <sub>3</sub>	0.4356
Butane	C <sub>4</sub> H <sub>10</sub>	0.2871
Butene	C <sub>4</sub> H <sub>8</sub>	0.3267
Carbon Dioxide	CO <sub>2</sub>	0.7723
Carbon Monoxide	CO	0.9802
Carbon Tetrachloride	CCl <sub>4</sub>	0.3465
Carbon Tetrafluoride	CF <sub>4</sub>	0.4752
Carbonyl Fluoride	COF <sub>2</sub>	0.2673
Carbonyl Sulfide	COS	0.6733
Chlorine	Cl <sub>2</sub>	0.8218
Chloroform	CHCl <sub>3</sub>	0.4356
Chlorine Trifluoride	ClF <sub>3</sub>	0.4257
Cyanogen	C <sub>2</sub> N <sub>2</sub>	0.4950

## Gas Conversion Constants for the MFCs (continued)

Gas	Symbol	MFC Conversion Constant (H <sub>2</sub> = 1.000)
Cyclopropane	C <sub>3</sub> H <sub>6</sub>	0.5050
Deuterium	D <sub>2</sub>	0.9901
Diborane	B <sub>2</sub> H <sub>6</sub>	0.5446
Dichlorosilane	SiH <sub>2</sub> Cl <sub>2</sub>	0.4356
Dimethylamine	(CH <sub>3</sub> ) <sub>2</sub> NH	0.6634
Dimethylether	(CH <sub>3</sub> ) <sub>2</sub> O	0.5842
Ethane	C <sub>2</sub> H <sub>6</sub>	0.5446
Ethyl Chloride	C <sub>2</sub> H <sub>5</sub> Cl	0.2871
Ethylene	C <sub>2</sub> H <sub>4</sub>	0.6139
Ethylene Oxide	C <sub>2</sub> H <sub>4</sub> O	0.5842
Fluorine	F <sub>2</sub>	0.9208
Fluoroform	CHF <sub>3</sub>	0.5644
Freon 11	CCl <sub>3</sub> F	0.3762
Freon 12	CCl <sub>3</sub> F <sub>2</sub>	0.3861
Freon 13	CClF <sub>3</sub>	0.4257
Freon 13 B1	CBrF <sub>3</sub>	0.4059
Freon 14	CF <sub>4</sub>	0.4703
Freon 21	CHCl <sub>2</sub> F	0.4554
Freon 22	CHClF <sub>2</sub>	0.5050
Freon 23	CHF <sub>3</sub>	0.5644
Freon 113	CCl <sub>2</sub> F-CClF <sub>2</sub>	0.2277
Freon 114	CCl <sub>2</sub> F <sub>4</sub> -CClF <sub>2</sub>	0.2554
Freon 115	CClF <sub>2</sub> -CF <sub>3</sub>	0.2713
Freon 116	CF <sub>3</sub> -CF <sub>3</sub>	0.2277
Germane	GeH <sub>4</sub>	0.6436

## Gas Conversion Constants for the MFCs (continued)

Gas	Symbol	MFC Conversion Constant (H <sub>2</sub> = 1.000)
Helium	He	1.3762
Hexamethyldisizane	HMDS	0.1386
Hydrogen	H <sub>2</sub>	1.0000
Hydrogen Bromide	HBr	0.9703
Hydrogen Chloride (Dry)	HCl	0.9802
Hydrogen Fluoride	HF	0.9901
Hydrogen Iodide	HI	0.9505
Hydrogen Selenide	H <sub>2</sub> Se	0.8317
Hydrogen Sulfide	H <sub>2</sub> S	0.8416
Isobutane	C <sub>4</sub> H <sub>10</sub>	0.3069
Isobutylene	C <sub>4</sub> H <sub>8</sub>	0.3366
Krypton	Kr	1.3762
Methane	CH <sub>4</sub>	0.8020
Methylamine	CH <sub>3</sub> NH <sub>2</sub>	0.5644
Methyl Bromide	CH <sub>3</sub> Br	0.6436
Methyl Chloride	CH <sub>3</sub> Cl	0.6832
Methyl Fluoride	CH <sub>3</sub> F	0.7525
Methyl Mercaptan	CH <sub>4</sub> S	0.5842
Neon	Ne	1.3861
Nitric Oxide	NO	0.9901
Nitrogen	N <sub>2</sub>	0.9950
Nitrogen Dioxide	NO <sub>2</sub>	0.7525
Nitrogen Trioxide	N <sub>2</sub> O <sub>3</sub>	0.4356
Nitrogen Trifluoride	NF <sub>3</sub>	0.5446
Nitrous Oxide	N <sub>2</sub> O	0.7426

## Gas Conversion Constants for the MFCs (continued)

Gas	Symbol	MFC Conversion Constant (H <sub>2</sub> = 1.000)
Oxygen	O <sub>2</sub>	0.9802
Ozone	O <sub>3</sub>	0.7327
Pentaborane	B <sub>5</sub> Hg	0.2871
n Pentane	C <sub>5</sub> H <sub>12</sub>	0.2376
Perchloryl Fluoride	ClO <sub>3</sub> F	0.4455
Phosgene	COCl <sub>2</sub>	0.5050
Phosphine	PH <sub>3</sub>	0.7822
Phosphorous Pentafluoride	PF <sub>5</sub>	0.3465
Propane	C <sub>3</sub> H <sub>8</sub>	0.3861
Propylene (Propene)	C <sub>3</sub> H <sub>6</sub>	0.4653
Silane	SiH <sub>4</sub>	0.6733
Silicon Tetrachloride	SiCl <sub>4</sub>	0.3168
Silicon Tetrafluoride	SiF <sub>4</sub>	0.3960
Sulfur Dioxide	SO <sub>2</sub>	0.7228
Sulfur Hexafluoride	SF <sub>6</sub>	0.2970
Trichlorosilane	Cl <sub>3</sub> HSi	0.3267
Trimethylamine	(CH <sub>3</sub> ) <sub>3</sub> N	0.3168
Tungsten Hexafluoride	WF <sub>6</sub>	0.2871
Uranium Hexafluoride	UF <sub>6</sub>	0.2178
Vinyl Bromide	C <sub>2</sub> H <sub>3</sub> Br	0.5248
Vinyl Chloride	C <sub>2</sub> H <sub>3</sub> Cl	0.5347
Vinyl Fluoride	C <sub>2</sub> H <sub>3</sub> F	0.5743
Xenon	Xe	1.3762

## ***F MAINTAIN HIGH PURITY GASES***

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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

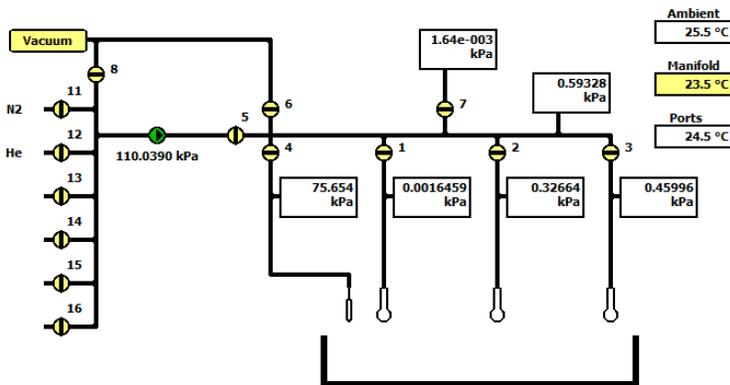
This is the preferred method for removing trapped air.

1. Go to **Unit > Enable Manual Control** (if the analyzer schematic is not displayed, go to **Unit > Show Instrument Schematic**).

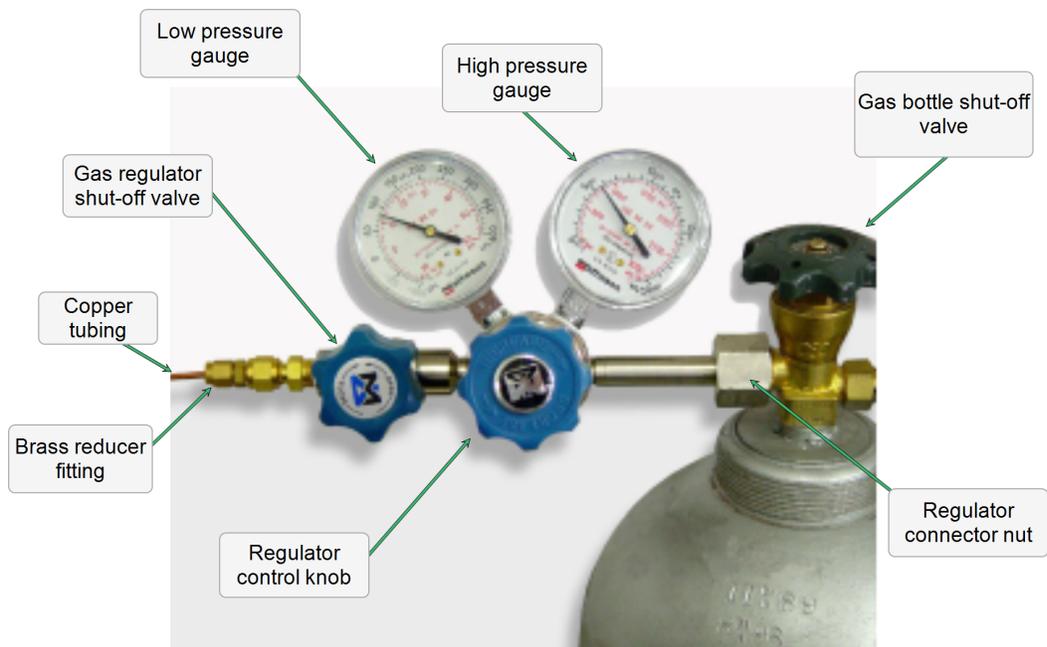


If multiple instruments are installed, choose the correct *Unit* menu.

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.



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...
<b>The regulator has not been filled with gas and the gas line is attached to the instrument:</b>	Close the gas cylinder valve.
	Open the regulator shutoff valve.
<b>The regulator is filled with gas:</b>	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.

## G PEAK DETECTION / INTEGRATION OPTIONS

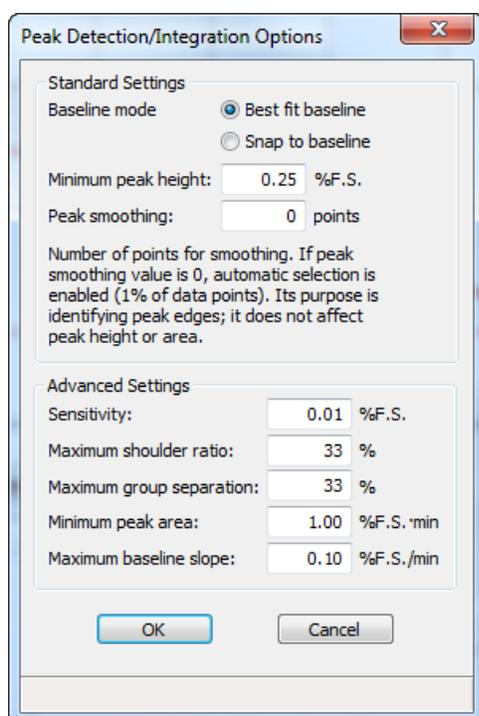


This section applies only to dynamic analysis samples.

### **File Open > [.RPO file] > [Integration button]**

(or select the *Peaks* tab while in the *Peaks Editor*, right click the graph area, and select *Integration Options*)

Peak detection parameters can be customized using the **Integration** button on the *Report Options* window. Peak detection options can be customized while creating the sample file or after analysis.



The TCD detects and records all deviations from baseline, but only those which satisfy the criteria established in this window are reported as peaks.



Peak detection is accomplished through a combination of noise, height, and area thresholds.

## ***BASELINE MODE***

The Baseline Mode affects how the *Find All Peaks* function works.

If *Best Fit Baseline* is selected, the bottom of the peaks is placed in the baseline that best describes the signal outside the range of the peaks. This assumes a linear baseline between the beginning and the end of the peak.

If *Snap to Signal* is selected, the bottom of the peaks is moved to the signal recorded, not the best fit baseline between the peaks.

## ***MINIMUM PEAK HEIGHT***

Sets the minimum height for peaks to be identified and included in the peak table. This value is expressed in terms of the trace's Y-axis units. Use a value of 0 (zero) to include all peaks.

## ***SMOOTHING***

Smoothing allows the application to average the points before using them, so that noise spikes are ignored. Specify the number of points to average into a single value during the peak picking process.

The smoothing parameter can be turned off by setting the value at 1 or 0 (zero). A setting of 1 disables smoothing, and the peak edges are interpolated to the best X-axis value. A setting of 0 also disables smoothing, but the peak edges (the points where the peak begins and ends) are not interpolated. Instead, the nearest data point is used as the peak edge.

## ***SENSITIVITY***

Sensitivity sets the noise rejection level for identifying the peaks in a trace. Use a value from -100 to 100%. For example, a sensitivity level of 5% means that 5% of all local maxima in the trace are greater than the noise and are, therefore to be considered as peaks. A value of 100% identifies all local maxima (or minima for traces with transmission Y units) as peaks. One possible exception to note is the combination of other rejection parameters (such as *Minimum Peak Area* and *Minimum Peak Height*) which can reject peaks even when the sensitivity is set to 100%.

The sensitivity can also be set to negative values to define a specific noise level (in Y units) for peak rejection. For example, a sensitivity setting of -2.5% sets the noise rejection to 2.5 V. This means that maxima with an amplitude of 2.5 V or less will be considered as baseline noise instead of as peaks. (As opposed to the *Minimum Peak Height* rejection parameter which eliminates refined peaks by using their height above the baseline.)

A setting of 0 (zero) automatically sets a default noise level for the trace.

## MAXIMUM SHOULDER RATIO

There can also be *shoulder peaks* (also called *combination peaks*) within a group. (See [Maximum Group Separation on the next page](#) for a description of peak groups.) Shoulders are usually small peaks that are overlapped on the front or the tail of a larger peak. These peaks can also be called *leaders* and *followers*, respectively. As with baseline groups, the areas of these peaks can be calculated incorrectly. If the larger *parent* peak has a long tail with a much smaller peak riding on it, most of the area under the trace belongs to the parent peak. However, if the area of these peaks was determined using baseline grouping, the smaller peak would be calculated by using vertical drop lines at the edges. This would give the parent peak too little area, and the rider peak too much.

The application can detect these shoulder peaks. The areas of shoulder peaks are calculated by drawing a skimmed baseline from the leading edge to the trailing edge. Either an exponential or a straight skim line can be used. The skim type is specified by a secondary method parameter (see the Grams/32 manual Appendix or the Method Editor) and the default is exponential skimming. The remaining area between the shoulder peak baseline and the group baseline is considered to be part of the parent peak.

The *Max Shoulder Ratio* parameter is used to specify whether the peaks that are overlapped in the front or the tail of much larger peaks should be identified as shoulder peaks. To use shoulder peak detection, use a non-zero value for the *Max Shoulder Ratio* parameter. After a baseline group has been identified, the application looks for peaks within the group that satisfy the following shoulder peak criteria:

Shoulders must have a significantly higher Y value at one edge than the other. More importantly, the height of the shoulder above the common value must be much smaller than the height of the “parent” (larger) peak above the same valley. It must be smaller by the Max Shoulder Ratio setting. For example, a setting of 33 implies that shoulders must be smaller than 33% of their parents in terms of height above the common valley. The areas for shoulder peaks are calculated by drawing a skimmed baseline from the left edge to the right edge of the peak. The remaining area between the shoulder peak baseline and the group common baseline is considered to be part of the parent peak.

Shoulder peaks can only be calculated within a group of peaks. (See [Maximum Group Separation on the next page](#) for a description of peak groups.) If the *Max Group Separation* parameter is set to 0 (no groups), a *Max Shoulder Ratio* parameter value is not used. Use a value of zero to specify no shoulder peak detection.

A value of 33% works well with most data. Use a setting of zero to treat shoulder peaks with a perpendicular drop to the common group baseline instead of a skimmed baseline.

If the application detects unwanted baseline noise peaks, try increasing the *Sensitivity* setting. Conversely, if some peaks are not detected, decrease the value.

## **MAXIMUM GROUP SEPARATION**

The application normally calculates peak areas by drawing a *valley-to-valley* baseline from the leading edge to the trailing edge of every identified peak. However, in many traces, the valleys between peaks do not always drop back to the original baseline. If a *valley-to-valley* baseline is used for this type of peak, the calculated area does not accurately reflect the true area under the peak.

The application provides a parameter that allows the calculation of *Baseline Groups*. A group of peaks is defined by a common baseline that extends from the leading edge of the first peak in the group to the trailing edge of the last. The areas of grouped peaks are calculated by dropping vertical lines from the peak edges down to the group baseline. (Note there may also be *Report Groups* defined by the method and assigned group letters from A through Z. Unlike these *Baseline Groups*, the *Report Groups* need not be next to one another. The two types of groups are not related.)

The Max Group Separation parameter is used to determine which peaks in a trace have a common baseline. When using peak grouping, the application compares the width (actually double the largest half width) of every identified peak in a trace to the width of the following peak. The *Max Group Separation* parameter specifies a percentage of the smallest of these two widths in X units. If the edges of two adjacent peaks differ by less than this value, the two peaks constitute a group and are given a common baseline. For example, if two adjacent peaks in the trace have largest half widths of 1 and 1.5 respectively, and the *Max Group Separation* parameter is set at 20%, then a difference of less than 0.4 X units between the adjacent edges of these peaks would make them a group with a common baseline. If the same two peaks have adjacent edges that are greater than 0.4 X units apart, they do not define a group, and each peak has its own separate baseline.

The areas for grouped peaks are calculated by drawing imaginary vertical lines from the peak edges to the common baseline. Any peaks that share common edges are automatically considered a group and are given a common baseline for any *Max Group Separation* setting greater than 0. To specify no peak grouping (each identified peak has its own baseline), use a setting of 0. A value of 33% for this parameter works well with most data. Use a setting of zero to force all baselines to be drawn from peak valley to valley.

## **MINIMUM PEAK AREA**

This parameter sets the minimum area required for refined, processed peaks to be recognized, identified, and included in the peak table. Any peak with a calculated peak area smaller than the current setting is not detected. Values for this parameter are expressed in terms of the trace X-axis units multiplied by the Y-axis units (e.g., millivolt-minutes).

Setting the *Sensitivity* parameter to large values (greater than 20%) can cause noise spikes (or dips) on the sides of major peaks to be identified as peaks themselves. If the areas of the peaks are smaller than the *Minimum Peak Area* parameter, major peaks in the trace may not be identified at all. Exercise caution when using high Sensitivity settings with the *Minimum Peak Area* parameter.

If peaks are not detected by the application, lower the setting with a smaller value. This parameter must be adjusted in conjunction with the *Peak Sensitivity* and/or *Minimum Peak Height* parameters above. Peak rejection is accomplished through a combination of peak height and peak area rejection parameters. If the application is not detecting the peak(s) of interest, decrease both parameters.

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---

## *H ADVANCED REPORTS - PYTHON MODULE*

---

See [Mic Module Python Calls on page H - 18](#)

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.

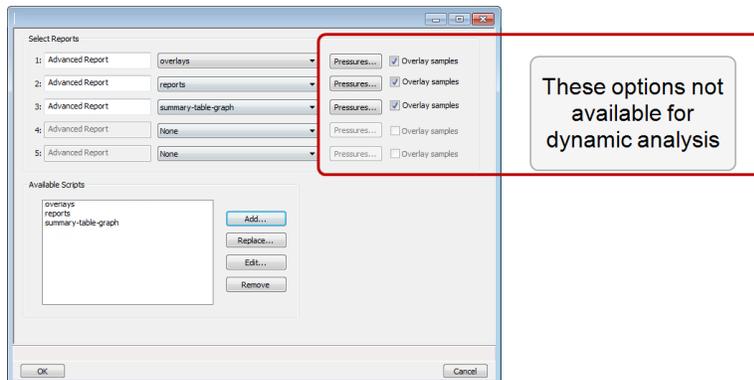
## ADVANCED REPORT OPTIONS



The *Selected Reports* list box may display a *User-Defined* option rather than an *Advanced* option. These options are the same.

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 H - 18](#) 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* 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. **PC** Click **Pressures** to add pressure points to the report. Click **OK** to return to the *Report Options* tab.
8. **PC** 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 Report Options Fields and Buttons Table

Field or Button	Description
<b>Add</b> [ <i>button</i> ]	Click to add additional Python reports.
<b>Available Scripts</b> [ <i>group box</i> ]	Lists the available reports and provides the option to add, replace, edit or remove reports.
<b>Overlay samples</b> (if shown) [ <i>check box</i> ] <b>PC</b>	Use to overlay samples as defined by the function.
<b>Advanced Report 1 through 5</b> [ <i>drop-down box</i> ]	Use the drop-down lists to select currently-defined functions used to define the report calculations and output.
 For fields and buttons not listed in this table, see <a href="#">Common Fields and Buttons on page 2 - 4</a> .	

## 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.

## 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
<b>Add</b>	Adds one or more scripts to the <i>Available Scripts</i> box. The added scripts then become available as options in the <i>Selected Reports</i> section.
<b>Edit</b>	Edits the script stored within the application but does not affect the original .py text file.
<b>Overlay samples</b>	Select to enable the overlay sample files process.
<b>Pressures</b>	Select to include or exclude pressures from the report. <ul style="list-style-type: none"> <li>• <b>Calculation pressure range.</b> Enter the minimum and maximum pressures to be used in the pressure table.</li> <li>• <b>Cancel.</b> Discards any changes or cancels the current process.</li> <li>• <b>Exclude All.</b> Select to exclude all pressure points in the table.</li> <li>• <b>Include All.</b> Select to include all pressure points in the table.</li> <li>• <b>OK.</b> Saves and closes the active window.</li> </ul>
<b>Remove</b>	Removes the script from the <i>Available Scripts</i> box but does not affect original .py text file
<b>Replace</b>	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:

<p><b>Summary A</b> label 1: val1 label 2: val2 label 3: val3</p> <p><b>Summary B</b> label 4: val4 label 5: val5 label 6: val6</p>
---

## TABULAR REPORT

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

```
import mic
import numpy as np

mic.table("My Tables")

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

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

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

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

The result is:

My set A		
x	y	
1.0	0.5	
2.0	1.0	
3.0	1.5	

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

## GRAPHIC REPORT

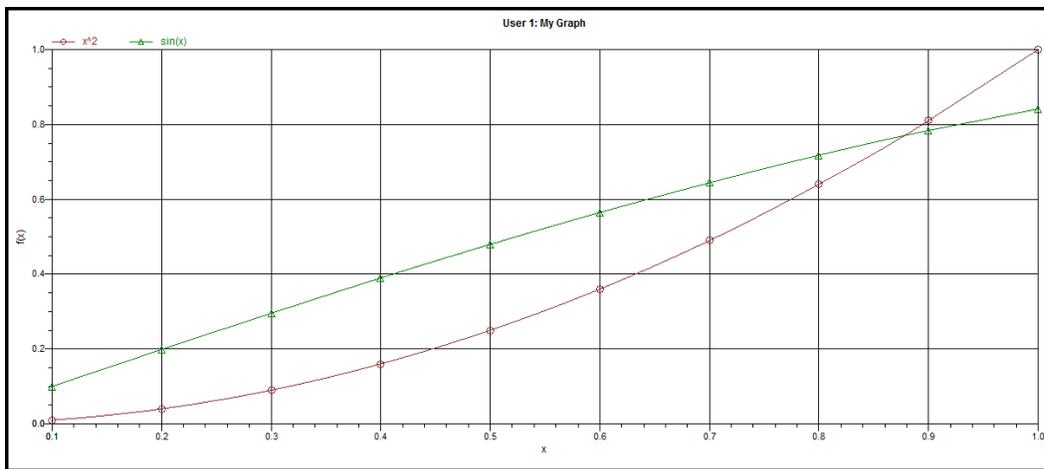
This script is an example of the mic module producing a graph with two curves:

```
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 FOR PHYSICAL ADSORPTION

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

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

```
import mic

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

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

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

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

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

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

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

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

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

## ACQUIRE BASIC INFORMATION FOR CHEMICAL ADSORPTION

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

To acquire the adsorption isotherm and other basic information about the sample being edited, the calls *mic\_chem.isotherm*, *mic.sample\_information* and *mic.adsorptive\_data* are applied.

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

```
import mic

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

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

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

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

```
"%8.3f" % dcf,  
"%8.3f" % mol_weight,  
analysis_gas ] )
```

## ACQUIRE REPORT RESULTS

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

```
import mic  
  
sa = mic.report("bet", "surface area")  
c = mic.report("bet", "bet constant")  
vm = mic.report("bet", "monolayer capacity")  
esa = mic.report("tplot", "external surface area")  
vol = mic.report("tplot", "micropore volume")  
  
mic.summary( "BET and T-plot Results" )  
  
mic.summary.add( "Report Results",  
[ "bet surface area",  
  "bet constant",  
  "bety 6" ,  
  "tplot external surface area",  
  "tplot micropore volume"],  
[ "%10.5f" % sa,  
  "%10.5f" % c,  
  "%10.5f" % vm,  
  "%10.5f" % esa,  
  "%10.5f" % vol ] )
```

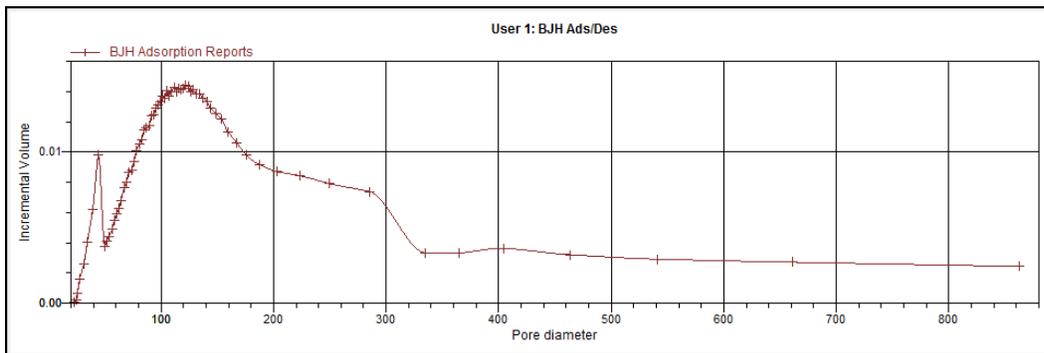
The result is:

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

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

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

The result is:



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

## ACQUIRE OVERLAY SAMPLE DATA FOR PHYSICAL ADSORPTION

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

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

```
import mic

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

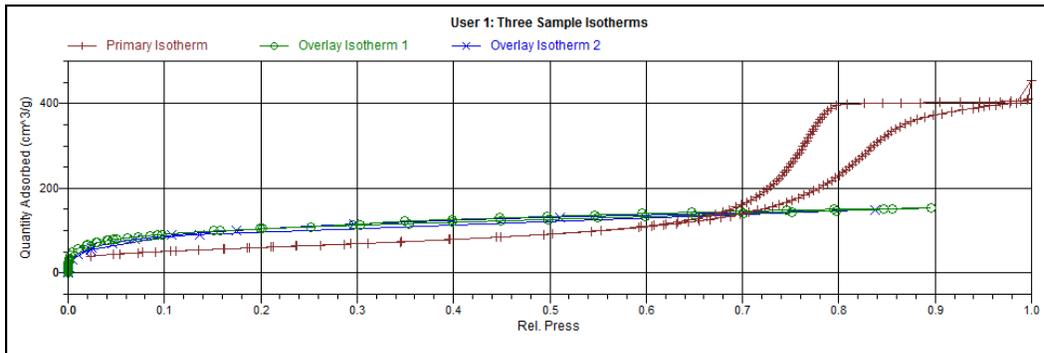
mic.graph( 'Three Sample Isotherms',
          'Rel. Press',
          'Quantity Adsorbed (cm^3/g)' )

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

mic.summary( "A summary report" )

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

The results are:



**Two samples**

Primary Sample: 12 mm Tube N2 Silica-Alumina ADS-DES with FS  
Overlay Sample 1: Activated Carbon Hexane Dosed from Port 3 - 2  
Overlay Sample 2: Activated Carbon Tube C4 Butane Port 3

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

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

## ACQUIRE OVERLAY SAMPLE DATA FOR CHEMICAL ADSORPTION

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

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

```
import mic

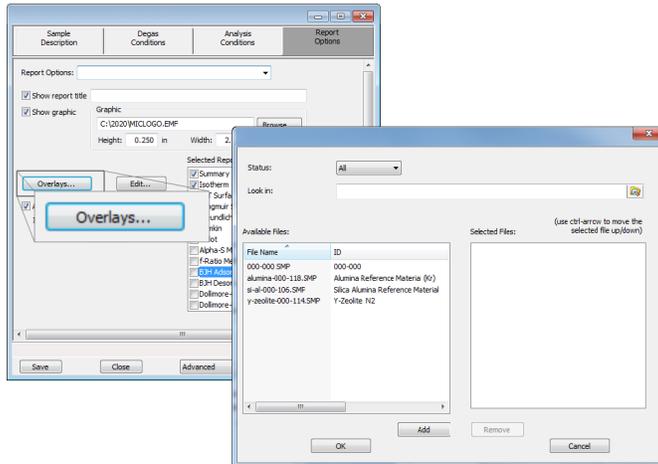
p0, q0      = mic.chem_isotherm('primary')
p0r, q0r    = mic.chem_isotherm('repeat')
p1, q1      = mic.chem_overlay(1, 'primary')
p1r, q1r    = mic.chem_overlay(1, 'repeat')
p2, q2      = mic.chem_overlay(2, 'primary')
p2r, q2r    = mic.chem_overlay(2, 'repeat')
mic.graph( 'Graphical Report 1', 'Abs.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

mic.summary( "A summary report" )

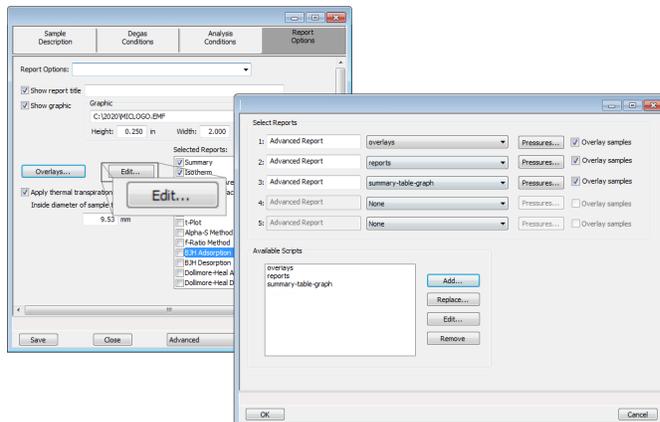
mic.summary.add( "Sample and Two Overlays",
                [ "Primary Sample:",
                  "Overlay Sample 1:",
                  "Overlay Sample 2:" ],
                [ mic.sample_information('sample description'),
                  mic.sample_information('sample description',1),
         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

## ENABLE THE USE OF OVERLAY DATA

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



- Double click a file name in the *Available Files* box to move the file to the *Selected Files* box.
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 H - 3](#).



## ACQUIRE METAL COMPOSITION DATA FOR CHEMICAL ADSORPTION

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

```
import mic
import pprint as pp

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

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

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

## ***MIC MODULE PYTHON CALLS***

### ***TABLES***

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='-', marker='a', graphtype='both' )
```

Keyword arguments:

```

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

```

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

## ***ADD A CURVE USING THE SECOND Y-AXIS***

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

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

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

## ***CREATE A NEW GRAPHICAL REPORT***

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

Keyword arguments:

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

## **GET PRIMARY ISOTHERM DATA FOR CHEMICAL ADSORPTION**

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

Keyword arguments:

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

Usage:

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

## **GET PRIMARY ISOTHERM DATA**

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

Keyword arguments:

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

Usage:

```
p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.overlay(1, 'rel')

p    --- array of pressure (relative or absolute);
      empty-array if overlay is unavailable
qads --- array of cumulative quantity adsorbed;
      empty-array if overlay is unavailable
num_ads --- number of points in the adsorption curve;
          0 if overlay is unavailable
warm_fs --- warm free-space; 0.0 if overlay is unavailable
cold_fs --- cold free-space; 0.0 if overlay is unavailable
mass    --- sample mass; 0.0 if overlay is unavailable
desc    --- sample description; empty-string if
          overlay is unavailable
```

## GET OVERLAY ISOTHERM DATA FOR CHEMICAL ADSORPTION

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

Get overlay isotherm data.

Keyword arguments:

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

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

Usage:

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

## GET OVERLAY ISOTHERM DATA

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

Keyword arguments:

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

Usage:

```
p, qads, num_ads, warm_fs, cold_fs, mass, desc = mic.overlay(1, 'rel')
```

```
p    --- array of pressure (relative or absolute);
      empty-array if overlay is unavailable
qads --- array of cumulative quantity adsorbed;
      empty-array if overlay is unavailable
num_ads --- number of points in the adsorption curve;
          0 if overlay is unavailable
warm_fs --- warm free-space; 0.0 if overlay is unavailable
cold_fs --- cold free-space; 0.0 if overlay is unavailable
mass    --- sample mass; 0.0 if overlay is unavailable
```

```
desc      --- sample description; empty-string if
           overlay is unavailable
```

## GET ADSORPTIVE DATA FOR EACH SAMPLE

```
mic.adsorptive_data( sample_number = 0 )
```

Keyword arguments:

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

Usage:

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

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

## GET SAMPLE INFORMATION ITEM

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

Keyword arguments:

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

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

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

Usage:

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

## GET REPORT RESULTS

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

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

Keyword arguments:

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

Usage:

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

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

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

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

```
porewidth --- array of pore dimension boundaries (angstroms);
              empty-array if unavailable.
incvol      --- array of incremental pore volumes (cm3/g);
              empty-array if unavailable.
desc       --- Name of data set; empty-string if unavailable.
```

## **GET IMPORTED PORE DATA**

```
mic.imported_pore_data( import_number = 1 )
```

Keyword arguments:

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

Usage:

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

```
xdat --- array of pore dimension boundaries (angstroms);  
empty-array if unavailable.
```

```
ydat --- array of incremental pore volumes (cm3/g);  
empty-array if unavailable.
```

```
desc --- Name of data set; empty-string if unavailable.
```

## **GET METAL COMPOSITION FOR CHEMICAL ADSORPTION**

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

Get information about the active metals in this sample

Keyword arguments:

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

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

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

Usage:

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

In the above first usage case, the list of active metals is returned.

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

The metal\_property keywords which one can use are

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

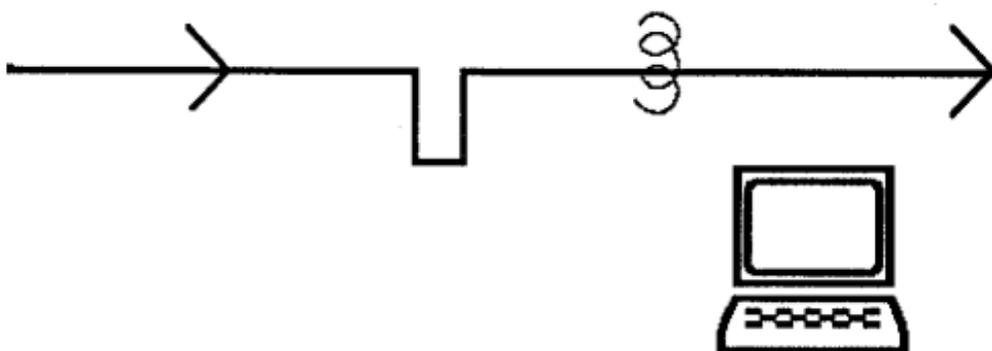
Or as just mentioned, one can make the call `metal_composition( metalname )` without any metal\_property keyword provided to see the whole dictionary.

## I TEMPERATURE PROGRAMMED ANALYSES

---

Most temperature-programmed experiments are based on the following highly simplified steps:

1. Gas flows into the analyzer.
2. The gas interacts with the sample as the temperature changes.
3. Gas flows past the detector.
4. The detector collects data.
5. The application plots and calculates results.



### How the Detector Works

The detector contains heated filaments that measure the difference in gas thermal conductivity sensed between the gases flowing over the sample and reference filaments.

The gases flowing past the detector cool the filament by extracting heat. How quickly any type of gas removes heat from the detector is determined by its thermal conductivity<sup>1)</sup>. A gas with a high thermal conductivity cools the filament rapidly, and more power is required to maintain its temperature. A gas with a lower thermal conductivity removes heat from the filament more slowly.

When the sample reacts with the gas, it causes changes in the composition of the gas and, consequently, changes the thermal conductivity of the gas. These changes are sensed by the detector as an increase or decrease in the amount of power required to maintain the filament at a constant temperature.

---

1) The thermal conductivity of a gas is its ability to conduct heat. Each gas has a distinct thermal conductivity.

### **Data that are Collected**

The detector reports the amount of electricity (in volts) required to keep its temperature constant during the analysis.

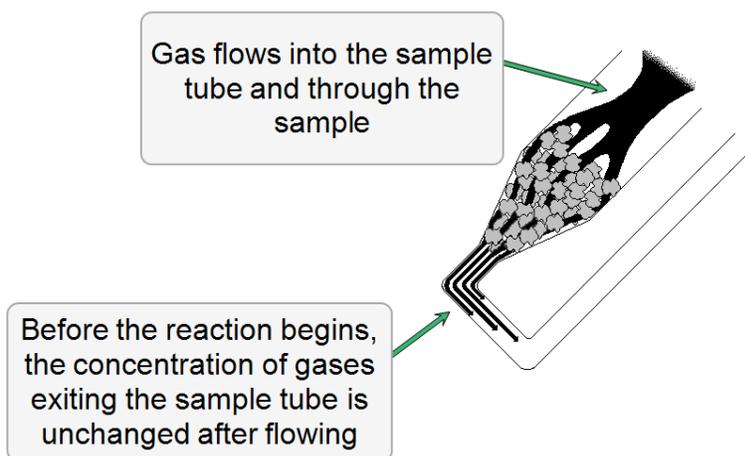
### **The Role of the Sample Temperature**

Because the sample's temperature determines how rapidly it interacts with the analysis gas (or if it reacts at all), data are collected over the range of temperatures specified.

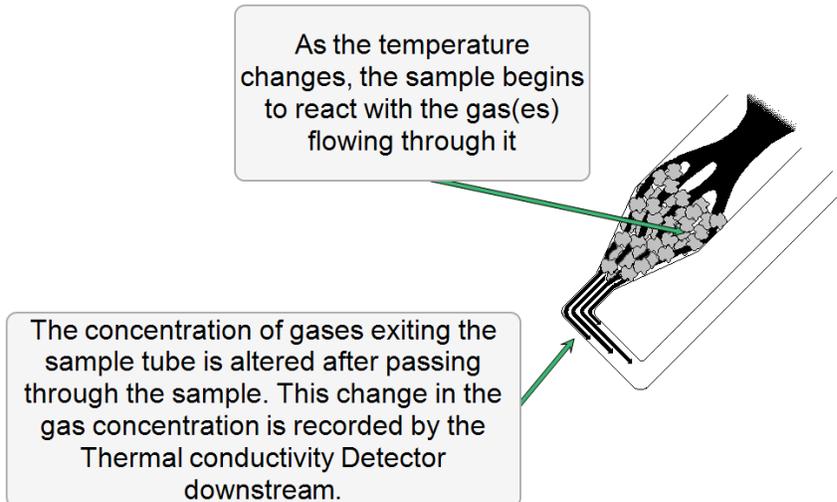
In some experiments, you may prefer to start collecting data at a very low temperature to establish a baseline where the gas is completely unaffected by the sample. In other cases, you may prefer to collect data after a reaction has begun. In still other experiments, your primary interest may be determining the temperature at which the maximum reaction occurs.

For example, consider the example of a Temperature-Programmed Reduction (TPR). During the TPR, a metal oxide is reacted with hydrogen to form a pure metal. This reaction is referred to as "reducing" the metal; for example, TPR of a catalyst containing Platinum. Argon, which has a very low thermal conductivity, is used as a carrier gas. It is blended in a fixed proportion with hydrogen, an analysis gas with a much higher thermal conductivity. Then the gas mixture flows through the analyzer, through the sample, and past the detector.

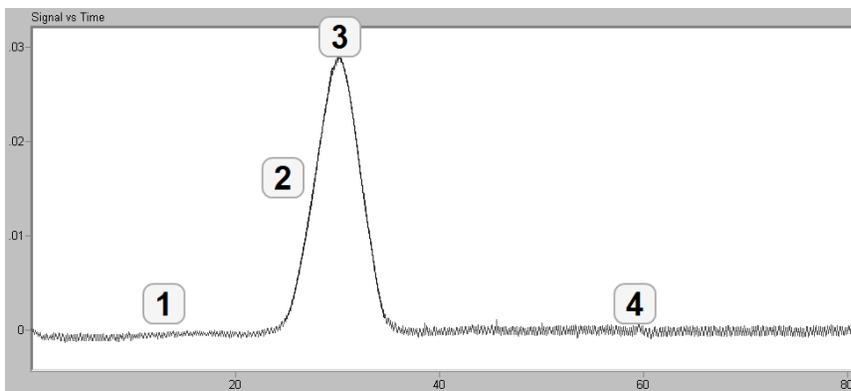
When the gas blend begins flowing over the sample, a baseline reading is established by the detector. This baseline is established at a low enough temperature that no reduction of the sample is occurring, so the baseline level recorded by the detector is that of the thermal conductivity of the two gases in their fixed proportion. In other words, the proportion of gases flowing over the detector is the same as the proportion of gases entering the analyzer, because at the low temperature, there is no interaction with the sample.



The temperature is then changed, and when a critical temperature is reached, hydrogen atoms in the gas flow react with the sample, forming  $H_2O$  molecules. The  $H_2O$  molecules are removed from the gas stream using a cold trap. As a result, the amount of hydrogen in the argon/hydrogen gas blend inside the analyzer decreases, and the proportion between the two gases shifts in the direction of argon, as does the mixture's thermal conductivity.



Since argon has a lower thermal conductivity than hydrogen, the mixture's thermal conductivity consequently decreases. The flowing gas removes heat from the filament more slowly, requiring less electricity to maintain a constant filament temperature. The instrument records the electrical demand as it changes (this is called the detector signal). The detector signal is recorded continuously over a range of temperatures. When these readings are graphed, the data form one or more peaks. Peaks can be positive or negative; negative peaks are show in this example.



## Negative Peaks Legend

Item	Description
1	Baseline readings. The gas(es) is (are) not reacting with the sample, so there is no change in the signal from reading to reading.
2	As the temperature changes, the sample begins to react with one of the gases. Therefore the gas mix is then made up of a larger proportion of the other gas. This causes a shift in the mixture's thermal conductivity. The detector measures this change by recording the change in the amount of electricity required to maintain constant filament temperature.
3	As temperature continues to increase, the interaction reaches a maximum, then begins to diminish.
4	As fewer and fewer sample atoms are available to bond with the analysis gas, there is less and less change in the mix of gases flowing into the analyzer and past the detector, so the thermal conductivity shifts back toward the baseline value.

This example illustrates the fundamental concept upon which the analyzer operates. Of course, the various types of analyses the analyzer can perform result in different types of traces. For example, a pulse chemical adsorption analysis results in a series of peaks that gradually increases in size as the sample is dosed with separate increments of gas. Initially, the gas uptake by the sample results in smaller peaks. But when all the active sites are saturated, no more gas can be taken up and the peaks become equal.

## PEAK AREA

The area beneath each peak is calculated to provide information about the volume of gas reacted during the analysis. See [3Flex Links on page iv](#) for a link to the calculations document.

## AUTOMATIC OPERATION

The analyzer application provides a simple format to specify all the analysis conditions for the experiment; create a sample file which contains sample information and a list of specific steps the analyzer will follow to perform the experiment(s). Then, the instrument automatically performs the analysis, from controlling the gas mixture and flow rate to monitoring the temperature and pressure. After analysis, use the Peak Editor to adjust the peaks to create reports that contain the data needed, without baseline noise or other undesirable effects.

Because up to 99 experiments can be specified and each experiment can contain up to 99 steps, the analyzer can perform a wide variety of preparation and analysis functions automatically.

## ***COLD TRAP***

In some cases, it is preferred to trap substances resulting from the reaction. In the previous example, H<sub>2</sub>O is produced during the analysis. If the gas flow is passed through the cold trap at an appropriate temperature, the water can be removed before the gas flows past the detector.

## ***INJECTION LOOP***

Injection loops are provided for injecting carefully measured doses of gases for analyses such as Pulse Chemisorption. The analyzer is shipped with a 0.5 cm<sup>3</sup> loop installed. A 1 ml loop is also available. If the sample file is set so that a loop is used for introducing gas into the analyzer, the instrument will automatically dose the sample as specified in the sample file.

## ***SAMPLE PREPARATION AND CALIBRATION***

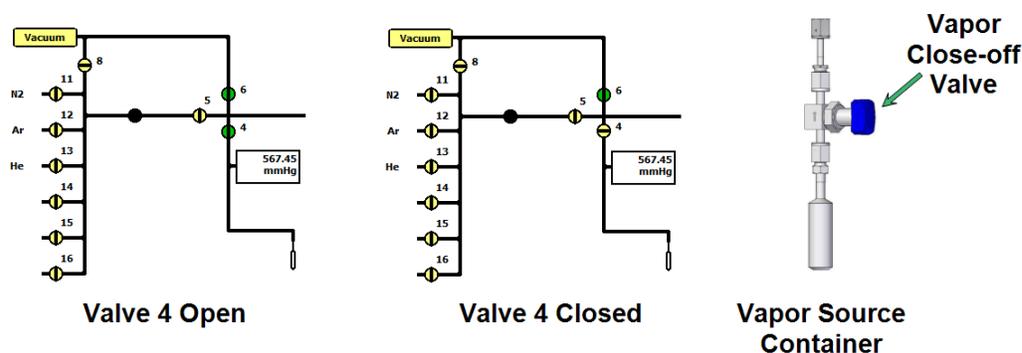
Depending on the type of experiment(s) to be performed, sample preparation and/or calibration may be required. Specific instructions are contained in the appropriate sections of this manual.

A sample is prepared for analysis by removing unwanted adsorbates from the surface of the sample. This is usually accomplished by flowing gas over the sample and may include heating the sample. The flowing gas may be inert or chemically active gases may be used to activate the surface.

Calibration routines provide the analyzer and application with the appropriate information to convert electrical signals to physically meaningful data such as volume adsorbed, loop volume, and gas concentration.

## J VAPOR PURIFICATION

Physical adsorption of gases is generally used to calculate pore size and surface area of solid materials. Using vapor adsorptives allows the sorption capacity and isosteric heat of adsorption, the energy released as molecules adsorb on the sample surface, to be calculated by using isotherms collected at different temperatures with the same vapor and adsorbent. Heats of adsorption data are very useful in research applications. In the case of competitive adsorption, the vapor with the highest heat of adsorption will adsorb first and have the strongest interaction with the surface. However, if the heat of adsorption is too high, the molecule will be so strongly adsorbed that desorption or regeneration of the material may be difficult. In order to properly collect vapor isotherms, a purified vapor must be used. A liquid-filled reservoir is used as the source of the vapor. This procedure describes a freeze-thaw method to remove dissolved gases and air within the reservoir so the vapor purity is suitable for analysis on the 3Flex. The general principles of this method could also apply to vapor purification on other gas adsorption instruments. The basic principle is to evacuate non-condensed species while the vapor reservoir is immersed in a cryogenic bath. At the pressures achieved during the purification process, nitrogen and oxygen are not condensed. The use of liquid nitrogen is limited to vapors that will not sublime at cryogenic temperatures.



1. Fill a clean vapor reservoir with liquid. Fill halfway (10 ml) if using water. Fill with approximately 20 ml for other liquids.
2. Attach vapor reservoir to the 3Flex. See [Vapor Source Container Installation on page 11-11](#) in the 3Flex Operator Manual.
3. Firmly close the blue vapor close-off valve above the liquid-filled reservoir (blue valve on the vapor container).
4. On the instrument schematic, open valve 4 to evacuate the space above the vapor close-off valve.
5. Close instrument valve 4 on the instrument schematic, then open the vapor close-off valve on the reservoir. Allow the pressure to equilibrate in the vapor container.
6. Submerge the vapor reservoir in a cryogen bath. The use of liquid nitrogen as the cryogen should be limited to vapors with no sublimation pressure at cryogenic temperatures. Wait for the pressure in the vapor container to drop as low as possible. This may only be 150 torr for the first cycle but should be near zero after two or three cycles. A lab jack is useful to hold and adjust the dewar filled with liquid nitrogen.

7. Once the vapor has condensed and has frozen, open valve 4 on the instrument schematic with the cryogen bath still in place and evacuate the vapor container (the vapor close-off valve should still be open). Pressures in the range of  $10^{-4}$ - $10^{-5}$  torr should be achievable.
8. Close valve 4 on the instrument schematic and remove the cryogen bath. Let the vapor container thaw. The vapor close-off valve should remain open during this step. To expedite the process, a warm water bath may be used temporarily to raise the temperature of the vapor container closer to the ambient temperature. However, a layer of ice formed on the reservoir could also create a barrier for heat transfer, so it is best to not introduce the water bath immediately. If pressure stabilizes near the calculated saturation pressure at room temperature, the vapor is free of impurities.
9. If the pressure does not stabilize around the calculated saturation pressure, repeat steps 3 through 8 until the pressure at the last step stabilizes near the calculated saturation pressure of the vapor. When the vapor is pure, the pressure should stabilize around the same value after each thawing cycle. Typically, three total purification cycles (two repeats) is sufficient.

## K WETTED MATERIALS



Contact Micromeritics for assistance before running a gas such as ammonia or pyridine that is incompatible with some of the system components.

### Wetted Materials

Material	Location
304 stainless steel , 403 stainless steel , Ceramic (Al <sub>2</sub> O <sub>3</sub> ), Silicon, SiO <sub>2</sub> , Si <sub>3</sub> N <sub>4</sub> , Gold, Viton, Low out gassing epoxy resin	Vacuum gauge
316 Stainless Steel, Hastelloy C-22, 17-7 PH, 430SS, Nickel, Kalrez (FFKM)	Mass Flow Controller (MFC) (for chemical adsorption only)
Aluminum	NW/KF ring in vacuum line
Aluminum alloys, stainless steels, fluoroelastomer and nitrile O-rings, hydrocarbon lubricant, felt, rare earth magnets, silicon nitride, phenolic resin, carbon-fiber reinforced epoxy resin, fire retardant polypropylene, polyamide and PVC.	Turbo pump
Borosilicate Glass	Physical adsorption sample tubes, filler rods
Buna-N	<ul style="list-style-type: none"> <li>Gas inlet manifold, valve plungers, and O-rings</li> <li>NW/KF ring in vacuum line</li> </ul>
Buna-N, FKM (Viton), stainless steel	TranSeal (optional)
Copper	Gas inlet lines
FFKM (Kalrez)	<ul style="list-style-type: none"> <li>Optional sample port O-rings</li> <li>Servo valve</li> </ul>
Gold-plated copper	Turbo vacuum pump gasket and vacuum gauge
Gold-plated nickel/iron	TCD filaments
Inconel	Transducers
Kel-F (PCTFE )	<ul style="list-style-type: none"> <li>Valve seats analysis manifold</li> <li>Valve seat exhaust valve (chemical adsorption only)</li> </ul>

## Wetted Materials (continued)

Material	Location
<b>PCTFE (Kel-f), Buna-N, FKM (Viton), Stainless steel, Borosilicate Glass</b>	Check seal (optional)
<b>PTFE, FPM (fluroelastomer), Aluminum</b>	Diaphragm roughing pump
<b>Quartz</b>	Chemical adsorption sample tubes, filler rods, wool filter discs
<b>Stainless steel</b>	<ul style="list-style-type: none"> <li>• Manifolds, valve bodies, plumbing, VCR gaskets, port transducers, filler rods.</li> <li>• Gas inlet lines(optional)</li> <li>• Ferrules in exhaust line (chemical adsorption only)</li> <li>• TCD block</li> </ul>
<b>Teflon (PTFE)</b>	Ferrules in gas inlet manifold, P <sup>0</sup> tube
<b>Viton type A (FKM)</b>	Sample port O-rings

## *L WORKSHEETS*

---

Worksheets in this section may be copied as needed.

- [Sample Data Worksheet](#)



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