

AUTOPORE[®] IV

MERCURY INTRUSION POROSIMETER



OPERATOR MANUAL

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ABOUT THIS MANUAL

Log in to your [customer portal](#) to access the following:

- Calculations
- Error Messages
- AutoPore IV Operator Manual in PDF format

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

The following icons may be found in this manual:



NOTE - Notes contain important information applicable to the topic.



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



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

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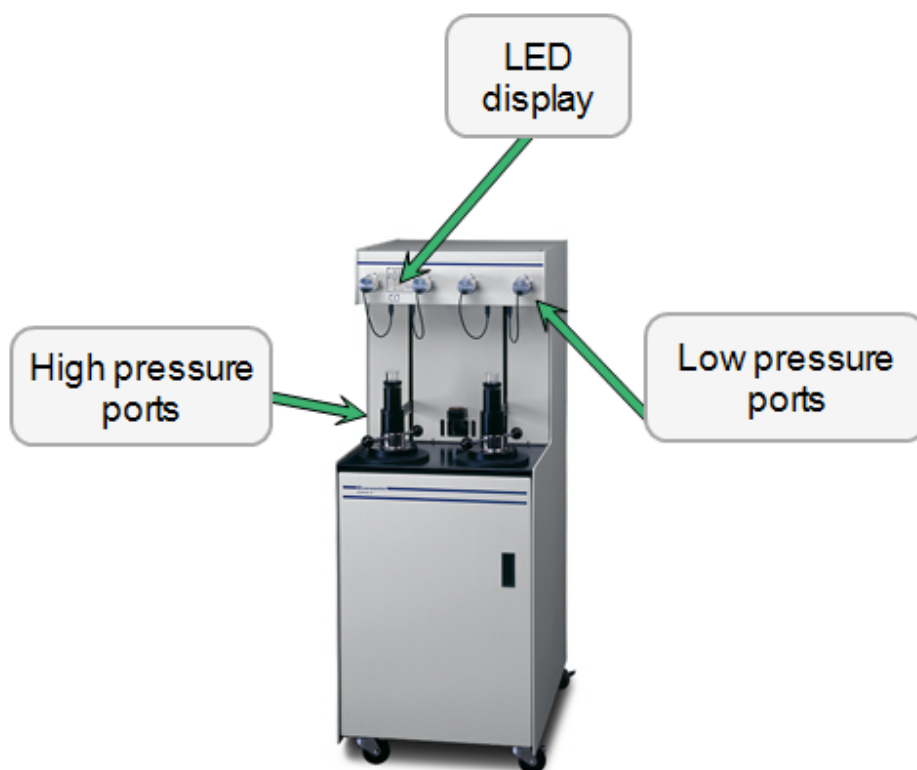
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1 ANALYZER COMPONENTS

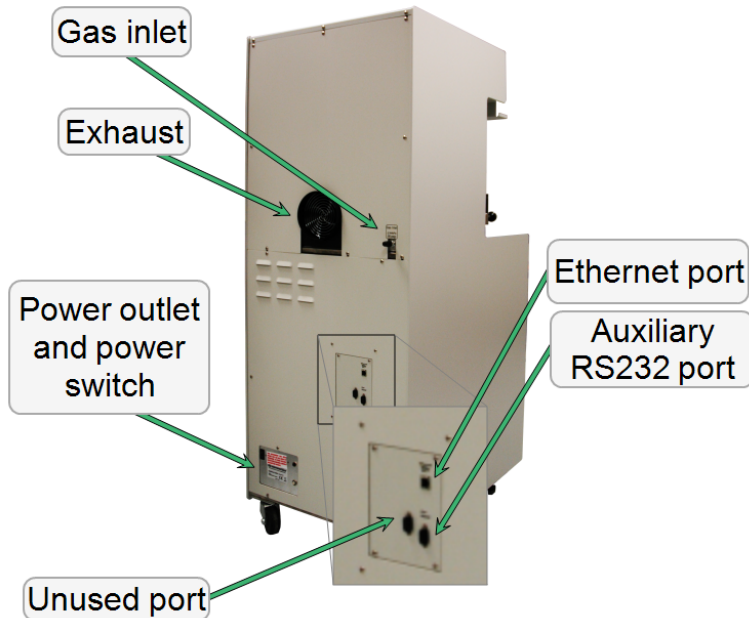
FRONT PANEL



Front Panel Components

Component	Description
High Pressure Ports [LED light]	Pressurized. Illuminates when the high pressure system is pressurized, when the computer system is not connected, or when the computer is connected but the application is not running.
Low Pressure Ports [LED light]	<p>Pressurized. Illuminates when the low pressure system is pressurized, when the computer system is not connected, or when the computer is connected but the application is not running.</p> <p>Hg Up. Illuminates when the amount of mercury in the low pressure system is sufficient to fill the penetrometers.</p> <p>Hg Drained. Illuminates when mercury has been drained from the low pressure system.</p>
Power [LED light]	Illuminates when power is supplied to the analyzer.

REAR PANEL



Rear Panel Components

Component	Description
Aux RS232 Port	Used by service personnel.
Ethernet Port	Port for an Ethernet cable allowing communication between the analyzer and the computer.
Exhaust Port	Provides ventilation for the analyzer. This exhaust can be vented to the outside using flexible tubing similar to the type used on a home clothes dryer, and a vent system which pulls air from the exhaust port.
Gas Inlet Port	Used for connecting an external gas supply to the analyzer when generating pressures in the low pressure system.
Power cord connector	For setting the power voltage and connecting the analyzer to the power supply.
Power Switch	Powers the analyzer on and off. It also provides electrical overload protection.
Reset buttons	<p>10 Amp. Resets the internal vacuum pump.</p> <p>25 Amp. Resets the internal vacuum pump in a high pressure analyzer.</p>

THE BASIC WORKFLOW

Before a sample can be analyzed, it must be:

1. Weighed and the mass recorded, and
2. A sample file must be created that describes the sample and gives the analysis conditions and other parameters for the analysis. This file also includes a pressure table, which lists the pressure points at which data are collected during the analysis.

To begin the analysis, a sample is placed in a penetrometer then the penetrometer is installed into a low pressure port. The low pressure analysis consists of two phases:

1. The first phase is the evacuation of gases from the penetrometer. The penetrometer is then filled automatically with mercury.
2. The second phase is the collection of data at pressures up to and including the last low pressure point specified in the pressure table.

When the low pressure analysis is complete, remove the penetrometer from the low pressure port and weigh it, then install it in a high pressure port. The mass of the penetrometer with sample and mercury can be entered in the sample file or in the window for starting the high pressure analysis.

Pore volume data are calculated by determining the volume of mercury remaining in the penetrometer stem. As pressure increases, mercury moves into the sample's pores, vacating the stem (this is called intrusion). Intrusion of different size pores occurs at different pressures. (The greater the pressure, the smaller the pore diameter into which the mercury can be forced.) Because mercury has a high surface tension and is nonwetting to most materials, its angle of contact and radius of curvature can be used to calculate the pore diameter into which it intrudes at a given pressure.

The volume of mercury in the penetrometer stem is measured by determining the penetrometer electrical capacitance. Capacitance is the amount of electrical charge stored per volt of electricity applied. The penetrometer capacitance varies with the length and diameter of the penetrometer stem that is filled with mercury.

When the penetrometer is initially filled with mercury, the mercury extends the entire length of the penetrometer. As increasing pressure causes the mercury to intrude into the sample's pores, the volume of mercury in the penetrometer stem decreases by an amount equal to the volume of the pores filled. This decrease in the length of the penetrometer stem that is filled with mercury causes a reduction in the penetrometer capacitance. The analysis application converts measurements of the penetrometer capacitance into data points showing the volume of mercury intruding into the sample's pores.

[Theory on page D - 1](#) provides a thorough discussion of the theory of porosimetry.

SPECIFICATIONS FOR AUTOPORE IV

Characteristic	Specification
Electrical	
Frequency	50 to 60 Hz
Power	600 VA maximum
Voltage	100/120/220/240 VAC \pm 10%
Gas	
The gas must be dry, clean, inert, and regulated to 60 psig.	
High Pressure	
Measurement	
Model 9510/9520	From atmospheric pressure to 60,000 psia (414 MPa)
Models 9500/9505	From atmospheric pressure to 33,000 psia (228 MPa)
Pore Diameter Range	
Model 9510/9520	6 to 0.003 μ m
Models 9500/9505	6 to 0.005 μ m
Resolution	
Model 9510/9520	0.3 psi (2070 Pa) from 5000 psia (34 MPa) to 60,000 psia (414 MPa), and 0.1 psi (689 Pa) from atmospheric pressure to 5000 psia (34 MPa)
Models 9500/9505	0.2 psi (1400 Pa) from 3000 psia (21 MPa) to 33,000 psia
Transducer Accuracy	\pm 0.1% of full scale (transducer manufacturer's specifications)
Intrusion	
Accuracy	\pm 1% of maximum penetrometer stem volume
Resolution	Better than 0.1 μ L
Low Pressure	
Measurement	0 to 50 psia (345 kPa)
Pore Diameter Range	3.6 μ m to >900 μ m
Resolution	0.01 psi (69 Pa)
Servo Control Precision	1% of target, min. .05 psia, no overshoot
Transducer Accuracy	\pm 1% of full scale (transducer manufacturer's specifications)

Characteristic	Specification
Penetrometers	
Capillary Stem Intrusion Volumes	0.38, 1.1, 1.7, 3.1, and 3.9 cc
Sample Size	Maximum: a cylinder 2.54 cm diameter × 2.54 cm long (1 in. diameter × 1 in. long)
Physical	
Height	143 cm (56.25 in.)
Width	54.3 cm (21.38 in.)
Depth	78 cm (30.75 in.)
Weight	250 kg (550 lb)
Computer Requirements	
<ul style="list-style-type: none"> • Operating System. Windows 7 Professional or higher operating system is recommended for the best user experience. • Desktop Installation Required. The application should not be installed on a network drive with shared access. Multiple users cannot operate the application at the same time. • 10 Base T or 100 Base T Ethernet Port. If the computer is to be connected to a network, two Ethernet ports are required. If more than one Ethernet based unit is connected to the same computer, an Ethernet switch will also be required. • Read / Write Permissions. All users of the application will need Read/Write permission to all directories and subdirectories where the application is installed. • Drives. CD-ROM drive and thumb drive. 	
<i>In keeping with a policy of ongoing product improvement, specifications are subject to change without notice.</i>	

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2 ABOUT THE SOFTWARE

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

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

SOFTWARE SETUP



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

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



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

SOFTWARE UPDATES

Log in to your [customer portal](#) to access and download the latest analyzer software version.

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

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

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

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
File	Use to manage files used by the application — such as sample files, analysis conditions files, report options files, etc.
Unit [n]	Use to perform analyses, calibrations, and other analyzer operations. <i>Unit [n]</i> displays on the menu bar for each analyzer attached to the computer.
Reports	Use to run reports and view the results.
Options	Use to edit the default method, specify system configuration, specify units, and change presentation options.
Window	Use to manage open windows and display a list of open windows. A checkmark appears to the left of the active window.
Help	Provides access to the 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
Add	Add an item to the list.
Add Log Entry	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Append	Use to insert one row at the end of a table.
Autoscale	When enabled on report parameters windows, allows the x- and y-axes to be scaled automatically. <i>Autoscale</i> means that the x- and y-ranges will be set so that all the data is shown. If <i>Autoscale</i> is not selected, the entered range is used.
Axis Range	On report parameters windows, the <i>From / To</i> fields are enabled when <i>Autoscale</i> options are not selected. Enter the starting and ending values for the x- and/or y-axes.
Bar Code	Enter bar code reader information if a bar code reader is connected to the computer's USB port. If a bar code reader is not used, this alphanumeric field can be used to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Browse	Searches for a file.
Cancel	Discards any changes or cancels the current process.
Clear	Use to clear the table entries and display only one default value.
Close	Closes the active window.
Close All	Closes all active windows. If changes were made and not yet saved, a prompt displays for each changed file providing the option to save the file.
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Copies	Select the number of copies to print. This field is only enabled when <i>Print</i> is selected.
Delete	When working with pressure tables and pressures, Delete removes the selected information.
Destination	Select the report destination.

Common Fields and Buttons Table (continued)

Field or Button	Description
Edit	When working with report parameters, highlight the item in the <i>Selected Reports</i> list box and click Edit to modify the report details.
Exit	Exits the application. If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the application without saving the changes.
Export	Exports data in a sample file as a .REP, .TXT or .XLS file. When saved to a file, the data can be imported into other applications.
File	Select the destination directory. Enter a new file name in the <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.
File name	Select a file name from the list shown or enter a file name. If the required file type is not shown, select the type of file on the left.
From / To	When working with report parameters windows, enter the <i>From</i> and <i>To</i> range for x- and/or y-axes.
Insert	Use to insert one row above the selected row in the table.
List	Creates a list of sample or other type files. The list will contain file name, date / time the file was created or last edited, file identification and file status.
Name column	A list of files in the selected directory or library.
Next	Click to move to the next window or next step.
OK	Saves and closes the active window.
Open	Opens the selected file. Alternatively, double click the file name in the <i>Name</i> column to open the file.
Prev	Click to move to the previous window.
Preview	Previews predefined reports. Click the tabs at the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled.
Print	Sends the report to the selected destination (screen, printer or file).
Remove	Remove the selected file or files from the list.
Replace	Click to select another file where the values will replace the current file's values.
Replace All	Click to select another .SMP file where the values will replace all values for the active sample file. The original file will remain unchanged.

Common Fields and Buttons Table (continued)

Field or Button	Description
Report	Click to display a window to specify report output options.
Save	Saves changes to the active window.
Save As	Saves a file in the active window under a different file name.
Start	Starts the report, test, analysis, or operation.
Start Date	Displays a calendar to select the start date for the report.
View	<p>Operation. Use to display the data from the current analysis.</p> <p>Instrument Log. Use to display recent analyses, calibrations, errors or messages.</p> <p>Instrument Schematic. Use to display a schematic of the analyzer system.</p>

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.
HP Complete	<ul style="list-style-type: none"> • A high pressure analysis has been performed using this sample file. • A low pressure analysis must be performed on a sample before a high pressure analysis can be performed; files with the status <i>HP Complete</i> have finished one low pressure analysis and at least one high pressure analysis. • Multiple high pressure analyses can be performed on a sample, so files with the status <i>HP Complete</i> may have finished more than one high pressure analysis.
LP Complete	A low pressure analysis has been performed using this sample file. (Only one low pressure analysis may be performed on each file.)
No Analysis	Sample files which have not been used to perform an analysis.

File Type, Extension, and Location Table

File Type	File Name Extension	Default Location
Analysis Conditions	.ANC	Param Directory
Material Properties	.MTP	Param Directory
Methods	.MTH	Param Directory
Penetrometer	.PEN	Param Directory
Report Options	.RPO	Param Directory
Sample File	.SMP	Data Directory

File types available when printing or exporting reports:	
Portable document format	.PDF
Report	.REP
Spreadsheet	.XLS
Unicode	.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
Alt + [Unit n]	Opens the Unit [n] menu.
Alt + F	Opens the <i>File</i> menu.
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Alt + H	Opens the <i>Help</i> menu.
Alt + I	Opens the <i>Options</i> menu.
Alt + R	Opens the <i>Reports</i> menu.
Alt + W	Opens the <i>Window</i> menu.
Shift + F9	Opens the shortcut menu of (1) selected component on analyzer schematic when manual control is enabled or (2) onscreen reports.
Ctrl + N	Opens a new sample file.
Ctrl + O	Opens the <i>File Selector</i> .
Ctrl + P	Opens the <i>File Selector</i> to start a report from a selected .SMP file.
Ctrl + S	Saves the open file.
F1	Opens the online help operator manual.
F2	Displays the <i>File Selector</i> window.
F3	When in the <i>File Selector</i> window, opens the file search box.
F4	When in the <i>File Selector</i> window, opens the address bar.

Keyboard Shortcut Table (continued)

Field or Button	Description
F6	Cascades open windows.
F7	Tiles open windows.
F8	Opens the <i>File Selector</i> to start a report from a selected .SMP file.
F9	Closes all open reports.

OPTION PRESENTATION

Options > Option Presentation

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

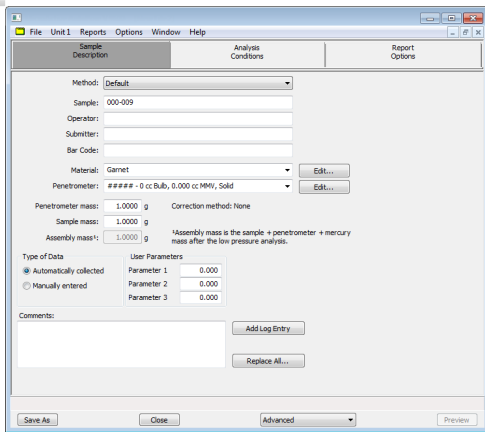
Option Presentation Display Table

Presentation Display	Description
Advanced	Displays all parts of sample information and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Restricted	Displays the sample 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 Splash Screen	Enables (or disables) the splash screen upon application startup.

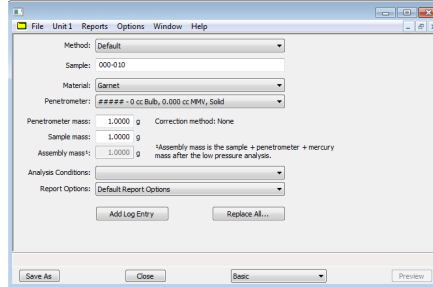


Specify or change the default option presentation by selecting **Options > Option Presentation**, or select *Basic* or *Advanced* from the drop-down list at the bottom of the window.

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



**Advanced
option presentation**



**Basic / Restricted
option presentation**



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

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 access to all files. Even though libraries do not store actual sample and parameter files, folders can be added or removed within each library.

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

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

METHODS

Options > Default Method

File > Open > [.MTH File]

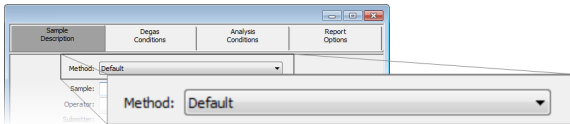


See also:

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

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.

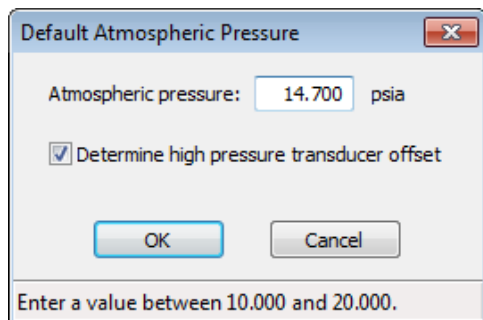


CONFIGURE THE ANALYZER

ATMOSPHERIC PRESSURE

Options > Atmospheric Pressure

For accurate analysis, the application must adjust pressure measurements to account for variations in local atmospheric pressure (barometric pressure).



Enter the current atmospheric pressure or accept the system default of 14.7 psia (0.1014 MPa). When the port is open, it is recommended to enter the value on the low pressure display. It is recommended to enter the current atmospheric pressure daily (or more frequently if atmospheric pressure rises or falls rapidly during the day).

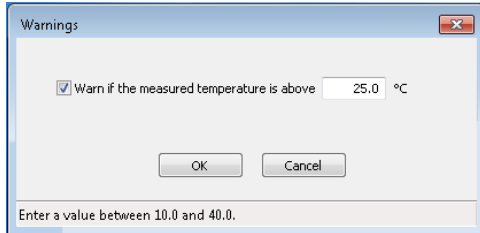
Select *Determine high pressure transducer offset* to enable the system to determine automatically the high pressure transducer offset.

If the atmospheric pressure is above the entered value and the *Determine high pressure transducer offset* option is selected, a warning will display.

MEASURED TEMPERATURE WARNING

Options > Warnings

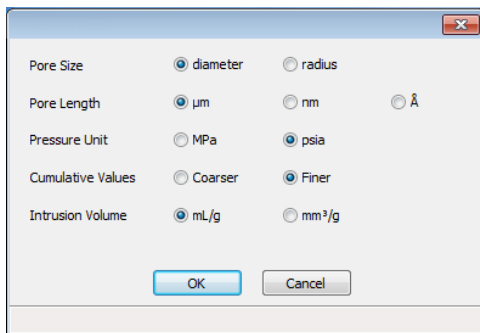
Enable this option to have the analyzer automatically display a warning if the measured temperature rises above the entered temperature.



SPECIFY UNIT SELECTIONS

Options > Units

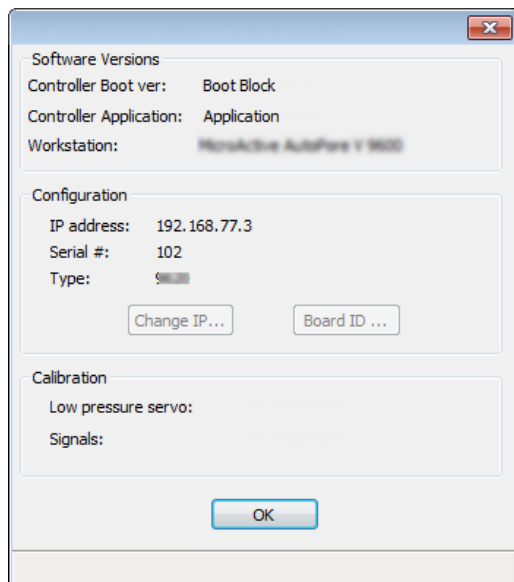
Use to specify how data should appear on the application windows and reports. This menu option is not available if using *Restricted* option presentation.



UNIT CONFIGURATION

Unit [n] > Unit Configuration

Use to display hardware / software configurations.



Unit Configuration Fields and Buttons Table

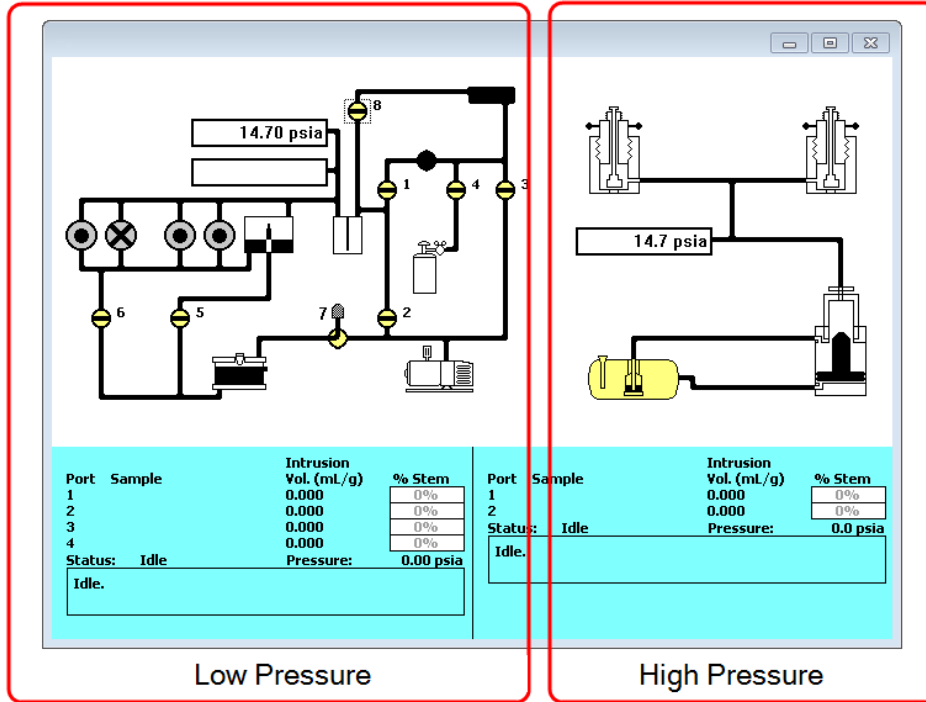
Field or Button	Description
Calibration <i>group box</i>	Displays the dates of the last high and low pressure servo calibrations and signals.
Configuration <i>group box</i>	Displays the IP address used by the analysis program, serial number, and type of analyzer. Change IP [<i>button</i>]. Displays the <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. Board ID [<i>button</i>]. Displays information from the electronic circuit boards in the analyzer. These parameters cannot be edited.
Software Versions <i>group box</i>	Displays the software versions of the controller start up code, analysis program, and type of analyzer.

INSTRUMENT STATUS

SHOW INSTRUMENT SCHEMATIC

Unit [n] > Show Instrument Schematic




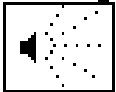
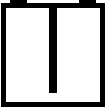
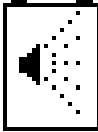


To operate the valves and elevator from the schematic, manual control must be enabled (**Unit [n] > Enable Manual Control**).



Analyzer Schematic Icon Table

Icon or Symbol	Description
	Open Valve. Green indicates an open valve.
	Closed Valve. Yellow indicates a closed valve.
	Servo Valve. Closed.
	Servo Valve. Open.

Low Pressure Schematic Icons

<p>Mercury Degasser. Displays the mercury level.</p>			
 <p>Drained</p>	 <p>Partially Filled</p>	 <p>Filled</p>	 <p>Overfilled. This alarm displays with a red background. This is an alarm state.</p>
<p>Mercury Trap. Displays the state of the mercury trap.</p>			
 <p>Empty</p>	 <p>Contains more than 6 mm of mercury. This is an alarm state.</p>		
<p>Mercury Reservoir. Displays the level of mercury in the reservoir.</p>			
 <p>If the mercury level is low, see Maintain Mercury Level on page 11 - 17.</p>		 <p>Level is OK.</p>	

High Pressure Schematic

Hydraulic Pump		
	<p>When the pump is operating, the target pressure displays below the icon. A green icon indicates the pump is ON. A yellow icon indicates the pump is OFF. To set the target pressure when the icon is yellow, either double-click the pump icon or right click the icon and select <i>Set</i>.</p>	
Intensifier. Displays the state of the intensifier limit switches.		
Midway	Top	Bottom

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:
<p>Valve options</p>	<p>Automatic. Automatically operates the servo valve during dosing or evacuation. Enter the target pressure.</p> <p>Close. Closes the selected valve.</p> <p>Disable. Select to disable the servo pump. To enable the pump, right click the servo pump icon and select <i>Set</i> to verify or set a target temperature. When the pump is enabled, the target temperature displays below the servo pump icon.</p> <p>Open. Opens the selected valve.</p> <p>Pulse. Use to quickly turn the valve on and off allowing the operation to proceed in small increments.</p> <p>Set. Use to set the servo pump target pressure.</p>

SHOW STATUS

Unit [n] > Show Status

Port	Sample	Intrusion Vol. (ml)	% Stem
1		0.000	0%
2		0.000	0%
3		0.000	0%
4		0.000	0%

Status: Idle Pressure: 0.00 psia

SHOW DASHBOARD

Unit [n] > Show Dashboard

The dashboard displays the following:

- Number of days until low pressure system service is due
- Days until high pressure system service is due
- Days until Hg filter replacement is due
- Low pressure manifold outgas rate
- Hg temperature (°C) mean $\pm 2\sigma$, min/max
- High pressure cycles completed
- Low pressure analyses completed / started
- High pressure analyses completed / started

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.

364 Days until low pressure system service is due	364 Days until high pressure system service is due	364 Days until Hg filter replacement is due	●●●●●●●● Low pressure manifold outgas rate
27.78 \pm 0.94 27.04/28.40 Hg temperature (°C) mean $\pm 2\sigma$, min/max	0 High pressure cycles completed	0/0 Low pressure analyses completed/started	0/0 High pressure analyses completed/started



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

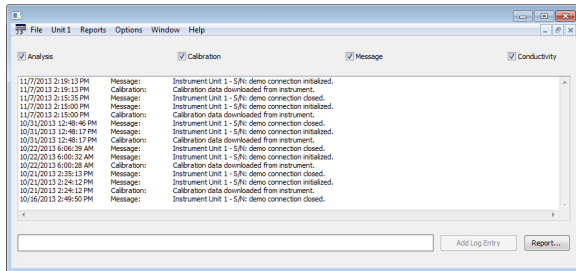
Field or Button	Description
Days until high pressure system service is due	The number of days until service is required on the high pressure system. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.
Days until Hg filter replacement is due	The number of days until the mercury filter replacement is due. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.
Days until low pressure system service is due	The number of days until service is required on the low pressure system. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.
Hg temperature	Displays the statistics of the manifold temperature reading. The mean, the value at two standard deviations, the minimum, and the maximum display.
High pressure analyses completed / started	Displays N/M where N is the number of high pressure 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.
High pressure cycles completed	Indicates the number of completed high pressure cycles. Cycles canceled or terminated by errors before the termination stage starts are not counted as completed.
Low pressure analyses completed / started	Displays N/M where N is the number of low pressure 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.
Low pressure manifold outgas rate	<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> <ul style="list-style-type: none"> • Three green LEDs are lit if outgas rate is below 30% of outgas rate limit. • At 30%, the left LED turns off. • At 60%, the center LED turns off. • At 90%, three green LED lights turn off and the center yellow LED is turned on. • At 110% and above, only the red LED is lit and attention is required.

SHOW INSTRUMENT LOG


Unit [n] > Show Instrument Log

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

- 7 days of analysis data
- 30 days of messages
- 30 days of calibration data



Instrument Log Fields and Buttons Table

Field or Button	Description
Add Log Entry	To manually enter information into the log file, enter the text into the text field to the left of the Add Log Entry button, then click Add Log Entry .
Analysis / Calibration / Message / Conductivity	Select the logs to display.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3 .

EXPORT FILES

File > Export



See also:

[Exported Data Example on page G - 1](#)

Provides the option to print the contents of one or more sample files to either the screen, a printer, or to a file. Intrusion 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

3 ABOUT SAMPLE FILES

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

A sample 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]

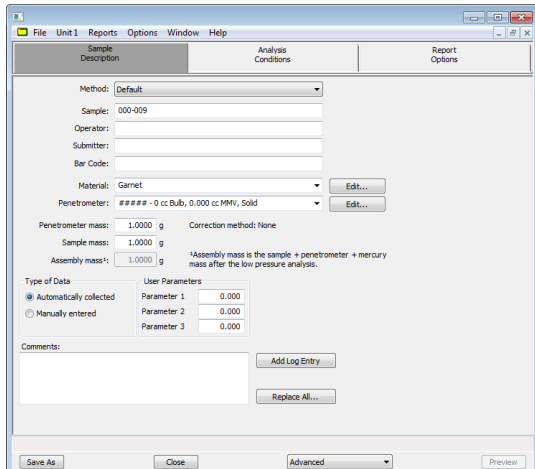
File > Open > [.SMP File]

Each analysis must be linked with a sample file before the analysis can proceed.

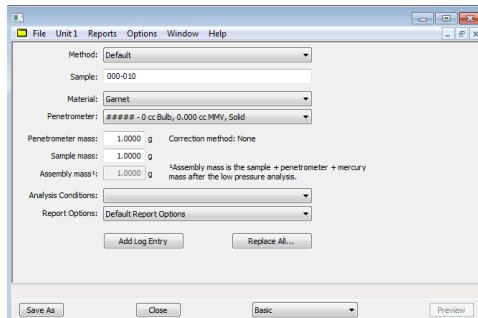
Specify or change the default display option by selecting **Options > Option Presentation** or select *Basic / Advanced* from the drop-down list at the bottom of the window.

The values for individual parameters in a file must be initially entered in *Advanced* mode and the file saved. Sample files can then be created in *Advanced*, *Basic*, or *Restricted* mode by selecting predefined *Methods* or *Analysis Conditions* and *Report Options*.

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



Advanced option presentation




Basic option presentation

Sample File Fields and Buttons Table

Field or Button	Description
Assembly mass [text box]	This field is entered after the low pressure analysis is completed. Sample + penetrometer + mercury mass after the low pressure analysis.
Bar Code	Enter bar code reader information if a bar code reader is connected to the computer's USB port. If a bar code reader is not used, this alphanumeric field can be used to enter additional information about the sample, such as a sample lot number, sample ID, etc. To rename the field label, go to Options > Default Method and deselect the Omit option.
Comments [text box]	Enter comments about the sample or analysis. Comments display in the report header.
Material [drop-down box]	Select the material to be analyzed from the drop-down list. See Material Properties on page 4 - 7 .
Method [drop-down box]	Select a method from the drop-down list. See Methods on page 2 - 12 .
Operator [text box]	Enter operator identification information. This field label may have been renamed or may not display if modified in Options > Default Methods .
Penetrometer [drop-down box]	Select a penetrometer file from the drop-down list, or click Edit to modify or create a new Penetrometer file. See Penetrometer Properties on page 4 - 9 .
Sample [text box]	Enter a sample description.
Submitter [text box]	Enter submitter identification information. This text box may have been renamed or may not display if modified in Options > Default Methods .

Sample File Fields and Buttons Table (continued)

Field or Button	Description
Type of Data [<i>group box</i>]	<p>Automatically collected. Select if the type of data will be automatically collected by the system while an analysis is running.</p> <p>Manually entered. Use to enter data manually that was collected from another source. If <i>Manually entered</i> is selected, the Intrusion Report becomes available in the <i>Basic/Advanced</i> drop-down list for pasting or importing data into the file.</p> <p>See Manually Enter Data on page 3 - 8.</p>
User Parameters [<i>group box</i>]	<p>These fields are primarily used for the SPC (Statistical Process Control) reporting to specify sample characteristics or its manufacturing process but may be used for other data by entering specific analysis conditions or sample criteria.</p> <p>The entered parameters display on the <i>Summary Report</i>. Some fields may not display (or may have a different field label) if modified in the method from which the sample file was created, either through Options > Default Method or File > Open > Method.</p>
<div style="display: flex; align-items: center;">  <p>For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.</p> </div>	

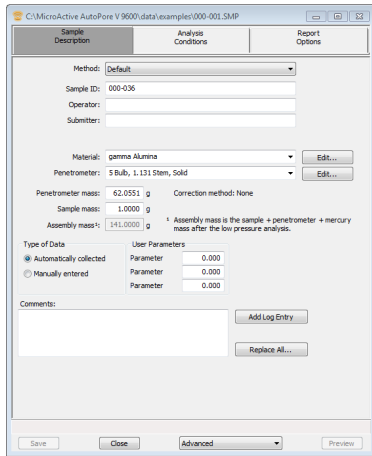
OPEN A SAMPLE FILE

File > Open > [.SMP File]

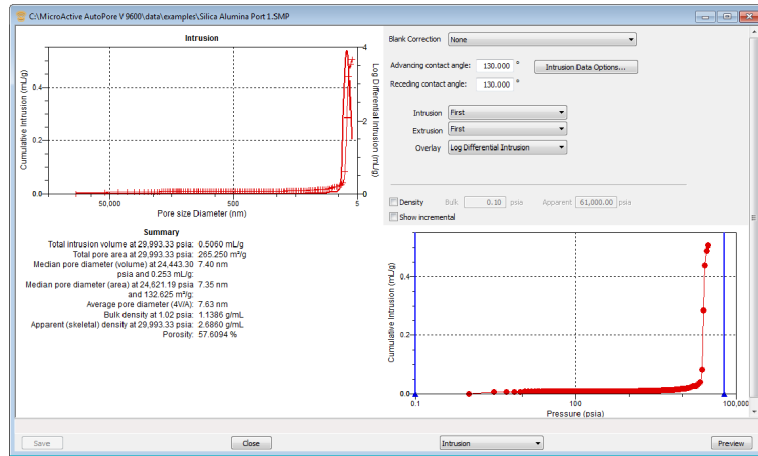


When working with an existing sample file, consider making a copy of the sample file to maintain the original configuration options.

File Type	File Status	Displays
Mercury Porosimetry	Analyzing Entered No analysis	Tabbed file editor
	HP Complete LP Complete	Report window



Example of tabbed file editor



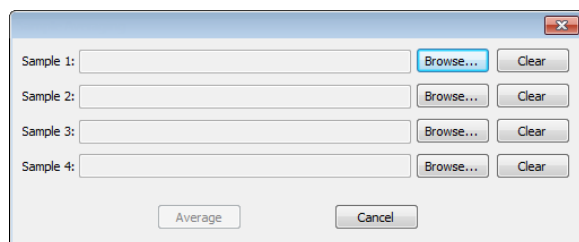
Example of report window

SAMPLE AVERAGING

File > Average

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

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



BASELINE ERROR CORRECTION METHODS

The penetrometer parts, the mercury, and the sample are all affected differently by the pressures exerted on them during analysis. Each material compresses to a different degree and at a different rate. Also, the increasing pressure within the high pressure port can cause the temperature of one or more of these materials to rise.

As a result, analysis data may show intrusion where none actually exists. For example, if the sample compresses sharply at a given pressure (compared to the compression of the mercury at that pressure), mercury moves from the penetrometer stem into the sample bulb to fill the space vacated by the shrinking sample. This reduction in the amount of mercury in the stem is interpreted by the software as intrusion.

These baseline errors can be reduced or eliminated through one of two correction methods:

- formula correction, and
- blank correction

The correction method is selected in the *Penetrometer Properties* file and displays in the *Sample* file.

CREATE BLANK CORRECTION FILE

File > Open > [enter new .SMP file name]

A sample file must be created even if there is no sample or if the sample is non-porous.

A blank correction file contains analysis data using one of the following:

- Mercury only (a blank run)
- A nonporous sample of the same mass and material as the samples to be analyzed



To be used as a correction file, the blank run should use the same analysis conditions and penetrometer properties as the sample analysis. Therefore, a separate blank correction file should be created for each type of sample and set of analysis conditions to be used.

No matter which type of blank correction run is used, the procedure is the same (follow the instructions for creating a sample file (even if there's no sample), then run the analysis. (When the blank file's sample file is created, select *None* for correction method.)

1. Create the sample file. Enter zero for the sample mass.
2. Perform the low and high pressure analysis. See [Perform a Low Pressure Analysis on page 5 - 11](#) and [Perform a High Pressure Analysis on page 5 - 16](#).



An alternative method for creating the blank correction file is to perform a *High Pressure Differential* analysis which analyzes the blank and sample simultaneously. See [Perform a High Pressure Analysis on page 5 - 16](#).

FORMULA CORRECTION

A formula for correction is available for each type of penetrometer. The formula is a part of the application software, therefore when the *Formula* option in the *Penetrometer Properties* file is selected, the formula is applied.

The recommended method of correction is by blank correction file. The formula is provided for use when comparison with historical data is required.

COMBINE A PENETROMETER CALIBRATION AND BLANK CORRECTION FILE

1. After the low pressure run, do not empty the penetrometer. Weigh it and record the assembly mass. Use a syringe to withdraw a small amount of mercury from the tip of the capillary so that the mercury column recedes approximately 1/8 in. to 1/4 in. into the stem. This enables measurement of any rise or fall of mercury in the stem during high pressure analysis and prevents spilling of mercury into the high pressure chamber. Then install the penetrometer in a high pressure port.
2. Create blank correction files by completing the high pressure run. See [Create Blank Correction File on the previous page](#).
3. Create a new *Penetrometer Properties* file for this penetrometer. See [Penetrometer Properties on page 4 - 9](#). From the file selection list, choose the default file for this type of penetrometer. Change the identifier to include the penetrometer serial number. Enter the calibrated volume and save the file. The *Penetrometer Properties* file now includes the calibrated penetrometer volume. A blank correction file has also been created for this penetrometer.

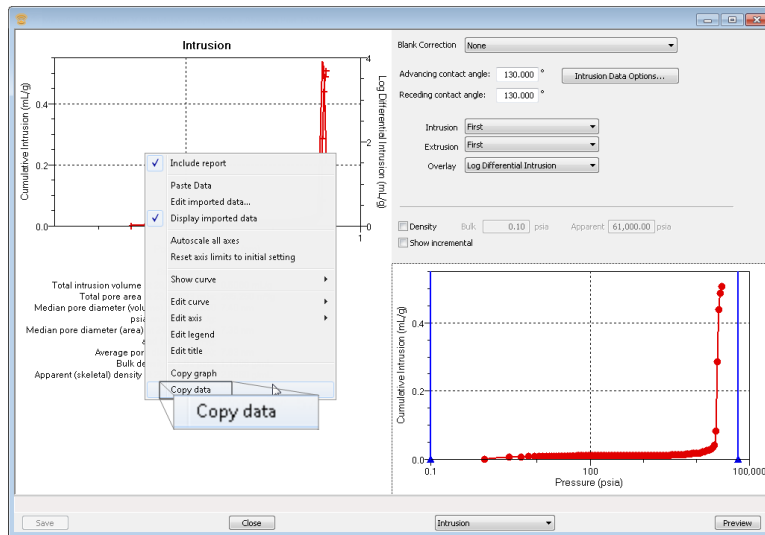
MANUALLY ENTER DATA

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:

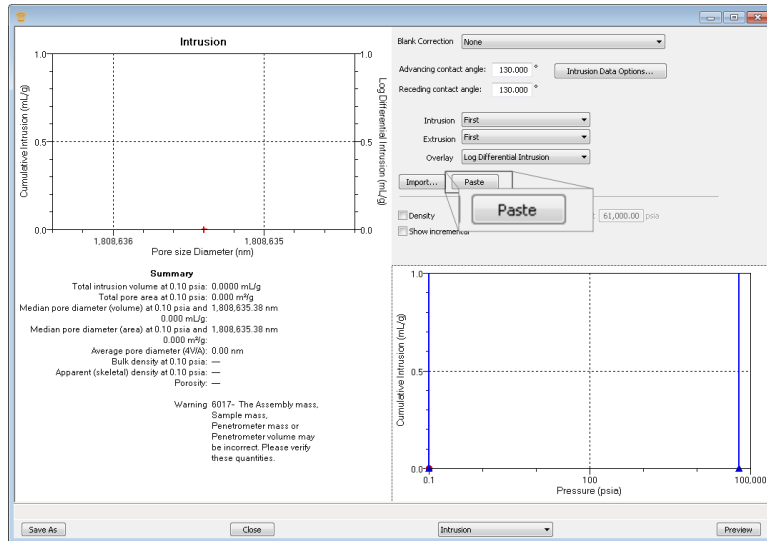
- Copy and paste onto the graph area of the interactive window
- Import data into the interactive window

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 *Intrusion*.



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:

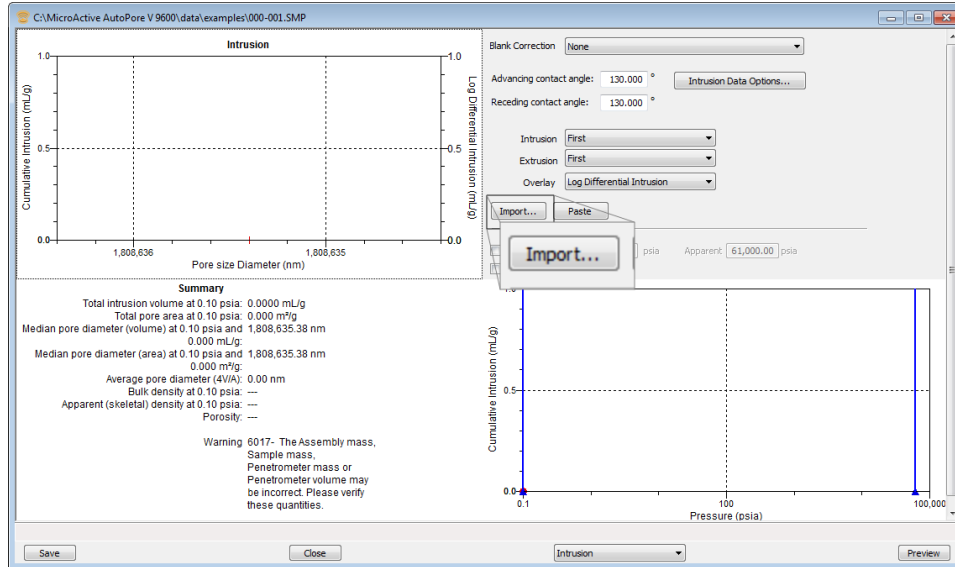
- Pressure (psi)
- Pressure (MPa)
- Pressure (kPa)
- Pressure (Pa)
- Pressure (bar)
- Pressure (mbar)
- Pressure (Atm)
- Intrusion (cm³)
- Intrusion (cm3)
- Intrusion (ml)
- Intrusion (mm³)
- Intrusion (mm3)

Sample Mercury Porosimetry ASCII Text File

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

To import the ASCII text file

1. 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 *Intrusion*..



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.

Blank Page

4 ABOUT PARAMETER FILES

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

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

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

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

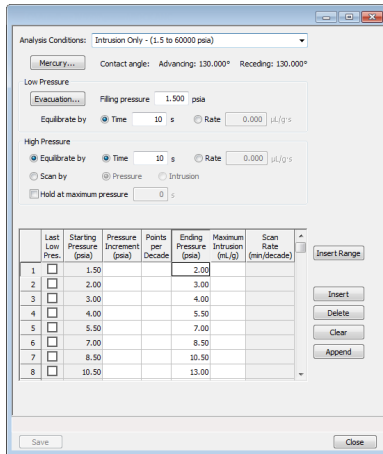
File Type	File Extension
Analysis Conditions	.ANC
Material Properties	.MTP
Method	.MTH
Penetrometer Properties	.PEN
Report Options	.RPO

ANALYSIS CONDITIONS

File > Open > [.ANC File]

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

Analysis conditions specify the parameters used to guide an analysis.





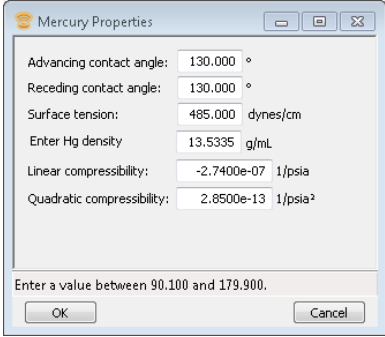
Analysis Conditions Fields and Buttons Table

Button	Use to Specify...
<p>High Pressure [group box]</p>	<p>Equilibrate by. Select the option for equilibration based on elapsed <i>Time</i> (in seconds) or decrease in <i>Rate</i> of intrusion (or extrusion) in mL/g per second.</p> <p>Scan by:</p> <ul style="list-style-type: none"> • Pressure. The analyzer goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment ends at the specified pressure. The pressure is programmed to increase or decrease at a rate to give a constant time per decade of pressure. Along the way, the analyzer takes intrusion points at the specified number of points per decade, ending at the specified ending pressure. Also, any points in the pressure table, as well as points separated by the maximum intrusion volume, are collected. • Intrusion. The analyzer goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment ends at the specified pressure. The pressure rate is the maximum achievable safe rate (up to 0.5 min/decade) and is pro-

Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	<p>grammed to increase or decrease at a rate to give a constant intrusion / extrusion rate. The analyzer takes intrusion points at the specified number of points per decade, ending at the specified ending pressure. Also collected are points in the pressure table, as well as points separated by the maximum intrusion volume.</p> <p>If both high pressure ports are in use, they may have different intrusion rates. In this case, the left port (port 1) determines when data are collected. The right port collects data at the same times as the left. This allows a differential analysis to be performed in this mode.</p> <ul style="list-style-type: none"> • Hold at maximum pressure. Enter additional amount of time to remain at the maximum pressure in a pressure table intrusion segment before beginning extrusion.
<p>Low Pressure [group box]</p>	<p>Evacuation . Select if the sample is a <i>Formed solid</i>, <i>Powder</i>, or <i>Other</i>. If <i>Other</i> is selected, the remaining fields are enabled. This window is also available from Unit [n] > Evacuate Low Pressure.</p> <div data-bbox="560 961 943 1524" data-label="Image"> </div> <ul style="list-style-type: none"> • Initially evacuate at. Enter the initial maximum evacuation rate. • Switch to medium at. Enter the method and pressure the system must reach before medium evacuation begins. <ul style="list-style-type: none"> ○ Use pressure transducer ○ User vacuum gauge

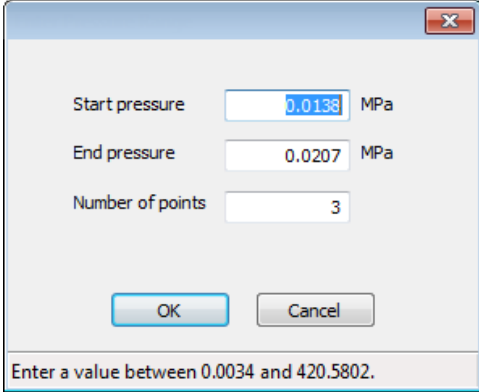
Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	<ul style="list-style-type: none"> ○ Skip medium evacuation ● Switch to fast at. Enter the pressure the system must reach before fast evacuation begins. ● Evacuation target. Enter the evacuation pressure. ● Continue evacuating for. Enter the evacuation duration. <p>Filling pressure. The penetrometer is filled with mercury at this pressure. It is recommended to set the filling pressure slightly lower than the first low pressure point on the pressure table.</p> <hr/> <div style="display: flex; align-items: flex-start;">  <p>A filling pressure of at least 0.5 psia is recommended. Because mercury generates pressure and because fill pressures less than 0.5 psia can fail to fill the corner radii and gaps between the glass and sample in the penetrometer, using a lower pressures may reduce the accuracy of data.</p> </div> <hr/> <div style="display: flex; align-items: flex-start;">  <p>If the filling pressure is higher than any point in the table, an error message occurs. Delete the pressures lower than the filling pressure or change the filling pressure.</p> </div> <hr/> <p>Equilibrate by. Select the option for equilibration based on elapsed <i>Time</i> (in seconds) or decrease in <i>Rate</i> of intrusion (or extrusion) in mL/g per second.</p>
<p>Mercury [button]</p>	<p>Enter the mercury properties. Mercury properties may change with variations in temperature.</p> <div style="border: 1px solid gray; padding: 5px; margin: 10px 0;">  </div> <p>Advancing contact angle. Enter the advancing (intrusion) contact angle.</p>

Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	<p>Receding contact angle. Enter the receding (extrusion) contact angle.</p> <p>Surface tension. Enter the surface tension of mercury.</p> <p>Enter Hg Density. Select to manually enter a density.</p> <p>Linear compressibility. Enter the linear compressibility coefficient.</p> <p>Quadratic compressibility. Enter the quadratic compressibility coefficient.</p>
Table Options	<p>Last Low Pressure. Select the row to indicate the last low pressure.</p> <p>Starting Pressure. This column is not editable. The data are taken from the <i>Ending Pressure</i> of the preceding row and the <i>Filling Pressure</i> for the first row.</p> <p>Pressure Increment. Enter the pressure increment for this segment if a sequence of linearly spaced pressures is preferred. Either the <i>Pressure Increment</i> or <i>Points Per Decade</i> can be specified. If one is entered, the other is automatically set to zero and displayed as blank. By default, both columns are blank (zero) when a new row is inserted or appended. If both columns are blank, only the <i>Ending Pressure</i> will be used.</p> <p>Points per Decade. Enter the number of points per decade for this segment if a sequence of logarithmically spaced pressures is preferred. Either the <i>Pressure Increment</i> or <i>Points Per Decade</i> (but not both) can be specified. If one is entered, the other is automatically set to zero and displayed as blank. By default, both columns are blank (zero) when a new row is inserted or appended.</p> <p>Ending Pressure. Enter the ending pressure for this segment.</p> <p>Maximum Intrusion. The analyzer automatically takes additional readings between points on the pressure table when this volume of additional intrusion is detected. Enter the intrusion volume per gram of sample that must be reached in order for additional data pair readings to be recorded. Use 0 to prevent readings between pressure points. See Use of the Maximum Intrusion Volume Option on page E - 1. The <i>Maximum Intrusion</i> entry is set to the same value as the preceding row by default when a new row is inserted or appended.</p>

Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	<p>Scan Rate. If scanning by pressure, enter the minutes per decade for this segment. If scanning by intrusion, enter the intrusion rate for this segment. The <i>Scan Rate</i> column is set to blank (zero) and disabled for all rows up to and including the <i>Last Low Pressure</i>. If <i>Scan By Pressure</i> or <i>Intrusion</i> is selected, the column title will display the appropriate units (min/decade) or (mL/g-sec) and each high pressure row must contain a value. The value for the previous row is set by default when a new row is inserted or appended, or the default value (5 min/decade or 0.001 mL/g-sec) for the first high pressure row.</p> <p>Insert Range. Click to display the <i>Enter Pressure Range</i> window for entering parameters for the system to autofill the table with starting pressure, ending pressure, and the number of points to insert within the specified range.</p> <div style="border: 1px solid gray; padding: 5px; margin: 10px 0;">  </div>

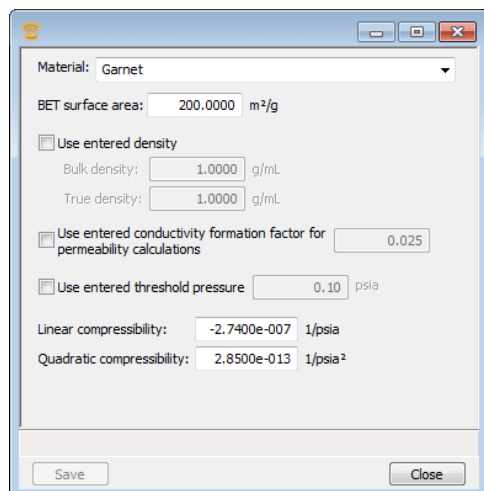


For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 3](#).

MATERIAL PROPERTIES

File > Open > [.MTP File]


Material properties specify the properties of the material to be used in an analysis. This information is only required if it is to be used in a report. See [Report Options on page 4 - 10](#).



Material Properties Fields and Buttons Table

Field or Button	Description
BET Surface Area [text box]	Enter the BET surface area.
Linear compressibility [text box]	Enter the linear compressibility coefficient.
Quadratic compressibility [text box]	Enter the quadratic compressibility coefficient.
Use entered conductivity formation factor... [check box]	Select if using the entered conductivity formation factor for permeability calculations and enter the factor in the text box.
Use entered density [check box]	Select if using the entered density and enter the bulk and /or true density in the appropriate text boxes. The entered values are used in the intrusion summary results, Mayer-Stowe report, and the pore structure report. The Mayer-Stowe particle size calculation has meaning mostly in the range of interstitial filling. For larger pressures after which the interstitial space is filled, the particle size calculation has less meaning.

Material Properties Fields and Buttons Table (continued)


Field or Button	Description
Use entered threshold pressure [<i>check box</i>]	Select if using the entered threshold pressure and enter the threshold pressure in the text box.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.

PENETROMETER PROPERTIES

File > Open > [.PEN File]

Penetrometer properties specify the properties of the penetrometer to be used in an analysis.

Penetrometer Properties Fields and Buttons Table

Field or Button	Description
Maximum head pressure [<i>text box</i>]	Enter the maximum head pressure. See Select a Penetrometer on page 5 - 1 .
Constant [<i>text box</i>]	Enter the penetrometer constant provided with the penetrometer. Verify the field contents if the <i>Replace</i> option has been used.
Correction Method [<i>selection</i>]	<p>Blank. If using the blank correction method, click Browse to select a sample file. See Create Blank Correction File on page 3 - 6.</p> <p>Formula. See Formula Correction on page 3 - 7.</p> <p>None.</p>
Mass [<i>text box</i>]	Mass of the empty, assembled penetrometer (excluding the spacer).
Penetrometer [<i>drop-down box</i>]	Enter identifying information for this file.
Stem volume [<i>text box</i>]	Enter the penetrometer stem volume provided with the penetrometer.
Volume [<i>text box</i>]	Enter the volume of the penetrometer. This is required to calculate density or when using the blank correction formula.
 <p>For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.</p>	

REPORT OPTIONS

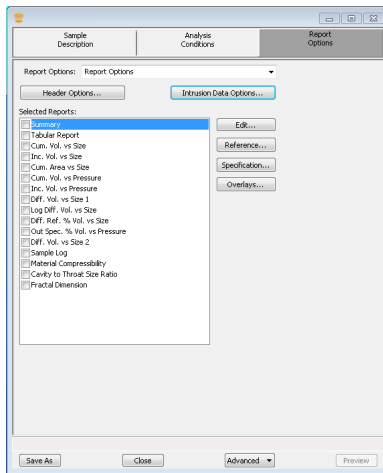
File > Open > [.RPO File]

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

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

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

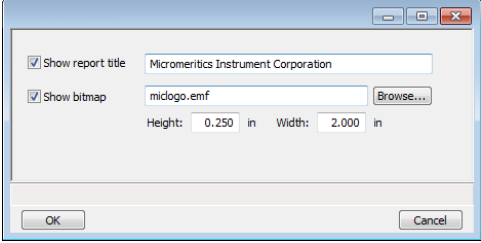
Report Options files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis. See [Overlay Multiple Mercury Porosimetry Graph Options on page 6 - 23](#).



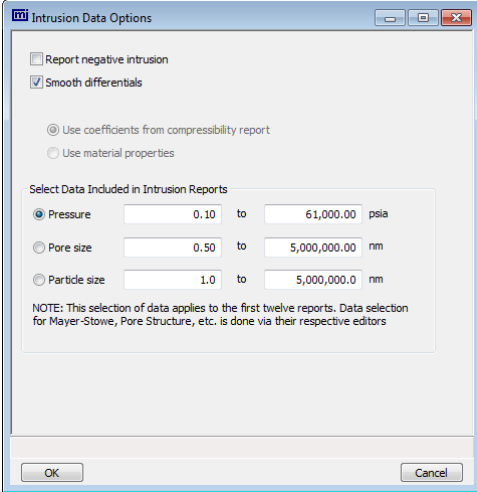
Report Options Fields and Buttons Table

Field or Button	Description
Edit [button]	<p>Edit graph options.</p> <p>Plot Points. Select to plot points on the graph.</p> <p>Plot Curve. Select to plot curves on the graph.</p> <p>Show Histogram. Select to show the graph as a histogram. When selected, the <i>Plot Points</i> and <i>Plot Curve</i> selections are disabled.</p>

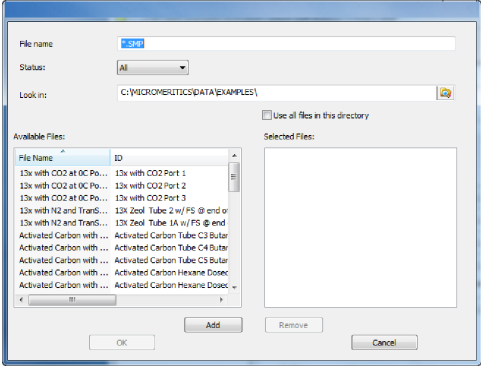
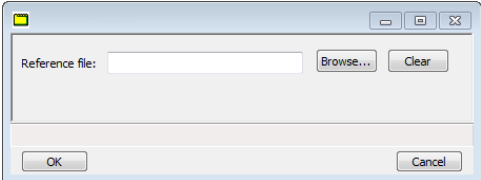
Report Options Fields and Buttons Table (continued)

Field or Button	Description
Header Options [<i>button</i>]	 <p> Show report title. Enter a report title to appear on the report header. </p> <p> Show bitmap. Displays the selected graphic on the report header. </p> <p> Height / Width. Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report. </p>

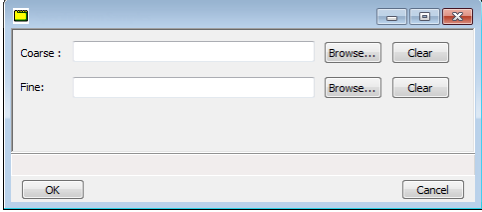
Report Options Fields and Buttons Table (continued)

Field or Button	Description
Intrusion Data Options [button]	<p>This button is also located on Intrusion reports.</p>  <p>Report negative intrusion. Select to report small incorrect polarities (negative intrusions or positive extrusions) which may indicate the presence of noise, improper blank correction, or analyzer malfunction.</p> <p>Select Data Included in Intrusion Reports. Enter range of pressure. This range applies to the first twelve reports on the options list. When these selections are changed in the intrusion report, the lower right graph x-axis and the intrusion data in the graph will change accordingly. The two blue range bars can then be used to select the required range of data.</p> <ul style="list-style-type: none"> • Pressure. The default behavior of the application is to select data using pressure as this is the quantity which is varied during mercury porosimetry analysis. The maximal range of data (all data) is selected by default. It is advisable to use this default setting unless there is a specific goal is required. • Pore size. Uses the Washburn equation to translate the user entered pore size range to a corresponding pressure range used for data selection. • Particle size. This option is less commonly used. In the <i>Material Properties</i> window, select the checkbox <i>Use entered density</i> and complete the <i>Bulk density</i> and effective <i>Particle density</i> fields. See Material Properties on page 4 - 7. The effective <i>Particle density</i> and <i>Bulk density</i> quantities are necessary to compute the interstitial porosity of the powder used in the Mayer-Stowe calculation.

Report Options Fields and Buttons Table (continued)

Field or Button	Description
	<p>Smooth differentials. Select to apply smoothing to any differentials reported in tables or graphs.</p> <p>Use coefficients from compressibility report. Select to have the application use the coefficients from the <i>Material Compressibility</i> report rather than from the <i>Material Properties</i>.</p> <p>Use material properties. Select to have the application use the parameters from <i>Material Properties</i> rather than from the <i>Material Compressibility</i> report.</p>
Overlays [button]	<p>Select the files to overlay.</p> 
Reference [button]	<p>Select a sample file to compare analysis results with the current sample.</p> 
Selected Reports [checkbox]	<p>Select the report names to include in the report.</p>

Report Options Fields and Buttons Table (continued)

Field or Button	Description
<p>Specification [<i>button</i>]</p>	<p>Selects the sample files to be used for the boundaries of the coarse and fine specifications. This helps in determining if the results of the current sample are within the specified boundaries.</p> 



For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 3](#).

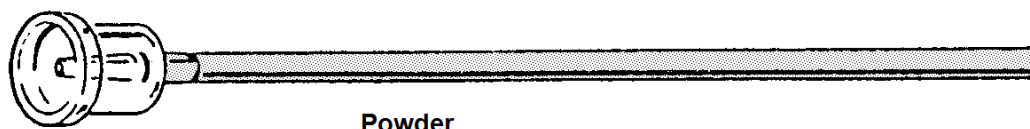
5 PERFORM AN ANALYSIS

A low pressure analysis must be performed on a sample before a high pressure analysis can be performed.

SELECT A PENETROMETER

Selecting the most appropriate penetrometer with which to test a particular material depends on sample form or shape, sample porosity, and the quantity of sample.

Penetrometers are available with three sample volumes, with five intrusion capacities, and in configurations for either solid pieces or powders. Refer to the *Penetrometer Selection Guide* in this section.



Ensure the sample nearly matches the size of the sample bulb and that the capillary volume is large enough to satisfy intrusion.

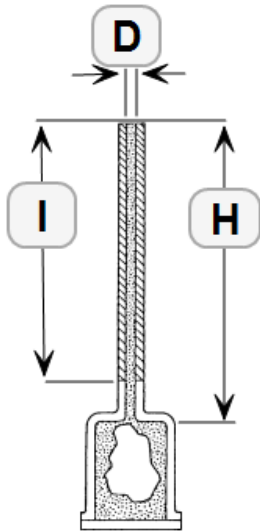
Penetrometer Selection Guide

Bulb Volume (cc)	Sample Type	Maximum Measurable Volume (cc)	Total Stem Volume (cc)	Maximum Head Pressure		Physical Dimensions			Part Number
				(psia)	(kPa)	I (mm)	H (mm)	D (mm)	
3	Solid	0.387	0.412	4.68	32.3	227	242	1.473	950-61713-00
3	Solid	1.116	1.190	4.68	32.3	227	242	2.502	950-61715-00
3	Powder	0.387	0.412	4.68	32.3	227	242	1.473	950-61714-00
3	Powder	1.116	1.190	4.68	32.3	227	242	2.502	950-61716-00
5	Solid	0.366	0.392	4.45	30.7	215	230	1.473	950-61707-00
5	Solid	1.057	1.131	4.45	30.7	215	230	2.502	950-61709-00
5	Solid	1.716	1.836	4.45	30.7	215	230	3.188	950-61711-00

Penetrometer Selection Guide (continued)

Bulb Volume (cc)	Sample Type	Maximum Measurable Volume (cc)	Total Stem Volume (cc)	Maximum Head Pressure		Physical Dimensions			Part Number
				(psia)	(kPa)	I (mm)	H (mm)	D (mm)	
5	Powder	0.366	0.392	4.45	30.7	215	230	1.473	950-61708-00
5	Powder	1.057	1.131	4.45	30.7	215	230	2.502	950-61710-00
5	Powder	1.716	1.836	4.45	30.7	215	230	3.188	950-61712-00
15	Solid	0.366	0.392	4.45	30.7	215	230	1.473	950-61701-00
15	Solid	1.057	1.131	4.45	30.7	215	230	2.502	950-61703-00
15	Solid	1.716	1.836	4.45	30.7	215	230	3.188	950-61705-00
15*	Solid	3.007	(3.263)	4.45	30.7	215	230	4.813	950-61724-00
15*	Solid	3.857	(4.185)	4.45	30.7	215	230	4.813	950-61725-00
15	Powder	0.366	0.392	4.45	30.7	215	230	1.473	950-61702-00
15	Powder	1.057	1.131	4.45	30.7	215	230	2.502	950-61704-00
15	Powder	1.716	1.836	4.45	30.7	215	230	3.188	950-61706-00

* The first 3 mm of stem on these penetrometers have an inside diameter (D) of 1.5 mm. In computing maximum measurable (intrusion) volume, the value of I should be reduced by 3 mm.



- Maximum measurable volume = $[(3.14)(D^2)(I)/4] \times [0.001 \text{ cm}^3/\text{mm}^3]$
- Total Stem (Capillary) volume = $[(3.14)(D^2)(H)/4] \times [0.001 \text{ cm}^3/\text{mm}^3]$
- Maximum Head Pressure (psia) = $[H] \times [0.01934 \text{ psia}/\text{mmHg}]$
- Maximum Head Pressure (MPa) = $[H] \times [0.000133 \text{ MPa}/\text{mmHg}]$

A powder penetrometer should be used when the sample consists of small grains or particles. Chunks of material or formed objects (maximum size is 25 mm OD × 25 mm long) should only be installed in a “solid” penetrometer.

Best results, generally, are obtained when the bulb of the selected penetrometer is nearly filled by the minimum amount of sample that is representative. Next, the estimated pore volume of the sample should not exceed 90% nor be less than 25% of the total stem volume (see column 4 of the Penetrometer Selection Guide). Once materials of similar characteristics have been tested, it will usually be possible to select the optimum penetrometer almost without fail.

As an example, suppose the sample consists of a single sintered pellet of nickel (density 8.9 g/cc) weighing 29 g and having an estimated pore volume of 20% of the true sample volume to analyze. The following characteristics are calculated:

$$\text{Volume of sample} = \text{mass/density} = (29\text{g})/(8.9\text{g/cc}) = 3.26 \text{ cc}$$

$$\begin{aligned} \text{Approximate pore volume} &= \text{fractional pore volume} \times \text{sample volume} = \\ 0.20 (3.26 \text{ cc}) &= 0.652 \text{ cc} \end{aligned}$$

$$\begin{aligned} \text{Approximate total volume} &= \text{volume of pores} + \text{volume of sample} \\ (3.26 + 0.652) \text{ cc} &= 3.91 \text{ cc} \end{aligned}$$

Hence, the penetrometer listed sixth in the Penetrometer Selection Guide as solid, 5 cc sample volume, 1.131 cc total stem volume would be the appropriate choice unless the pellet shape dictates use of a larger one. The percent of maximum measurable intrusion volume required by this sample is $(0.652 \text{ cc}/1.131 \text{ cc}) \times 100\% = 58\%$, which falls below the suggested 90% maximum.

The penetrometer for powdered or granular materials is chosen similarly, but remember that the spaces among the material grains are likely to constitute a void of about 40%. As another example, assume that 15 g of a granular material (density 3.5 g/cc) had been determined the minimum quantity for representation. Assume the powder has low porosity: 3%.

$$\text{Volume of sample} = \text{mass/density} = (15 \text{ g})/(3.5 \text{ g/cc}) = 4.29 \text{ cc}$$

$$\begin{aligned} \text{Approximate pore volume of material} &= \text{fractional porosity} \times \text{sample volume} = \\ 0.03 (4.29 \text{ cc}) &= 0.13 \text{ cc} \end{aligned}$$

$$\text{Approximate volume of interstice} = (4.29 \text{ cc} + 0.13 \text{ cc}) (40/60) = 2.95 \text{ cc}$$

$$\text{Total volume of powdered sample} = 4.29 \text{ cc} + 0.13 \text{ cc} + 2.95 \text{ cc} = 7.37 \text{ cc}$$

Three powder penetrometers listed in the Penetrometer Selection Guide will contain 7.37 cc of sample. Considering the sample size, the one having a total stem volume of 0.392 cc is most appropriate. The sample requires approximately 33% $[0.13/0.39]$ of the stem capacity of the penetrometer. Optimum performance would be achieved if, instead of merely using the minimum 15 g of sample, the penetrometer were filled to capacity, which is approximately 30.5 g $[15 \times 15/7.37]$. The penetration volume would then be about 0.26 cc $[0.13 \times 15/7.37]$ or nearly 66% $[0.26/0.39]$ of the total stem volume.



The previous calculations assume that all interstitial volume will be filled with mercury at the filling pressure. A minimum fill pressure of 0.5 psia will fill cavities of approximately 500 μm diameter, whereas a filling pressure of 1.5 psia will fill cavities as small as 120 μm diameter. If some interstitial volume remains unfilled at this point, allowance for this additional volume must be made in choosing the appropriate stem volume.

The percentage of the maximum intrusion (stem) volume utilized in each station is displayed on the *Status Display* as a guide for the operator. A % STEM reading of less than 25% or more than 90% suggests the need for a procedural change. The first instance suggests a larger quantity of sample might give better resolution and the second indicates that the capillary is on the verge of being depleted.

PREPARE THE SAMPLE

To achieve gains in productivity and reduction in analyzer maintenance, as well as improved data quality, dry the sample material in a shallow pan at 150 °C or higher for one hour. A vacuum oven may be used, although it is not necessary to do so. Use of a vacuum oven is particularly beneficial if the oven is backfilled with dry nitrogen prior to opening. Once the sample is dried, minimize any re-exposure to the atmosphere.

The drying of samples prior to analysis is important, especially for sample types such as fluid cracking catalysts, porous silicas, porous aluminas, and zeolites, which are almost impossible to evacuate without fluidization unless first dried.

The preparation process consists of:

1. [Clean the Penetrometer below](#)
2. [Load the Sample on the next page](#)
3. [Seal the Penetrometer on page 5 - 7](#)
4. [Weigh the Assembled Penetrometer with Sample on page 5 - 9](#)
5. [Install the Penetrometer on page 5 - 10](#)

CLEAN THE PENETROMETER

Clean, dry penetrometers are essential for accurate, reproducible results.



It is recommended that rubber gloves be worn when handling penetrometers.



Never use an ultrasonic bath to clean penetrometers. Ultrasonic cleaning systems will damage the metal plating and remove the serial number information.

1. Dissolve Alconox[®] (or other suitable detergent) in water. Ensure the detergent is completely dissolved before placing the penetrometer into the solution.
2. Place the mercury waste container in a shallow pan of water in case of spills.
3. Hold the penetrometer upright over a mercury waste container to allow any accumulated mercury to drain out.
4. Remove the cap from the penetrometer. If the cap is difficult to remove, see [Penetrometer Nut on page 11 - 34](#).
5. Turn the penetrometer over and pour remaining sample into the waste container.
6. Immerse the penetrometer in the detergent solution. Clean the outside of the penetrometer stem and the bulb with a brush. Then clean the inside of the stem with one of the smaller brushes.

7. Rinse the penetrometer with warm water. Hold the penetrometer upright and ensure that water runs from the bulb through the stem freely.
8. Rinse the penetrometer with IPA.
9. Immerse the stainless steel cap and nylon closure components in the detergent solution. Clean with appropriate brushes and rinse in warm water.
10. If there is any mercury in the bottom of the detergent solution, dispose of the solution properly.
11. Use dry nitrogen to dry the penetrometer, cap, and closure components.

LOAD THE SAMPLE



To avoid transferring skin oils, it is recommended to wear latex gloves when handling penetrometers. Skin oils may affect results.

1. Enter the sample file name (or identifier) on the [Sample Data Worksheet on page F - 6](#).
2. Weigh the sample using an analytical balance. Record the mass on the data sheet as *Sample mass*.
3. Hold the penetrometer with the stem down and carefully pour the sample into the bulb.

When pouring powders into the bulb, place a finger over the stem opening in the center of the bulb so that powder does not enter the stem. A small funnel is useful for loading powders. Large granules or chunks may be loaded with forceps.

SEAL THE PENETROMETER

To avoid transferring skin oils, it is recommended to wear latex gloves when handling penetrometers. Skin oils may affect results.

It is important that penetrometers are clean and dry. See [Clean the Penetrometer on page 5 - 5](#).

1. A vacuum-tight seal is required. Therefore, vacuum grease (Apiezon H) must be used to fill the inevitable roughness of the ground glass lip and polished surface of the cap. To apply grease:

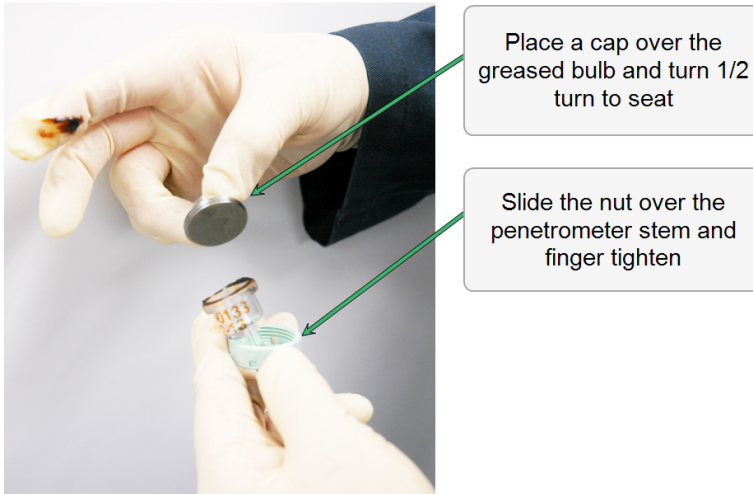


Follow these instructions carefully. Too much grease exposes the sample to an unwanted coating and is likely to cause slippage and misalignment of the mating surfaces. Too little grease results in an imperfect seal.

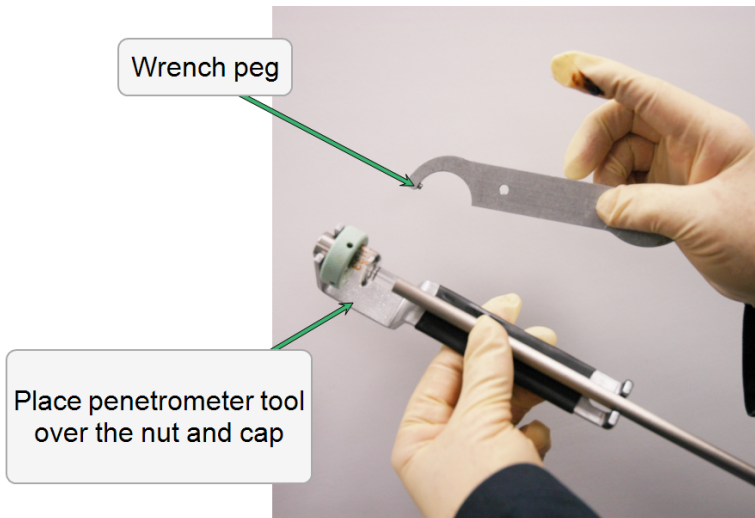
Lightly grease the the tip
of the bulb



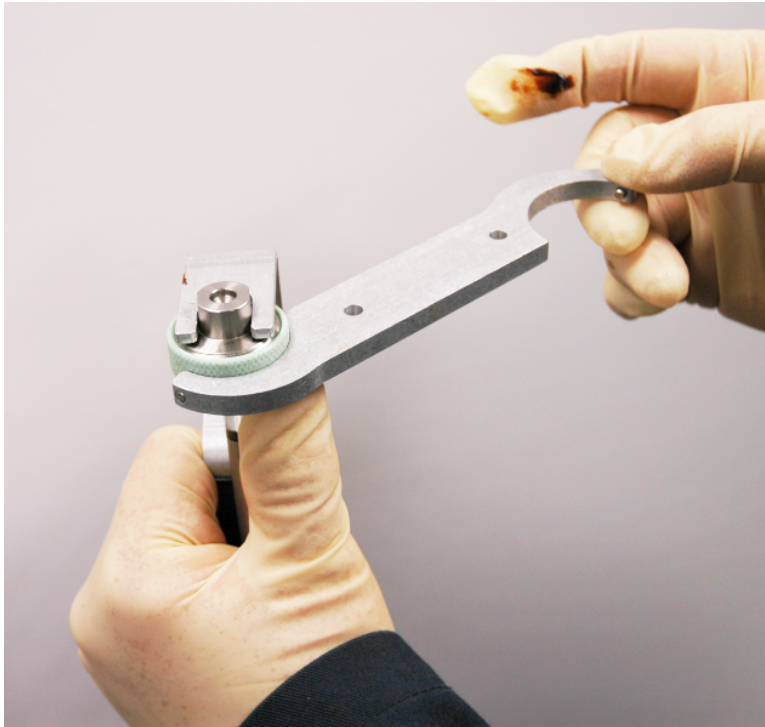
- a. Use a fingertip to apply a light coating of grease to the lip of the bulb.
 - b. Smooth the grease evenly around the lip of the bulb.
 - c. Remove all excess grease from both the inside and outside of the bulb.
2. Hold the penetrometer upright. Place a cap over the greased bulb and turn the cap one half turn to seat.



3. Place the penetrometer tool over the nut.



4. Place the wrench peg into a hole in the penetrometer nut. Use the penetrometer tool to stabilize the cap while tightening the nut counter-clockwise.



5. Weigh the penetrometer before installing it in the low pressure port. See [Weigh the Assembled Penetrometer with Sample below](#).

WEIGH THE ASSEMBLED PENETROMETER WITH SAMPLE



To avoid transferring skin oils, it is recommended to wear latex gloves when handling penetrometers. Skin oils may affect results.

1. Weigh the assembled penetrometer with sample using an analytical balance. Do not include the spacer when weighing.
2. Record the mass on the *Sample Data Worksheet* as *Sample + Penetrometer* mass.
3. Subtract *Sample mass* from *Sample + Penetrometer mass*. Record on the *Sample Data Worksheet* as *Penetrometer mass*.



The mass of the penetrometer must be determined by this method in order to account for the mass of the sealing grease, which varies with each application.

INSTALL THE PENETROMETER

- [*Install a Penetrometer in a High Pressure Chamber on page 10-1*](#)
- [*Install a Penetrometer in a Low Pressure Port on page 10-3*](#)
- [*Remove a Penetrometer from a High Pressure Chamber on page 10-5*](#)
- [*Remove a Penetrometer from a Low Pressure Port on page 10-6*](#)

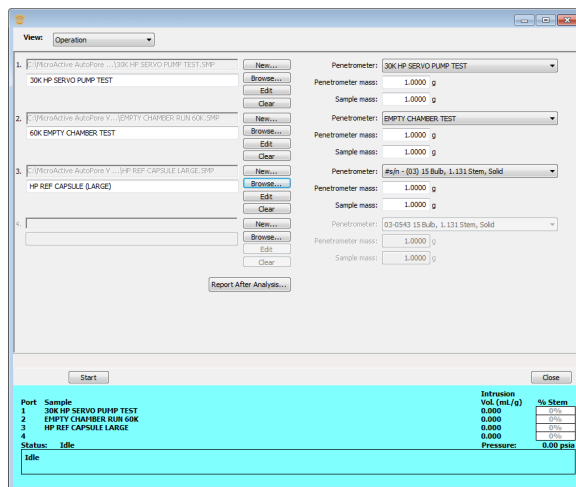
PERFORM A LOW PRESSURE ANALYSIS

Unit [n] > Low Pressure Analysis

- Select the sample files for analysis for each port
- Edit the penetrometer properties
- Edit analysis conditions
- Monitor analysis as data are collected



Prior to starting an analysis, verify that the tank pressure for the gas regulator is at least 200 psig. Pressures less than 200 psig may cause inaccurate data or termination of analysis.



1. Select *Operation* from the *View* drop down to enter or edit analysis information.
2. Install a Penetrometer in a Low Pressure Port. Install a steel rod with cap detector in any low pressure port that does not contain a penetrometer. See [Install a Penetrometer in a Low Pressure Port on page 10-3](#).
3. For a selected port, click **Browse** and select a sample file or click **New** to create a sample file. If needed, change the *Penetrometer mass* and/or *Sample mass* field for the sample material being analyzed.
4. 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. See [Monitor a Low Pressure Analysis on page 5 - 15](#).



- When the **Start** button is clicked, if any low pressure station does not show a cap detector reading that indicates the station has a penetrometer or rod and its cap detector in place, a warning message will be displayed and the analysis will not start.
- When the next scheduled pressure is above atmospheric pressure and a cap detector and rod or pen is not in place, the same warning will be displayed and the analysis will be suspended until the operator has corrected the problem and resumed.
- If a cap detector is removed when the pressure is above atmospheric pressure, the low pressure system will be vented to atmosphere and the analysis canceled, with an explanatory error message.

5. When the analysis is complete, remove the penetrometer and store (or dispose of) the sample material as applicable. See [Remove a Penetrometer from a Low Pressure Port on page 10-6](#).

Low Pressure Analysis Fields and Buttons Table

Field or Button	Description
Penetrometer mass	This information is pulled from the selected sample file. Modify the penetrometer mass in this field as needed.
Sample mass	This information is pulled from the selected sample file. Modify the sample mass in this field as needed.



For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 3](#).

EVACUATE LOW PRESSURE

See Low Pressure in [Analysis Conditions on page 4 - 2](#)

Unit [n] > Evacuate Low Pressure

Select the type of sample when performing a low pressure evacuation. This window is also available using the **Evacuation** button on the *Analysis Conditions* tab.

Select if the sample is a *Formed solid*, *Powder*, or *Other*. If *Other* is selected, the remaining fields are enabled.

Evacuate Low Pressure Fields and Buttons Table

Field or Button	Description
Initially evacuate at	Enter the initial maximum evacuation rate.
Switch to medium at	Enter the method and pressure the system must reach before medium evacuation begins. <ul style="list-style-type: none"> • Use pressure transducer • User vacuum gauge • Skip medium evacuation .
Switch to fast at	Enter the pressure the system must reach before fast evacuation

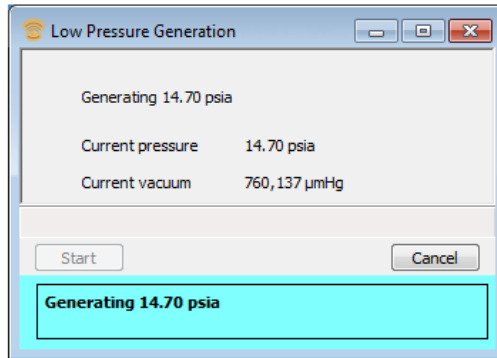
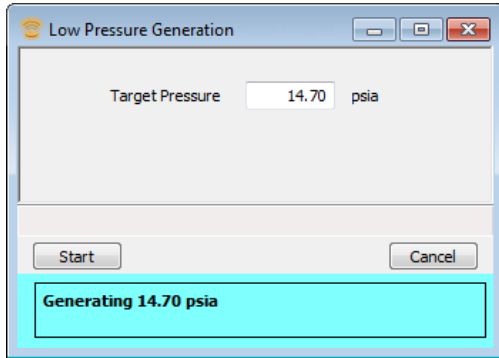
Evacuate Low Pressure Fields and Buttons Table (continued)

Field or Button	Description
	begins.
Evacuation target	Enter the evacuation pressure.
Continue evacuating for	Enter the evacuation duration.

GENERATE LOW PRESSURE

Unit [n] > Generate Low Pressure

Pressure can be generated in the low pressure system when a low pressure analysis is not already in progress.



Threaded penetrometer closures are required for pressures above 30 psia or 0.2068 MPa.

MONITOR A LOW PRESSURE ANALYSIS

This live graph displays data as they are collected. It also shows intrusion as a function of pressure. The status section below the graph displays the following for each port:

- Sample file name
- Intrusion volume
- Percent of the penetrometer stem that is filled with mercury



When monitoring a low pressure analysis, the analyzer schematic can be displayed to show the state of the low pressure system components. See [Show Instrument Schematic on page 2 - 16](#).

Low Pressure Analysis Fields and Buttons Table

Field or Button	Description
Pressure	Displays the current pressure in the low pressure system. The reading shown is from either the vacuum gauge or the 50 psia transducer, depending upon which is currently in range.
Resume	Restarts the suspended analysis.
Skip	Moves to the next step. Select the ports to skip.
Status	Displays current low pressure status.
Suspend	Suspends an analysis in progress.



For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 3](#).

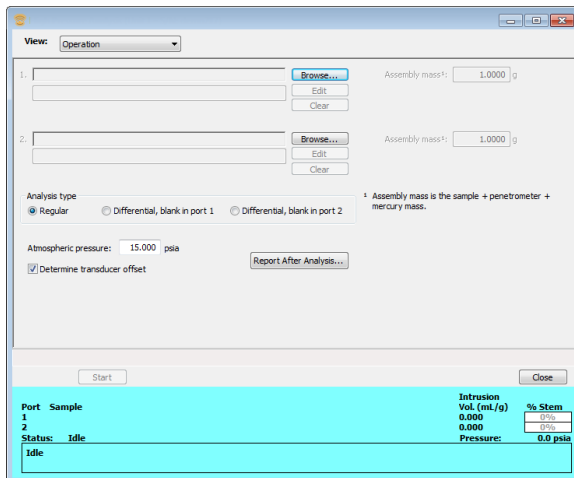
PERFORM A HIGH PRESSURE ANALYSIS

The analyzer is designed to perform two high pressure analyses simultaneously. To run only one analysis, the other chambers must be closed tightly and have sufficient high pressure fluid to be drawn into the vent valve.

Unit [n] > High Pressure Analysis

Low and high pressure analyses can be performed simultaneously.

- Select the sample files for analysis for each port
- Edit the penetrometer properties
- Edit analysis conditions
- Monitor analysis as data are collected



Port	Sample	Intrusion Vol. (mL/g)	% Stem
1		0.000	0.000
2		0.000	0.000

Status: Idle
Pressure: 0.0 psia



Each sample's high pressure analysis should be performed on the same analyzer as the low pressure analysis. The analyzer checks to see that the same analyzer is used before it begins the high pressure analysis; if it is not, the analyzer displays a warning message. The analysis can be continued or canceled.

1. Select the sample file with a status of *Low Pressure Complete* or *High Pressure Complete*.

If a selected sample file to be analyzed on a 9505 analyzer contains pressures exceeding 33,000 psia, the following message displays:

- Click **Yes** to proceed with the analysis; only pressures less than 33,000 psia will be used.
- Click **No** to cancel the analysis.

2. Install a Penetrometer in a High Pressure Port. See [Install a Penetrometer in a High Pressure Chamber on page 10-1](#).
3. Click **Start** to start the analysis immediately. A live graph of the analysis displays as data are being collected. See [Monitor a High Pressure Analysis on page 5 - 19](#).
4. Remove the penetrometer after the analysis. See [Remove a Penetrometer from a High Pressure Chamber on page 10-5](#).



If it was not requested that the transducer offset be determined (**Options > Atmospheric Pressure**), and if the current high pressure reading differs significantly from the atmospheric pressure, a prompt displays requesting verification that the system is at atmosphere.



For maintenance purposes, it is possible to perform a high pressure analysis with no penetrometer. Open the *High Pressure Analysis* window and enter the name of a file with a *No Analysis* status. Continue the analysis after a warning message displays.

High Pressure Analysis Fields and Buttons Table

Field or Button	Description
Analysis type	<p>Regular. Performs a differential analysis with the blank penetrometer in port 1.</p> <p>Differential, blank in port 1. Performs a differential analysis with the blank penetrometer in port 1.</p> <p>Differential, blank in port 2. Performs a differential analysis with the blank penetrometer in port 2.</p> <p>See High Pressure Differential Analysis on page 5 - 20</p>
Assembly mass	Sample + penetrometer + mercury mass.
Atmospheric pressure	Enter the atmospheric pressure if it differs from the default entry. The default entry is pulled from the information entered in Options > Atmospheric Pressure .
Determine transducer offset	Select to allow the system to determine the transducer offset. The default entry is pulled from the information entered in Options > Atmospheric Pressure .

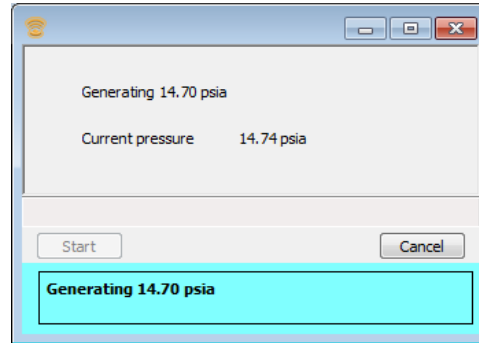
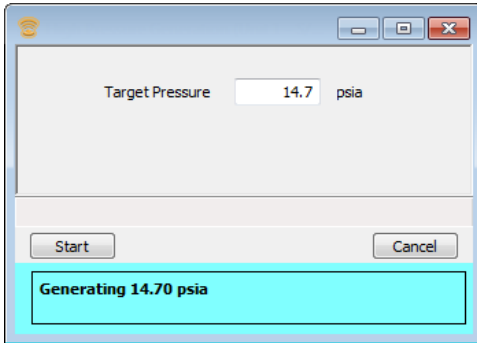


For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 3](#).

GENERATE HIGH PRESSURE

Unit [n] > Generate High Pressure

Pressure can be generated in the high pressure system when a high pressure analysis is not already in progress.



MONITOR A HIGH PRESSURE ANALYSIS

This live graph displays data as they are collected. It also shows an intrusion as a function of pressure. The status section below the graph displays the following for each port:

- Sample file name
- Intrusion volume
- Percent of the penetrometer stem that is filled with mercury



When monitoring a high pressure analysis, the analyzer schematic can be displayed to show the state of the high pressure system components.

High Pressure Analysis Fields and Buttons Table

Field or Button	Description
Pressure	Displays the current pressure in the high pressure system.
Skip	Moves to the next step. Select the ports to skip.
Resume	Restarts the suspended analysis.
Status	Displays the high pressure's current status.
Suspend	Suspends an analysis in progress.



For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 3](#).

HIGH PRESSURE DIFFERENTIAL ANALYSIS

If using a blank correction file as the correction method, the correction file must exist before the sample data file can be completed (since a blank correction file must be selected when the sample file is created).

If a blank analysis has not been performed in advance, run a differential analysis of the sample and the corresponding blank simultaneously. The application associates the sample file with the blank correction file after the analysis is completed.

An advantage of using differential analysis is that both the sample and blank penetrometers are subjected to nearly identical conditions (time, temperature, and pressure). Properly used, the differential analysis option provides the most accurate low porosity sample data possible.

1. Create a sample file. Also create a sample file for the blank penetrometer using *None* for the correction method. See [Sample Files on page 3 - 1](#).
2. Run the low pressure analysis on both files. See [Perform a Low Pressure Analysis on page 5 - 11](#).
3. To start the high pressure analysis, select either the *Differential, Blank in port 1* or *Differential, Blank in port 2*. Perform a high pressure analysis. See [Perform a High Pressure Analysis on page 5 - 16](#).

REFERENCE MATERIAL ANALYSIS


HIGH PRESSURE REFERENCE MATERIAL ANALYSIS

Unit [n] > High Pressure Reference Analysis

A reference material 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 material 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.

High Pressure Reference Material Analysis Fields and Buttons Table

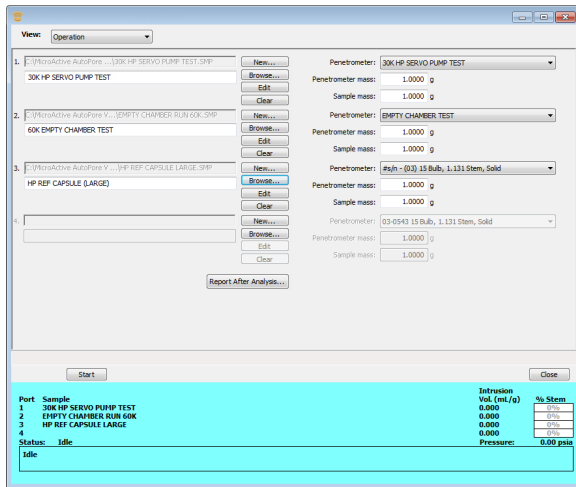
Field or Button	Description
Assembly mass	This information is pulled from the selected sample file. Sample + penetrometer + mercury mass.
 For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3 .	

LOW PRESSURE REFERENCE MATERIAL ANALYSIS

Unit [n] > Low Pressure Reference Analysis

A reference material 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 material 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.



Low Pressure Material Reference Material Analysis Fields and Buttons Table

Field or Button	Description
Penetrometer	Penetrometer description from the selected sample file.
Penetrometer mass	Penetrometer mass from the selected sample file.
Sample mass	Sample mass from the selected sample file.

For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 3](#).

LEAPFROGGING - SUGGESTED SEQUENCE FOR MAXIMUM THROUGHPUT

Leapfrogging is a procedure for running low and high pressure systems simultaneously to maximize throughput. This process describes leapfrogging a group of eight samples. It is possible to leapfrog groups of samples continuously.



If samples require longer vacuum preparation or heated evacuation, increase the throughput by preparing samples in a vacuum oven.

1. Prepare and weigh eight samples. Load eight penetrometers with samples then weigh them. Complete the [Sample Data Worksheet on page F - 6](#) and install samples 1 through 4 in the low pressure ports.
2. Create sample files for at least the first four samples.
3. Go to **Unit [n] > Low pressure analysis** to start the low pressure analysis.
4. When the run is complete, remove samples 1 through 4 from the low pressure ports and weigh them. Install samples 5 through 8 in the low pressure ports.
5. Start another low pressure run.
6. Place samples 1 and 2 in the high pressure chambers.
7. Go to **Unit [n] > High pressure analysis** to start the high pressure analysis.



While waiting for analysis to complete, samples files can be created and other samples can be prepared for analysis.

8. When the high pressure run is complete, remove samples 1 and 2 from the high pressure chambers and replace with samples 3 and 4.
9. Start another high pressure run.
10. Automatic reports specified in each sample file will print when that sample's high pressure run ends.
11. When the second low pressure run and the second high pressure run end, remove samples 3 and 4 from the high pressure chambers. Remove samples 5 through 8 from the low pressure ports and weigh them.
12. Install samples 5 and 6 in the high pressure chambers. Start another high pressure run.
13. When the third high pressure run is finished, install samples 7 and 8 in the high pressure chamber and start another high pressure run.

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6 ABOUT REPORTS

Reports > Open Report > [.REP file]

Opens a saved report.

Reports > Close Reports

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

Reports > Start Report

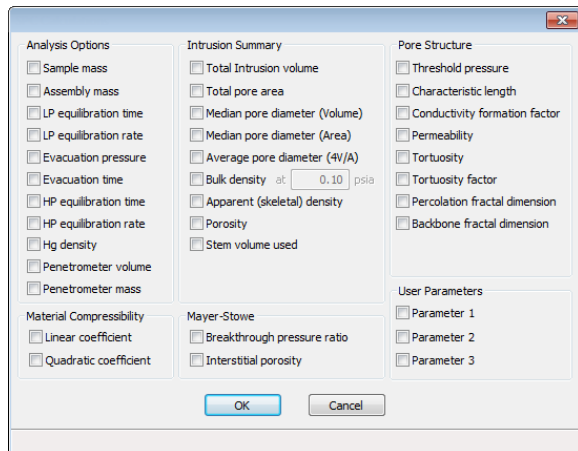
Generates a report an a sample analysis.

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

SPC REPORT

Reports > SPC Report Options

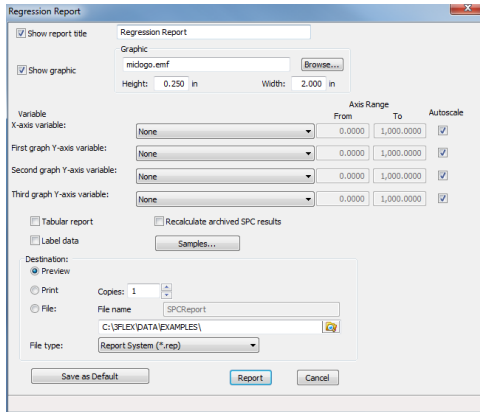
Use to generate reports with various SPC (Statistical Process Control) options. All selected variables must be computed for each sample file used in an SPC report; therefore, it is more efficient to select only the necessary variables.



REGRESSION REPORT

Reports > Regression Report



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



Regression Report Fields and Buttons Table

Field or Button	Description
Autoscale [check box]	When enabled, allows the x- and y-axes to be scaled automatically.
Axis Range [text box]	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if <i>Autoscale</i> is selected.
Label data [check box]	Use to label the points on the plot to correspond with the values in the sample files.
Recalculate archived SPC results [check box]	Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report. <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. If an earlier application version will not be reused, this warning message can be safely disregarded.</p> </div> <p>When this option is selected, this message will display:</p>

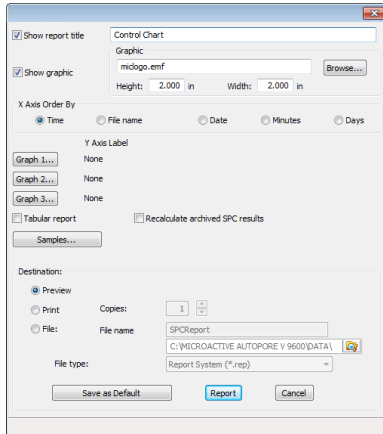
Regression Report Fields and Buttons Table (continued)

Field or Button	Description
	<p>Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.</p> <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>
Samples [button]	Select additional sample files to add to the report.
Save as Default [button]	Click to save selected report options as default report settings.
Show graphic [checkbox]	Use to show a graphic on the report header. Height / Width. Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title [checkbox]	Select and enter a report title to appear on the report header.
Tabular report [checkbox]	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X- and Y-Axis Variable [drop-down box]	Use to designate the x- and y-axes variables. The variables in the drop-down lists are those selected in the Reports > SPC Report Options window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.
	<p>For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.</p>

CONTROL CHART REPORT

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

Control Chart Fields and Buttons Table (continued)

Field or Button	Description
Recalculate archived SPC results [<i>check box</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/>  <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. If an earlier application version will not be reused, this warning message can be safely disregarded.</p> <hr/> <p>When this option is selected, this message will display:</p> <div style="border: 1px solid gray; padding: 5px; background-color: #f0f0f0;"> <p>Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.</p> </div> <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>
Report [<i>button</i>]	Generates the report.
Samples [<i>button</i>]	Select additional sample files to add to the report.
Show graphic [<i>check box</i>]	Use to show a graphic on the report header. Height / Width. Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title [<i>check box</i>]	Select and enter a report title to appear on the report header.
Tabular report [<i>check box</i>]	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.

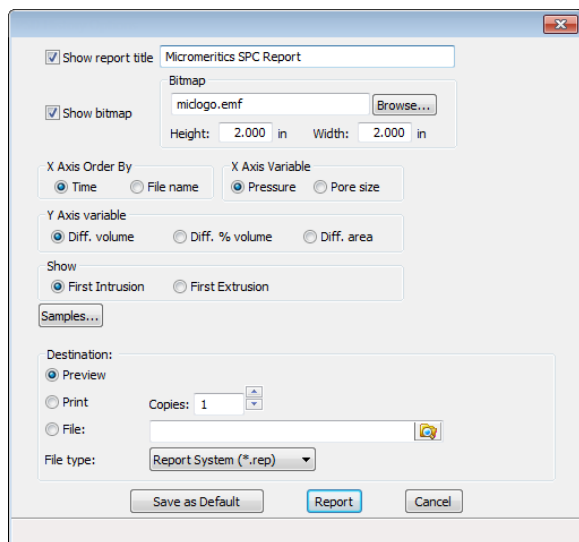
Control Chart Fields and Buttons Table (continued)

Field or Button	Description
X Axis Order by [group box]	Select the order in which x-axis statistics are placed. Sort by: Time. Time the files were analyzed. File name. Alphanumeric order. Date. Date the files were analyzed. Minutes. Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. Days. Number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3 .

PSD HISTORY REPORT

Reports > PSD History


The *PSD History Report* generates a sequence of full pore size distribution graphs.



PSD History Report Fields and Buttons Table

Field or Button	Description
Report [<i>button</i>]	Generates the report.
Samples [<i>button</i>]	Select additional sample files to add to the report.
Save as Default [<i>button</i>]	Click to save selected report options as default report settings.
Show [<i>selection</i>]	Select one of the following options: First Intrusion. Select to show the first intrusion segment. First Extrusion. Select to show the first extrusion segment.
Show bitmap [<i>checkbox</i>]	Use to show a graphic on the report header. Height / Width. Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title [<i>checkbox</i>]	Select and enter a report title to appear on the report header.

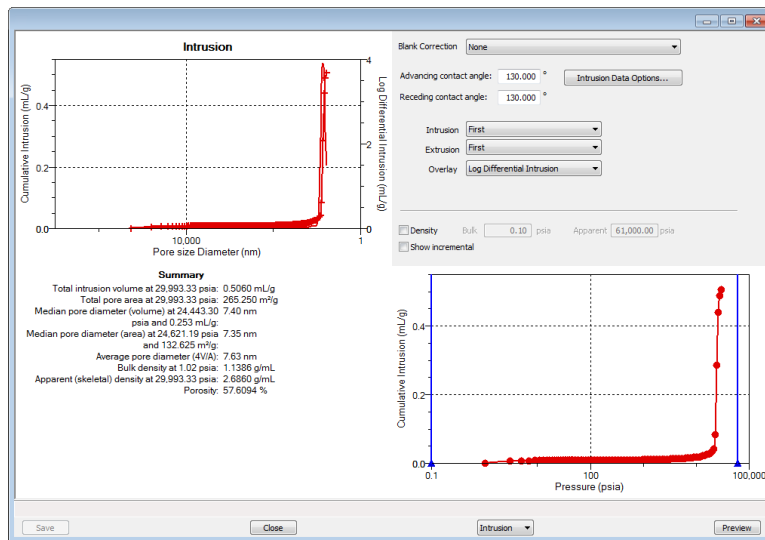
PSD History Report Fields and Buttons Table (continued)

Field or Button	Description
X-axis Order By [selection]	Select one of the following: Time. Time the files were analyzed. File name. Alphanumeric order.
X Axis variable [selection]	Select one of the following to display on the graph: Pressure. Pore size.
Y Axis variable [selection]	Select one of the following to display on the graph: Diff. volume. Diff. % volume. Diff. area.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3 .

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 will display:
 - an intrusion linear plot and log plot of the data collected during analysis
 - a summary of the analysis giving a single total intrusion volume and other important quantities

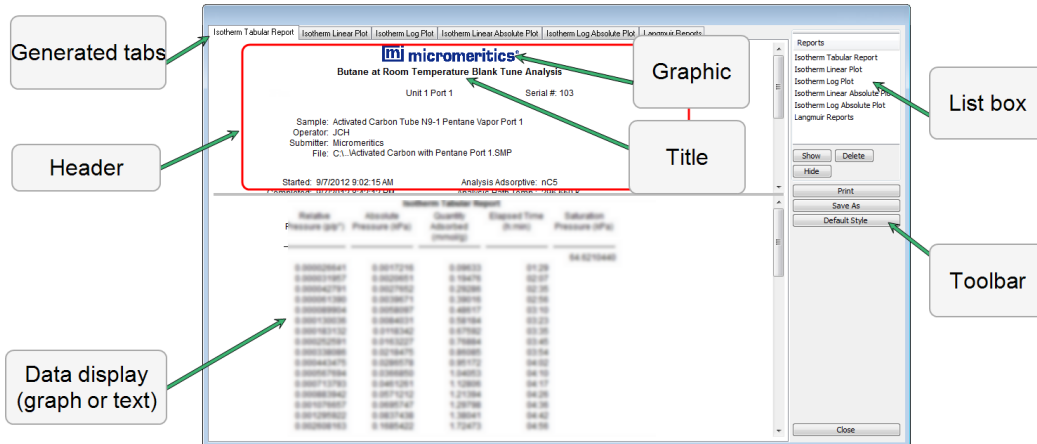


- Modify the intrusion graph to show all or first intrusion and/or extrusion segments by selecting an option from the *Intrusion* or *Extrusion* drop-down lists.
- Add an overlay to the intrusion graph by selecting an option from the *Overlay* drop-down list.
- To view the reports selected for generation during or after 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.
 - 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**.

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.

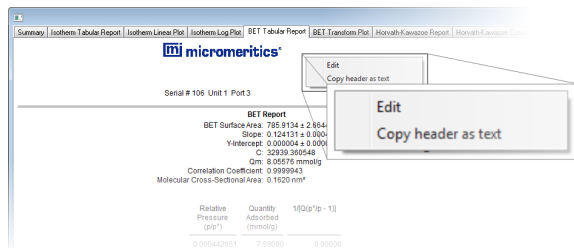


If configured, the report header can also contain a graphic and/or a title.

- 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.

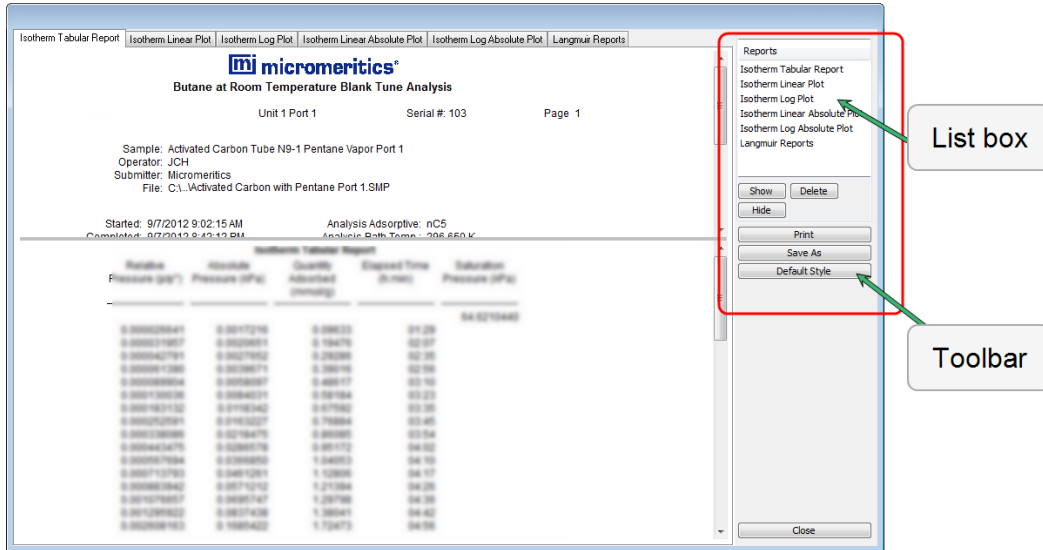
- Tabular and graphical reports contain sample and analyzer statistics — such as analysis date, analysis time, analysis conditions, etc.
- The headers contain notes of sample file changes occurring after analysis.
- Summary report headers contain the same information in tabular and graphical reports 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
Default Style [<i>button</i>]	Specify default report parameters for fonts and curve properties.
Delete [<i>button</i>]	Deletes the selected report in the <i>Reports</i> list box. Deleted reports will have to be regenerated if deleted in error.
Hide [<i>button</i>]	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the <i>Reports</i> list box.
Print [<i>button</i>]	Displays the <i>Print</i> window for report output.
Reports [<i>group box</i>]	Contains a list of all generated reports. The same reports display as tabs at the top of the report header unless the report has been hidden using the Hide button.
Show [<i>button</i>]	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 - 3](#).

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.

The screenshot shows the AutoPore IV software interface. At the top, there are navigation tabs: Summary, Error, Tabular Report, Cum. Vol. vs Size, Inc. Vol. vs Size, Diff. Vol. vs Size 1, Cum. Area vs Size, Log Diff. Vol. vs Size 1, and Volume Scale. The main window displays the Micromeritics logo and company name. Below this, it shows sample information: Sample: Rock Sample, Operator: N. KELLY, Submitter: Research Lab, File: C:\AutoPore\data\examples\ROCK.SMP. A data table titled "Tabular Report" is visible, with columns for Pressure (psia), Pore Diameter (nm), Incremental Pore Volume (mL/g), and Cumulative Pore Volume (mL/g). A context menu is open over the table, listing options: Resize column, Rename column, Move column, Align column, Show column, Table data font, Table header font, Edit title..., and Copy table as text.

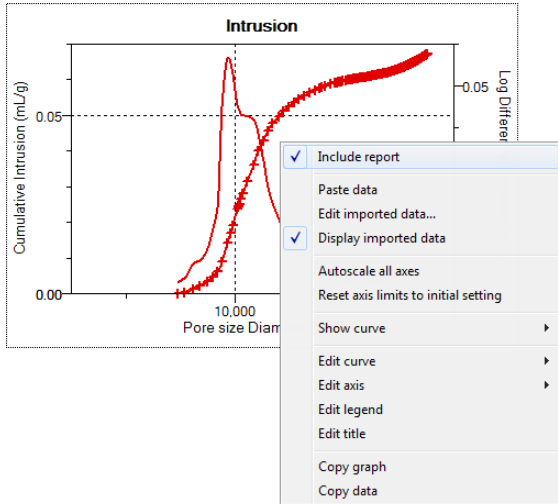
Tabular Reports Shortcut Options and Descriptions Table

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

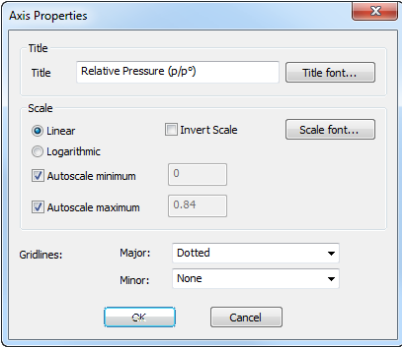
For fields and buttons not listed in this table, see [Common Fields and Buttons on page 2 - 3](#).

GRAPH FEATURES AND SHORTCUTS

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



Graph Shortcuts Options and Description Table

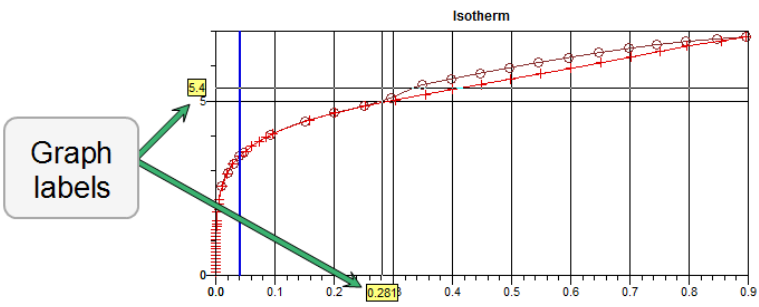
Field or Button	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Edit axis	<p>Use to edit the selected axis properties.</p>  <p>Autoscale minimum / maximum. To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box.</p> <p>Grid lines. Use to change how to display major / minor grid lines.</p> <p>Invert scale. Use to invert the scale.</p>

Graph Shortcuts Options and Description Table (continued)

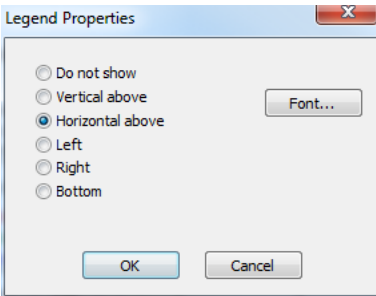

Field or Button	Description
	<p>Linear / Logarithmic. Select the option to scale the graph as linear or logarithmic.</p> <p>Scale font. Use to modify the font for the scale label. Deselect <i>Use default font</i> to enable font options.</p> <p>Title. Use to edit the selected axis label.</p> <p>Title font. 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>

Graph Shortcuts Options and Description Table (continued)

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

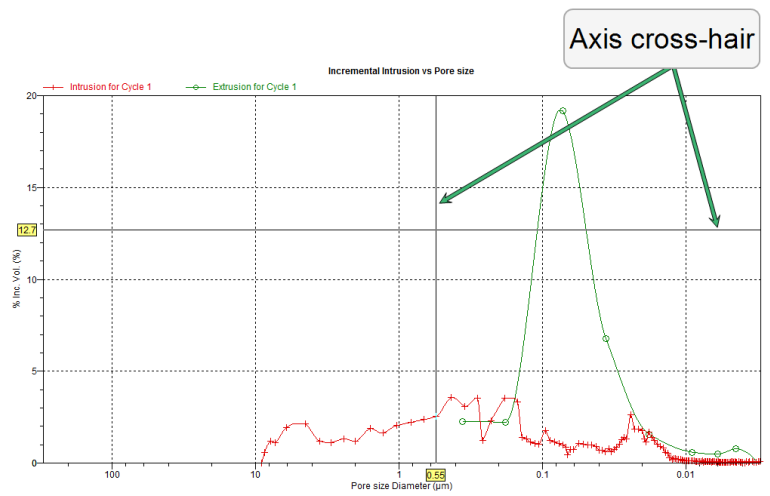


Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
Edit legend	Use to change the legend location and font. 
Edit title	Use to change the graph title and font.
Enable Overlays	If overlays have been selected, this option displays (or hides) the overlays.
Include report	When selected, places a checkmark to the left of the report in the <i>Select Reports</i> list box on the <i>Report Options</i> tab.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
Select overlays...	Displays the option to select files to overlay onto the active graph. To view the overlays, click <i>Enable Overlays</i> on the shortcut menu.
Show curve	Displays a list of all curves. Select the curve(s) to display.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3 .

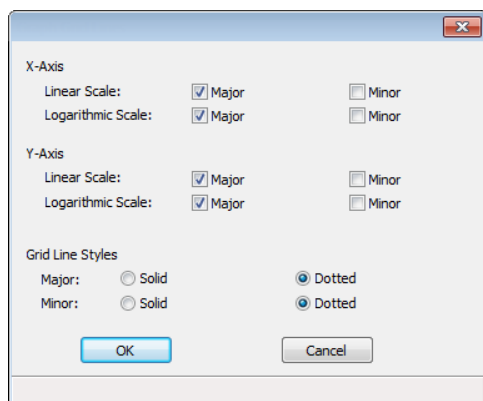
Axis Cross-Hair

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
Grid Line Styles [selection]	Select if the major and/or minor grid lines should appear as solid or dotted lines.
X-Axis / Y-Axis [selection]	Select major and/or minor lines to display in reports for the logarithmic and linear scales. Deselect this option to remove the grid lines.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3 .

Zoom Feature

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

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 following reports in the mercury porosimetry application can produce graphical results for a sample material's pore size distribution:

Physical Adsorption	Mercury Porosimetry
<ul style="list-style-type: none"> • BJH Adsorption / Desorption • Dollimore-Heal Adsorption / Desorption • Horvath-Kawazoe • DFT Pore Size 	<ul style="list-style-type: none"> • Cumulative • Differential • Incremental Intrusion graphs

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

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

Pore size distribution report overlays menu selections are:

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

IMPORT ASCII TEXT DATA

ASCII text file format rules

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

Two examples of a header format:

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

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

Sample ASCII Text File

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

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

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

OVERLAY PORE SIZE DISTRIBUTION DATA USING COPY/PASTE

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

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

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

Print Pore Size Distribution Overlay Data in Reports

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

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 - 8](#).
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.

- 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

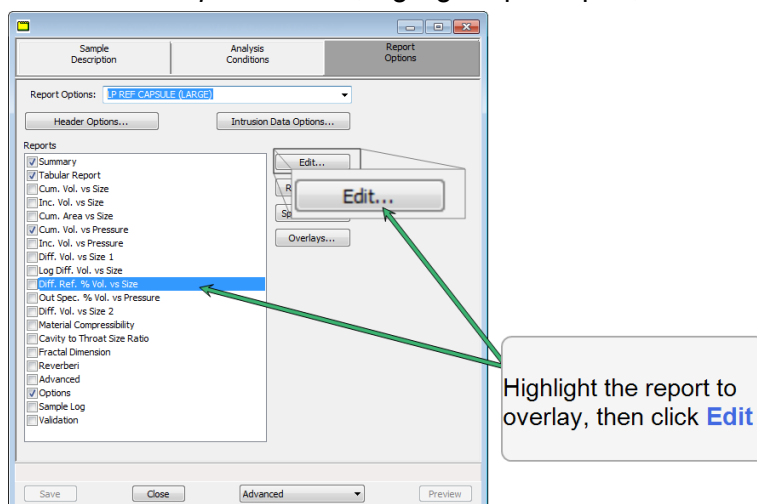
- Create an ASCII text file. See [Manually Enter Data on page 3 - 8](#).
- Copy the ASCII text data to the clipboard.
- Open a report with a *Complete* status.
- Select a pore-size distribution report from the drop-down list at the bottom of the window.
- Right click on the graph and select *Paste data* on the shortcut menu.
- 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

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

OVERLAY MULTIPLE MERCURY POROSIMETRY GRAPH OPTIONS

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

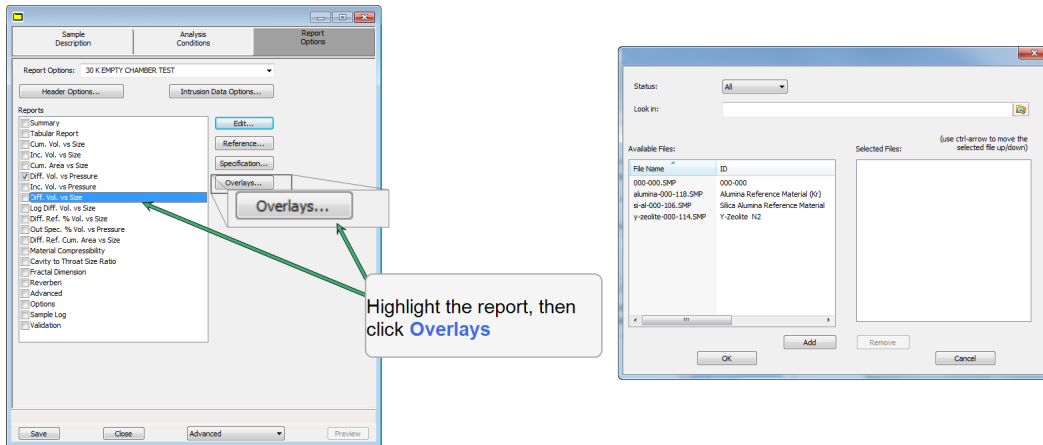


6. On the *Plot Options* window, select *Options* to include in the overlay. If the x- and/or y-axes are to be autoscaled, select *Autoscale*; otherwise, enter the *From* and *To* points for the axes.
7. In the *Y-Axis* group box, select *Variable* and/or *Overlay* options.
8. Click **OK** to return to the *Report Options* tab.
9. Click **Save**, **Save As**, or **Preview**.

OVERLAY MULTIPLE SAMPLE FILES

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

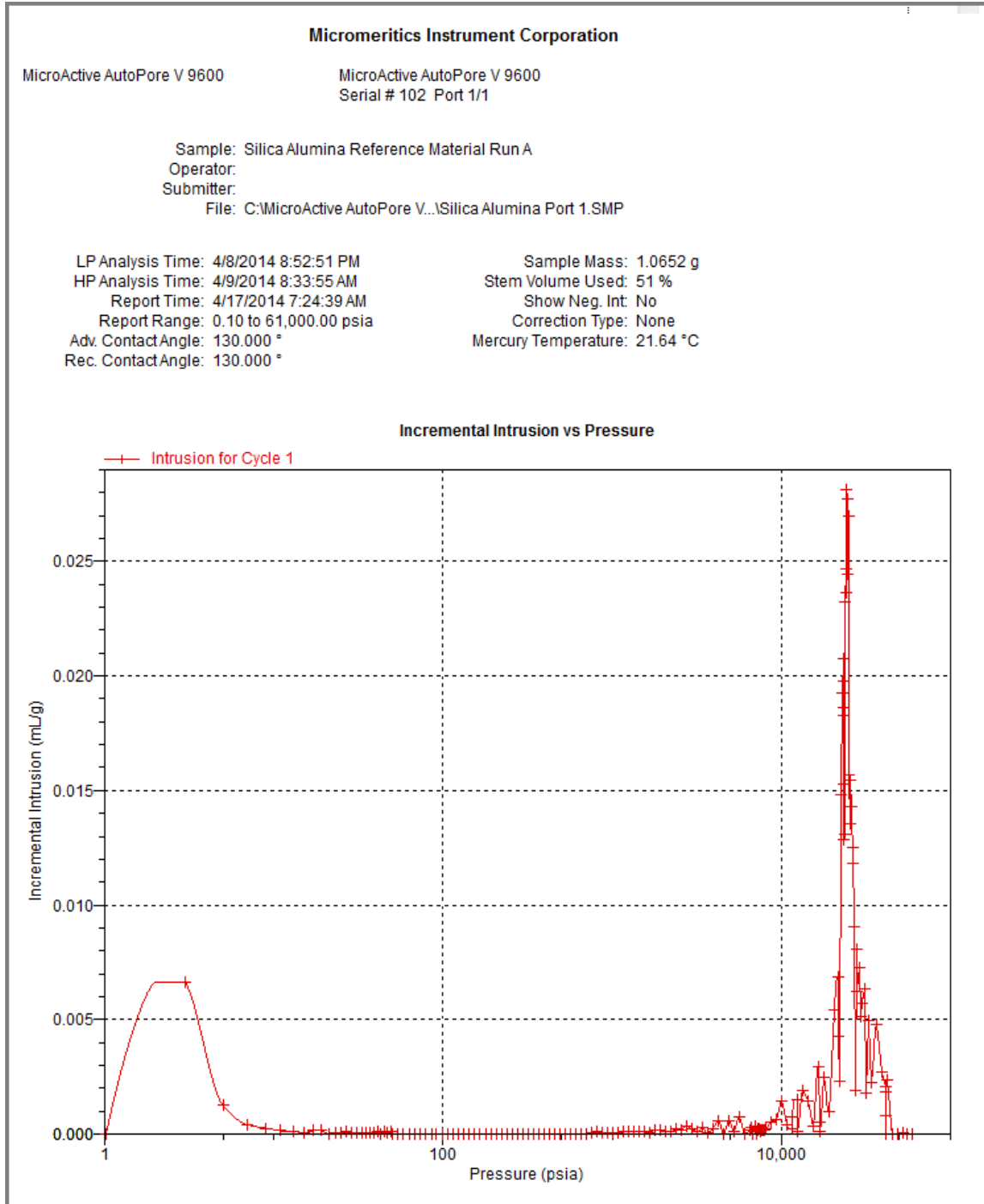
1. Go to **File > Open**.
2. Select the .SMP file, then click **Open**. Select *Advanced* from the drop-down list at the bottom of the window to display the *Report Options* tab.
3. Click the *Report Options* tab.
4. In the *Reports* list box, highlight a plot report, then click **Overlays**.



5. On the *Plot Overlay Sample Selection* window, move files from the *Available Files* box to the *Selected Files* box.
6. Click **Save**, **Save As**, or **Preview**.

REPORT EXAMPLES

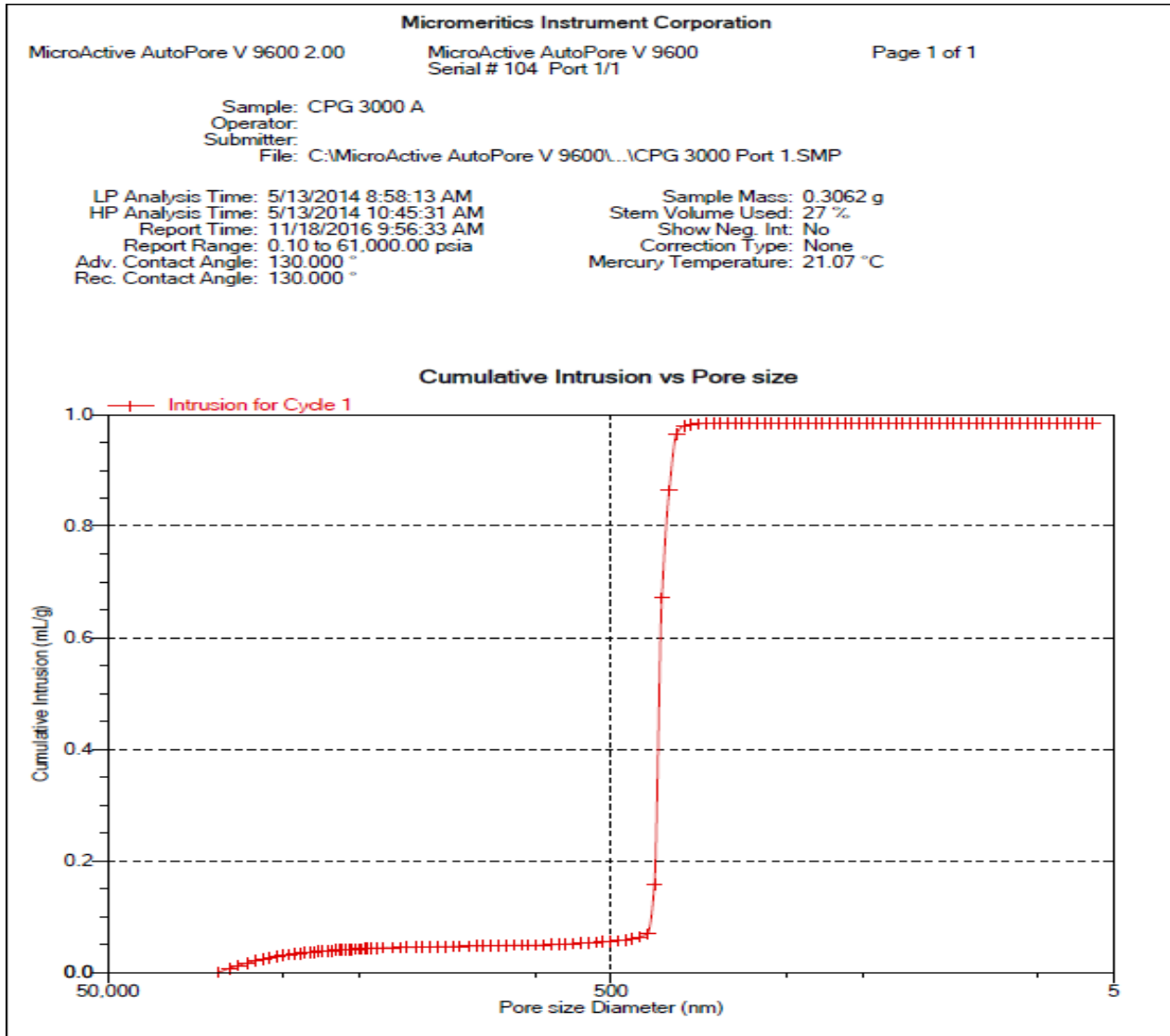
SILICA ALUMINA REFERENCE MATERIAL REPORT



GARNET TABULAR REPORT

Micromeritics Instrument Corporation					
MicroActive AutoPore V 9600		MicroActive AutoPore V 9600 Serial # 102 Port 1/1			
Sample: garnet ref mat 60k eqil (rate) 568					
Operator: jch					
Submitter: micromeritics performance test					
File: C:\MicroActive AutoPore V...\Garnet to 60K Port 1.SMP					
LP Analysis Time: 4/10/2014 3:16:40 PM		Sample Mass: 0.2899 g			
HP Analysis Time: 4/10/2014 5:21:38 PM		Stem Volume Used: 28 %			
Report Time: 4/17/2014 7:36:34 AM		Show Neg. Int: No			
Report Range: 0.10 to 61,000.00 psia		Correction Type: None			
Adv. ContactAngle: 130.000 °		Mercury Temperature: 21.28 °C			
Rec. ContactAngle: 130.000 °					
Tabular Report					
Pressure (psia)	Mean Diameter (µm)	Cumulative Pore Volume (mL/g)	Incremental Pore Volume (mL/g)	Cumulative Pore Area (m²/g)	Incremental Pore Area (m²/g)
1.02	176.49789	0.0000	0.0000	0.000	0.000
2.00	133.52629	0.0095	0.0095	0.000	0.000
3.00	75.44654	0.0206	0.0111	0.001	0.001
4.00	52.79904	0.0260	0.0053	0.001	0.000
5.00	40.72799	0.0314	0.0055	0.002	0.001
6.00	33.17779	0.0347	0.0032	0.002	0.000
6.99	28.00920	0.0379	0.0032	0.003	0.000
8.00	24.23739	0.0405	0.0026	0.003	0.000
8.99	21.36405	0.0429	0.0024	0.004	0.000
10.00	19.10306	0.0439	0.0011	0.004	0.000
10.99	17.27178	0.0460	0.0020	0.004	0.000
11.99	15.76445	0.0481	0.0021	0.005	0.001
13.00	14.49805	0.0499	0.0018	0.005	0.001
13.99	13.42028	0.0515	0.0016	0.006	0.000
14.99	12.49258	0.0531	0.0016	0.006	0.000
15.99	11.68533	0.0542	0.0011	0.007	0.000
16.99	10.97620	0.0555	0.0013	0.007	0.000
17.99	10.34770	0.0567	0.0012	0.008	0.000
18.99	9.78763	0.0577	0.0009	0.008	0.000
19.99	9.28496	0.0587	0.0010	0.008	0.000
20.99	8.83121	0.0598	0.0011	0.009	0.000
21.99	8.42027	0.0608	0.0010	0.009	0.000
22.99	8.04556	0.0616	0.0009	0.010	0.000
23.99	7.70264	0.0627	0.0010	0.010	0.001
24.99	7.38819	0.0635	0.0009	0.011	0.000
25.99	7.09860	0.0639	0.0004	0.011	0.000
26.99	6.83063	0.0653	0.0014	0.012	0.001
27.99	6.58155	0.0657	0.0004	0.012	0.000
28.99	6.35046	0.0671	0.0014	0.013	0.001
30.00	6.13444	0.0679	0.0009	0.014	0.001
30.45	5.98441	0.0683	0.0004	0.014	0.000
30.92	5.89437	0.0687	0.0004	0.014	0.000

CONTROLLED PORE GLASS PLOT



Blank Page

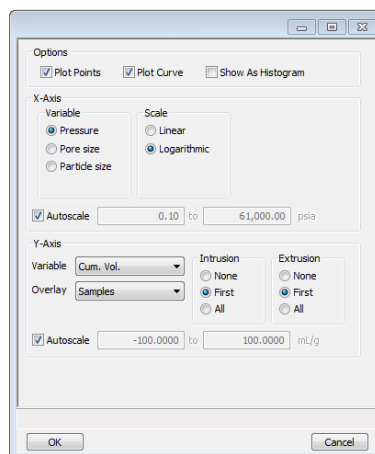
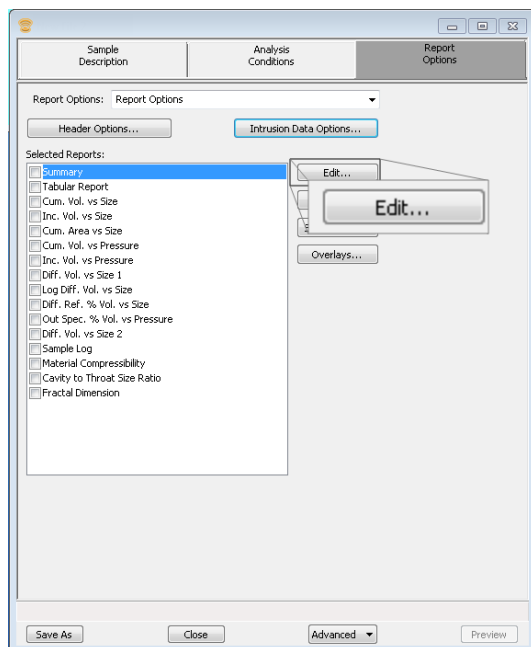
7 SELECTED REPORTS




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**.

GRAPH REPORT OPTIONS

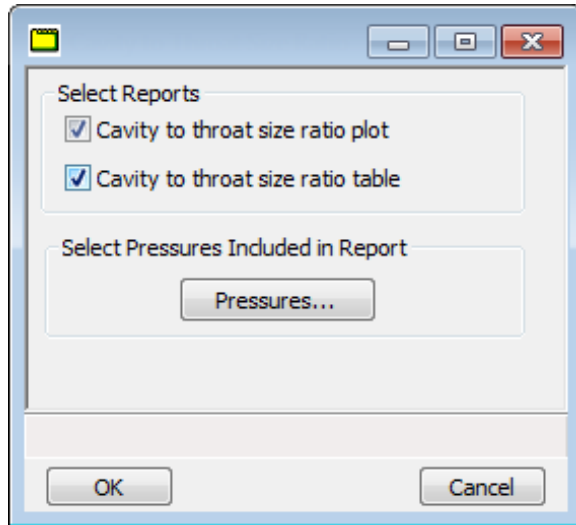
- Cumulative Area vs Size Plot Options
- Cumulative Volume vs Pressure Plot Options
- Cumulative Volume vs Size Plot Options
- Difference from Reference % Volume Plot Options
- Differential Intrusion Plot Options
- Differential Volume vs Size Plot Options
- Incremental Volume vs Pressure Plot Options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



Graph Report Options Fields and Buttons Table


Field or Button	Description
Autoscale [<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.
Options [<i>check box</i>]	<p>Plot Points. Select to plot the points on the graph.</p> <p>Plot Curve. Interpolated from data points.</p> <p>Show As Histogram. When selected, <i>Plot Points</i> and <i>Plot Curve</i> options are disabled.</p>
Overlays [<i>button</i>]	Select sample files that contain data to be overlaid onto the selected plot.
Reference [<i>button</i>]	Select a sample file to compare analysis results of the current sample.
Specification [<i>button</i>]	Specify sample files to use for the boundaries of the coarse and fine specifications.
X-Axis [<i>group box</i>]	<p>Select options for the x-axis.</p> <p>Variable.</p> <p>Scale.</p>
Y-Axis [<i>group box</i>]	<p>Select options for the y-axis.</p> <p>Variable. Select the y-axis variable from the drop-down list.</p> <p>Overlay. [<i>Optional</i>]. Select an option to overlay.</p> <p>Intrusion / Extrusion. Select the data points to plot.</p> <ul style="list-style-type: none"> • None. No intrusion (or extrusion) data points • First. Points from the first intrusion (or extrusion) cycle • All. Include all intrusion (or extrusion) data points
	<p>For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.</p>

CAVITY TO THROAT SIZE RATIO REPORT OPTIONS



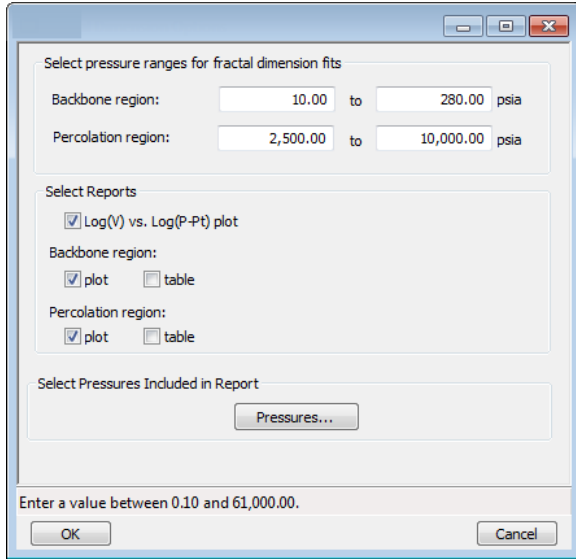
Select to show the cavity to throat size ratio plot ratio table.

Cavity to Throat Size Ratio Fields and Buttons Table


Field or Button	Description
Cavity to throat size ratio plot [<i>checkbox</i>]	Displays the ratio plot.
Cavity to throat size ratio table [<i>checkbox</i>]	Displays the ratio table
Pressures [<i>button</i>]	Enter the minimum and maximum calculation pressure range.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3 .

FRACTAL DIMENSION REPORT OPTIONS

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

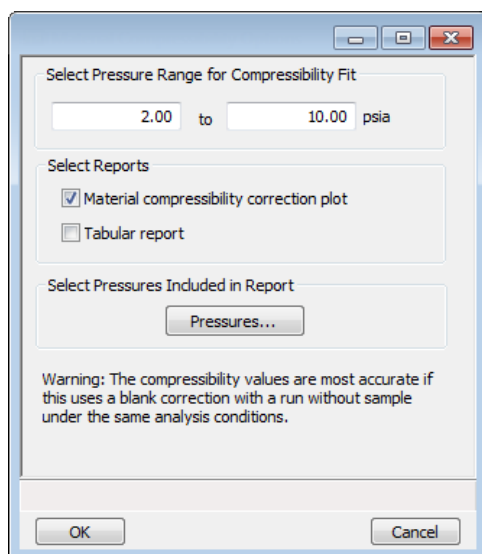


Fractal Dimension Report Fields and Buttons Table


Field or Button	Description
Backbone region [text box]	Enter the pressure at which the calculations are to be performed.
Percolation region [text box]	Enter the pressure at which the calculations are to be performed.
Pressures [button]	Select to enter minimum and maximum pressures.
Select Reports [check box]	Show Log(V) vs Log (P-Pt) graph. Select to generate an additional graph to help select linear range for calculations. Backbone region. Percolation region.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.

MATERIAL COMPRESSIBILITY REPORT OPTIONS

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



Material Compressibility Fields and Buttons Table

Field or Button	Description
Pressures [<i>button</i>]	Enter the Calculation pressure range.
Range [<i>text box</i>]	P1. Enter the beginning pressure. P2. Enter the ending pressure.
Select Reports [<i>group box</i>]	Material compressibility correction plot. Displays the results in graph format. The graph plots pressure on a log scale on the x-axis and the volume compressed readings as points on the y-axis, with the theoretical curve based on the calculated values overlaid. Tabular report. Displays the results in table format. The table displays pressure, volume compressed, predicted volume compressed, and error.
	For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.

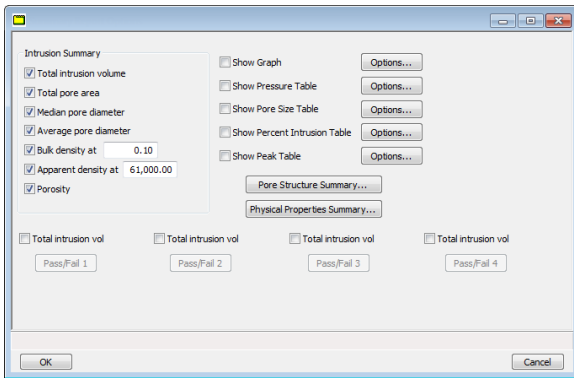
SAMPLE LOG REPORT

This report provides information on:

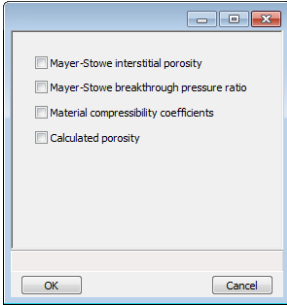
- Manual control operations performed during analysis
- Information entered using *Add Log Entry* on the sample file editor
- Warnings and/or errors which occurred during analysis

SUMMARY REPORT

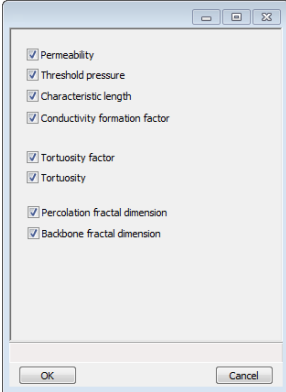
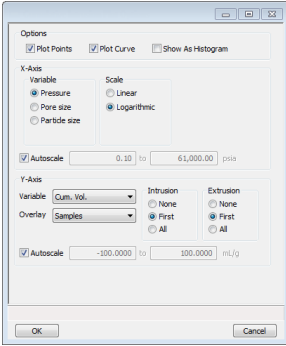
The *Summary Report* provides a condensed listing of selected data results.



Summary Report Fields and Buttons Table

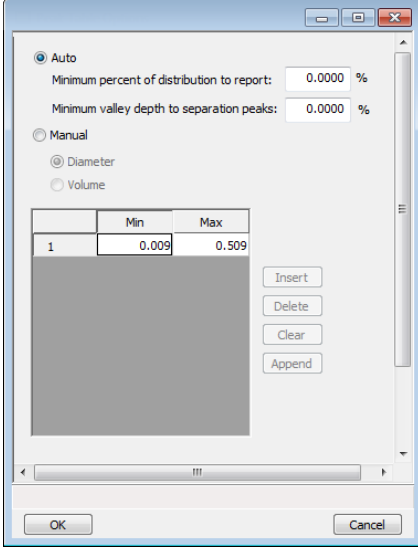
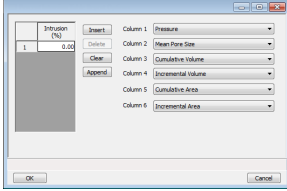
Field or Button	Description
Intrusion Summary [group box]	Select the intrusion options to include in the report. If <i>Bulk Density</i> is selected, enter the pressure for the measurement. If the entered pressure is below the filling pressure, the filling pressure will be used in the report.
Physical Properties Summary [button]	Select the physical properties to display on the report. 

Summary Report Fields and Buttons Table (continued)

Field or Button	Description
Pore Structure Summary <i>[button]</i>	Select the pore structure to be included in the report.  A dialog box titled "Pore Structure Summary" with a list of seven items, each with a checked checkbox: Permeability, Threshold pressure, Characteristic length, Conductivity formation factor, Tortuosity factor, Tortuosity, Percolation fractal dimension, and Backbone fractal dimension. At the bottom are "OK" and "Cancel" buttons.
Show Graph <i>[checkbox]</i>	Displays the report in graph format. Click Options to select how the graph should display.  A dialog box titled "Options" for graph display. It includes checkboxes for "Plot Points", "Plot Curve", and "Show As Histogram". Under "X-Axis", there are radio buttons for "Pressure", "Pore size", and "Particle size", and a "Scale" section with radio buttons for "Linear" and "Logarithmic". There are "Autoscale" checkboxes and input fields for both X and Y axes. The Y-axis section includes dropdowns for "Variable" (set to "Cum. Vol."), "Overlay" (set to "Samples"), and radio buttons for "Intrusion" and "Extrusion" (both with "None", "First", and "All" options).

See [Report Options on page 4 - 10](#) for a description of fields and buttons on this window.

Summary Report Fields and Buttons Table (continued)

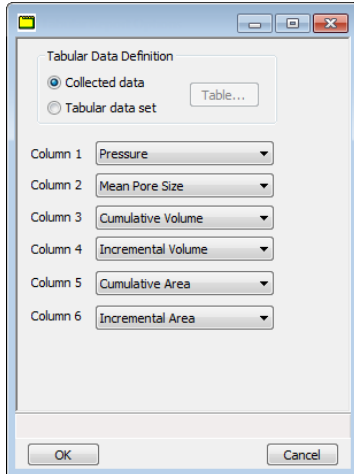
Field or Button	Description
<p>Show Peak Table [check box]</p>	<p>Displays the peak table in the report. Click Options to select how the graph should display.</p>  <p>Auto. Select to have the system automatically identify peaks based on the entered minimum valley depth to separation peaks. Enter the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

Summary Report Fields and Buttons Table (continued)

Field or Button	Description
Show Pore Size Table [check box]	Select to show pore size in the report. Click Options to select points to display. Use the drop-down fields to specify the data to appear in the specified columns for report generation.
Show Pressure Table [check box]	Select to display pressure points in the report. Click Options to select points to display. Use the drop-down fields to specify the data to appear in the specified columns for report generation.
Total intrusion volume [check box]	Enables the Pass/Fail button. <p>Upper / Lower. Specify upper and lower limits for the selected parameter. A range can be left open by not selecting the limit. In the text box to the right of <i>Upper / Lower</i>, enter operator instructions to be displayed if a failure is encountered.</p>
For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3 .	

TABULAR REPORT OPTIONS

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

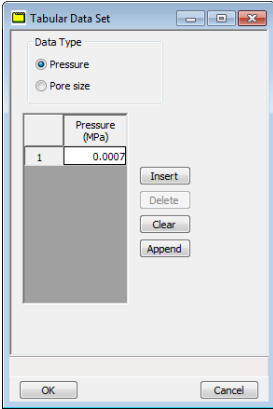



In the *Tabular Data Definition* group box, indicate select either *Collected data* or *Tabular data set* for this report.

Tabular Report Options Fields and Buttons Table

Field or Button	Description
Column [n] [drop-down box]	Use the drop-down fields to specify the data to appear in the specified columns for report generation.
Tabular Data Definition [group box]	<p>Collected data. Select to use data points collected during analysis. Data are collected at equilibration points on or about the pressure points specified in the pressure table used for each analysis.</p> <p>Tabular data set. Select to have a table of specific pressure points or pore sizes included in tabular reports. Allows for the comparison of data from various runs, because it interpolates values from each sample run at the points specified in the table. When this option is selected, the Table button is enabled.</p>

Tabular Report Options Fields and Buttons Table (continued)

Field or Button	Description
	 <ul style="list-style-type: none"> • Data Type. Select either <i>Pressure Points</i> or <i>Pore sizes</i> as the data type. • Click Insert to insert a data point immediately before the selected point. To complete the table quickly, enter the highest value in the set, then click Insert to enter points below that value.
	<p>For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.</p>

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8 DIAGNOSTICS

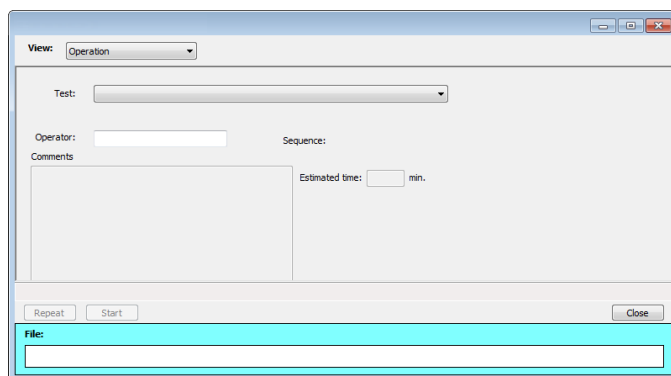
Unit [n] > Diagnostics

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

START DIAGNOSTIC TEST

Unit [n] > Diagnostics > Start Diagnostic Test

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



Start Diagnostic Test 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.
Next [button]	Starts the next test.
Operator [text box]	Enter information to identify the person running the service test.
Repeat [button]	Repeats the selected diagnostic test.
Report after test [check box]	Automatically generates reports to the selected destination when the test is complete.
Sequence	Sequence number assigned to the test.
Start [button]	Starts the diagnostic test.

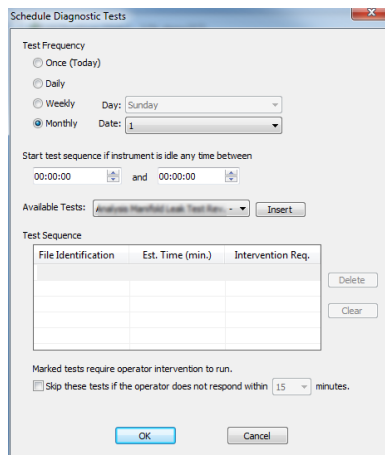
Start Diagnostic Test Fields and Buttons Table (continued)

Field or Button	Description
Test [<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 - 3 .	

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.



Schedule Diagnostics Test Frequency Fields and Buttons Table

Field or Button	Description
Available Tests [<i>drop-down box</i>]	Select one or more tests to run unattended.
Insert [<i>button</i>]	Inserts the selected test in the <i>Available Tests</i> drop-down list.
Skip these tests if the operator does not respond within [n] minutes [<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.

Schedule Diagnostics Test Frequency Fields and Buttons Table (continued)

Field or Button	Description
Start test sequence if instrument is idle any time between 00:00:00 and 00:00:00 [<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.
Test Frequency [<i>selection</i>]	Select how often the test is to run unattended.
Test Sequence [<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 Delete.</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 Insert.</p>
 <p>For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.</p>	

DIAGNOSTIC TEST REPORT

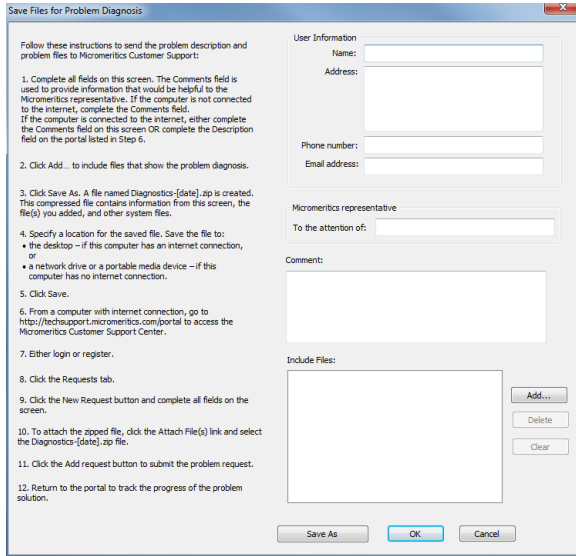
Unit [n] > Diagnostics > Diagnostic Test Report

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.




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, log in to the [customer portal](#).
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
Comment [text box]	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
Include Files	<p>Add. Click to select additional files to send with this problem diagnosis.</p> <p>Delete. Select the file in the <i>Include Files</i> box, then click Delete to remove the file from the list.</p> <p>Clear. Click to clear all files from the <i>Include Files</i> box.</p>
Save As [<i>button</i>]	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.
Micromeritics representative [<i>text box</i>]	Enter the name of your Micromeritics representative. This information will remain on the window each time files for problem diagnosis need to be submitted (can be modified as necessary).
User Information [<i>text box</i>]	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).
<div style="border: 1px solid green; padding: 5px;">  <p>For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.</p> </div>	

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9 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.

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**.

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

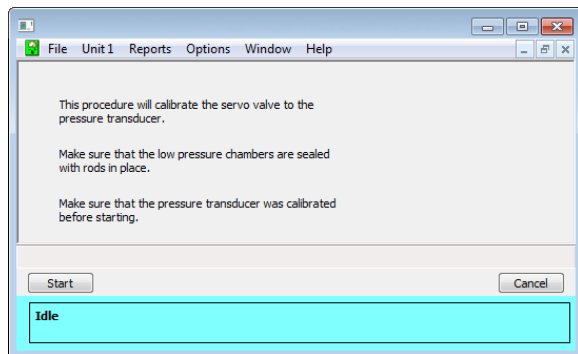
CALIBRATE THE LOW PRESSURE SERVO

Unit [n] > Calibration > Low Pressure Servo



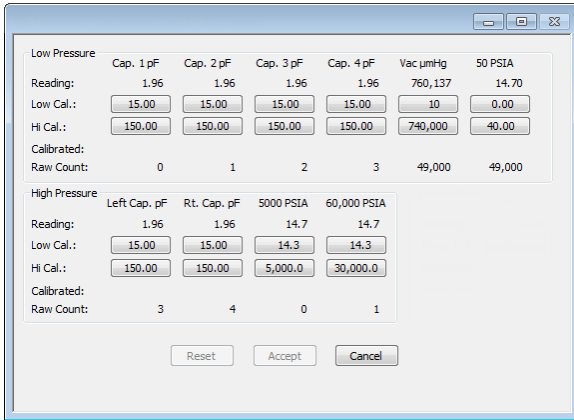
Ensure the pressure transducer was calibrated by a service technician before starting this process.

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



CALIBRATE SIGNALS

Unit [n] > Calibration > Signals




During calibration, the capacitance detectors, vacuum gauge, reference gauge, transducer, etc. are read. Analysis cannot be started while the calibration window is open.

To manually enter calibration data, click the button containing the value to be changed and enter the data on the popup window.

Calibration Signals Fields and Buttons Table

Field or Button	Description
Low Pressure	<p>Reading. Displays the current values from each capacitance detector.</p> <p>Low Cal. Displays the capacitance of the reference device with the lower value.</p> <p>Hi Cal. Displays the capacitance of the reference device with the higher value.</p> <p>Calibrated. The date the capacitance detector was last calibrated.</p> <p>Raw Count. The digital value produced by the analyzer for each capacitance detector.</p> <p>Vac µmHg.</p> <ul style="list-style-type: none"> • Reading. The current value from the vacuum gauge. • Low Cal. The pressure value read on a reference vacuum gauge after vacuum is achieved. • High Cal. The pressure value read on a reference vacuum gauge when higher pressure is applied.

Calibration Signals Fields and Buttons Table (continued)

Field or Button	Description
	<ul style="list-style-type: none"> • Calibrated. The date the vacuum gauge was last calibrated. • Raw Count. The digital value produced by the analyzer for the vacuum gauge. <p>50 PSIA</p> <ul style="list-style-type: none"> • Reading. The current value from the 50 psia transducer. • Low Cal. The pressure value read on a reference transducer after vacuum is achieved. • High Cal. The pressure value read on a reference transducer when higher pressure is applied. • Calibrated. The date the 50 psia transducer was last calibrated. • Raw Count. The digital value produced by the analyzer for the 50 psia transducer .
High Pressure	<p>Reading. Displays the current values for the left and right capacitance detectors.</p> <p>Low Cal. Displays the left and right capacitance detectors of the reference device with the lower value.</p> <p>Hi Cal. Displays the capacitance of the left and right capacitance detectors with the higher value.</p> <p>Calibrated. The date the left and right capacitance detectors were last calibrated.</p> <p>Raw Count. The digital value produced by the analyzer for left and right capacitance detectors.</p>
	<p>For fields and buttons not listed in this table, see Common Fields and Buttons on page 2 - 3.</p>

CALIBRATE THE PENETROMETER

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

The analyzer can determine sample density, or planning to use blank correction by formula), the calibrated empty volume of each penetrometer must be entered in the penetrometer properties file. The file can then be selected and used for analyses.

It is not necessary to determine a penetrometer's volume more than once, if the penetrometer and seal are kept together and a record is maintained.

To calibrate a penetrometer, perform a low pressure run with no sample. This fills the penetrometer with mercury, so that its volume can be determined.

1. Create a sample file. A sample file must be created to run a low pressure analysis, even though no sample is in place.
2. Go to **File > Open > [SMP]** file. Enter the file name to be used for the calibration. If planning to use blank correction files, see [Combine a Penetrometer Calibration and Blank Correction File on page 3 - 7](#). Follow the prompts to create and save the new file.
3. When creating the sample file:

On the *Sample Description* tab:

- select the *Penetrometer Properties* file that corresponds to the size and type of penetrometer to be calibrated, and
- enter 0 in the *Sample mass* field.

On the *Analysis Conditions* tab:

- edit the pressure table, if necessary, and
 - if this low pressure run will not be used as a part of a blank correction file, enter 1 in the *Filling pressure* field,. Use 1 pressure point at 15 psia.
4. Perform the low pressure analysis (see [Perform a Low Pressure Analysis on page 5 - 11](#)).
 5. Clean the penetrometer (see [Clean the Penetrometer on page 5 - 5](#)) if you do not plan to perform the high pressure analysis and use these data for a blank correction file.
 6. Repeat this process three times, recording values on worksheet. Average the volumes resulting from the low pressure run.
 7. Create a *Penetrometer Properties* file for this penetrometer (see [Penetrometer Properties on page 4 - 9](#)). Enter the calibrated volume in the *volume* field.

LOAD CALIBRATION FROM FILE

Unit [n] > Calibration > Load from File [button]

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 [button]

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

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

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

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10 HARDWARE COMPONENTS AND ACCESSORY INSTALLATION

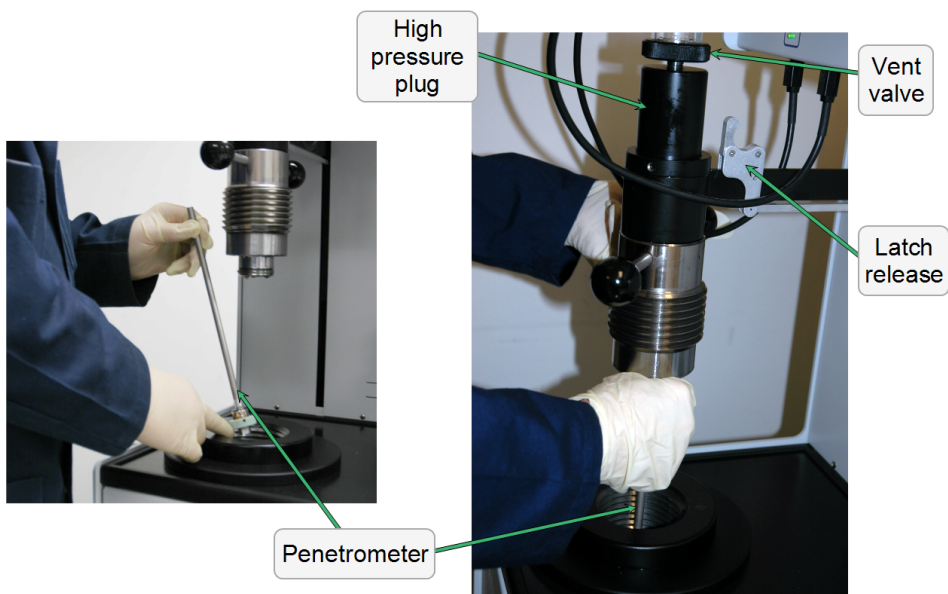


The equipment images in this document may differ slightly from your equipment, however the instructions are the same unless otherwise noted.

INSTALL A PENETROMETER IN A HIGH PRESSURE CHAMBER



Before opening the high pressure chamber, check the status display to ensure the system is not pressurized.



1. Turn the vent valve slowly counter-clockwise to release excess pressure.
2. Unscrew the chamber plug assembly by turning the arms counter-clockwise. Lift the chamber plug assembly as far as it will go. The chamber assembly contains a latching device which automatically locks into place when the assembly is in the topmost position.
3. Gently lower the penetrometer assembly, bulb down, into the chamber until it rests firmly on the banana plug.
4. Verify that the oil in the high pressure chamber just covers the ledge inside the chamber. Add more oil, if needed. See [Seal the High Pressure Chamber on page 11 - 19](#) and [Maintain High Pressure Fluid Level on page 11 - 18](#).

5. Pull the latch release down to unlock the chamber plug assembly. Gently lower the assembly while ensuring that the penetrometer is inserted into the assembly hole. After making sure the penetrometer goes into the hole, lower the assembly the rest of the way and screw the chamber plug in.
6. After the chamber plug is screwed, check the fluid level in the plastic cup above the vent valve to ensure all the air bubbles are out of the chamber, and sealing the vent valve. See [Seal the High Pressure Chamber on page 11 - 19.](#)

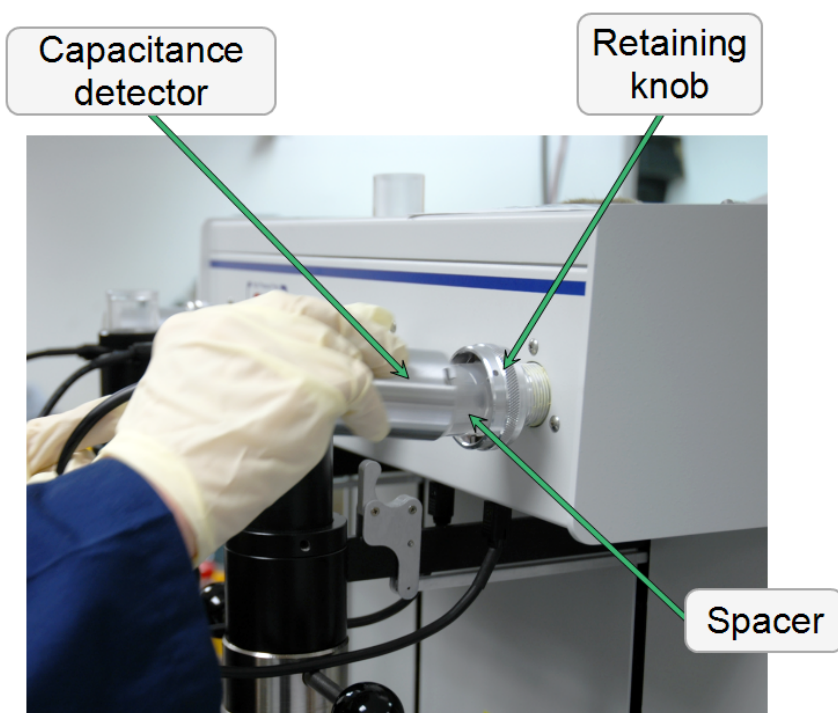
INSTALL A PENETROMETER IN A LOW PRESSURE PORT

If fewer than four samples are to be analyzed, a blank rod must be installed in each unused low pressure port.



If a penetrometer or blank rod is not installed in each port, vacuum conditions cannot be achieved and an analysis cannot be performed.

1. Remove the capacitance detector from the low pressure port by turning it counter-clockwise and pulling forward. Place it on top of the analyzer or stand it on the surface next to the high pressure chamber.



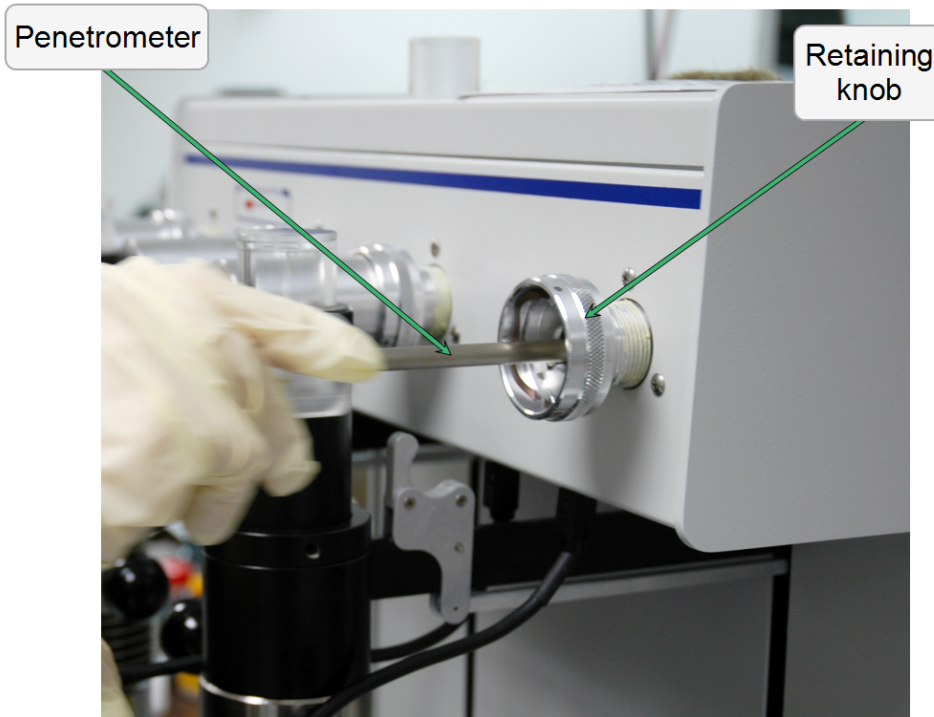
2. Turn the retaining knob counter-clockwise until it turns with little resistance.



Do not remove the knob; internal components may become misaligned.

3. If the low pressure port contains a blank rod, remove it. If it contains a penetrometer, see [Remove a Penetrometer from a Low Pressure Port on page 10-6](#).

4. If using a 3 cc penetrometer, install the spacer over the penetrometer stem. Insert the penetrometer stem into the port and push it in as far as it will go. The spacer is required for the 3 cc penetrometer only.



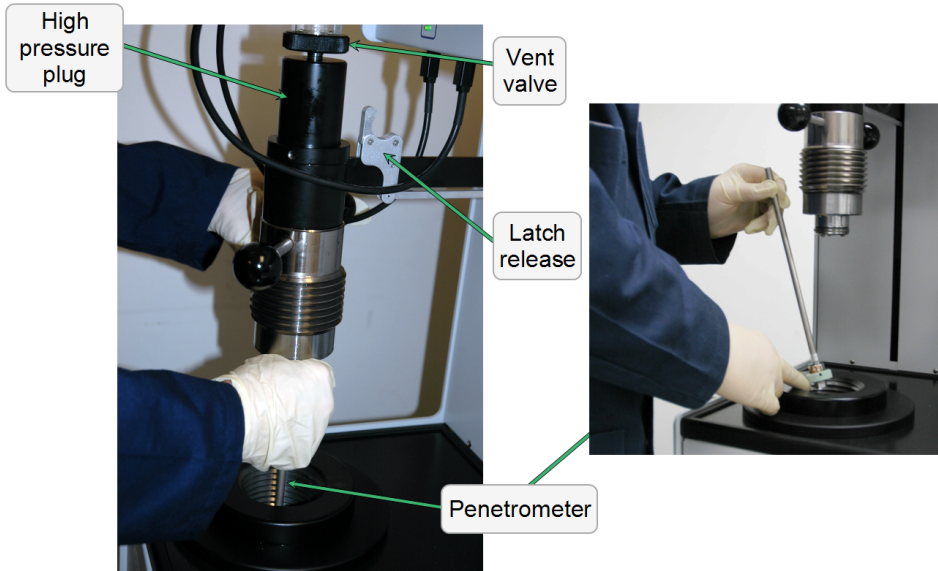
5. Tighten the retaining knob by turning it clockwise until the penetrometer is firmly seated. Do not tighten with excessive force.
6. Install the capacitance detector over the penetrometer and turn clockwise to tighten.

REMOVE A PENETROMETER FROM A HIGH PRESSURE CHAMBER



When analysis is finished, ensure the high pressure system indicator *PRESSURIZED* is not lit.

1. Loosen the vent valve counter-clockwise approximately 1/8 turn to make the plug removal easier.



2. Unscrew the chamber plug by turning the arms counter-clockwise. Lift the plug assembly as far as it will go. Fluid begins to drain from the vent valve. Pause a few seconds to allow fluid to drain back into the chamber.
3. Remove the penetrometer assembly. Hold it over the chamber for a few moments to allow the high pressure fluid to drain.
4. Clean the penetrometer. See [Clean the Penetrometer on page 5 - 5](#).

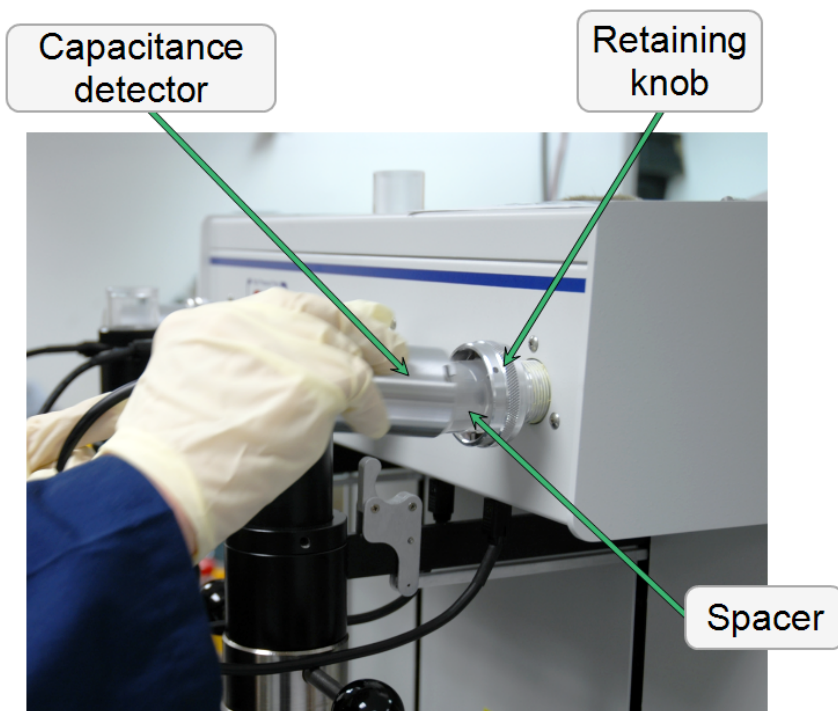
REMOVE A PENETROMETER FROM A LOW PRESSURE PORT

1. Ensure the low pressure ports have returned to near atmospheric pressure and the *Hg Drained* indicator on the front analyzer panel is illuminated.



Removing penetrometers when the *Hg Drained* indicator is not illuminated may allow mercury to spill from the port. See [Drain the Low Pressure System on page 11 - 10](#).

2. Hold the retaining knob to prevent rotation, then turn the capacitance detector counter-clockwise to loosen and remove it.



3. Turn the retaining knob counter-clockwise, then carefully withdraw the penetrometer assembly. Do not pull on the penetrometer cap.



As the penetrometer is withdrawn, tilt the bulb end down and the stem end up, so mercury does not spill from the open stem end.

4. Remove the penetrometer spacer if used.



If the assembly is not to be placed immediately in the high pressure chamber, store it with the stem upward so that none of the mercury will be spilled. The rack for full penetrometers (part number 962-25827-00) can be used for this purpose.

5. Weigh the penetrometer assembly (remove the spacer first) if density calculations are to be made or blank correction by formula is used. Record this mass on the *Sample Data Worksheet*.



Do not wait an extended period of time before performing the high pressure run. Mercury remaining in contact with the sample for long periods of time after the low pressure analysis may oxidize and reduce the reproducibility of results.

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11 MAINTENANCE AND TROUBLESHOOTING



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

The analyzer has been designed to provide efficient and continuous service; however, certain maintenance procedures should be followed to obtain the best results over the longest period of time. When unexpected results occur, some common operational problems not indicated on the window and their respective causes and solutions are provided:

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

Status display is too large for computer monitor.

Cause: Monitor resolution is set below 800 × 600.

Action: Refer to the computer operating system manual to reset the monitor resolution at or above 800 × 600.

Difficulty attaining adequate vacuum conditions during low pressure analyses.

Cause A: Vacuum pump oil is low.

Action A: Add vacuum pump oil.

Cause B: Vacuum hose not properly connected.

Action B: Ensure the connection to the vacuum pump is good and that external clamps are tight.

Cause C: Sample contains excess moisture adsorbed from atmosphere.

Action C: Prepare samples (prior to loading penetrometer) by heat and/or vacuum to remove moisture.



Never heat a sample that has contacted mercury.

Cause D: Proper sealing not achieved on penetrometers

Action D:

1. Use blank rods to test the ports to eliminate the possibility of having a leaking penetrometer cap / bulb seal.
2. Lightly grease the rods with a high grade vacuum grease, then insert the rods in the low pressure ports.

3. Tighten the retaining knobs on the low pressure ports. If vacuum conditions are satisfactory, check the penetrometer for scratches or chips in the bulb.

Cause E: Scratches or other imperfections in either the lip of the penetrometer bulb or the penetrometer cap.

- Action E:*
1. Polish the lip of the bulb and the cap using 600 grit emery paper or crocus cloth.
 2. Place the paper or cloth on a flat surface, grit-side up.
 3. Clean all grease from the surface to be polished with solvent.
 4. Lightly press the surface down on the grit and rub in a circular motion. A minute of this polishing action is usually sufficient, but it must be continued until the surface is free from flaws. Examination of the surface under low magnification (approximately 20X) helps determine when the surface is free from flaws.
 5. Clean the ground surface before regreasing.

Cause F: Leaking valves.

Action F: Check the valves for leaks. See [Check Valves for Leaks on page 11 - 29](#).

Cause G: Moisture has accumulated in the system.

Action G: Remove the accumulated moisture. See [Remove Moisture from the Analyzer on page 11 - 31](#).

The low pressure analysis is complete and the system has returned to near atmosphere, but the mercury drained indicator is not illuminated.

Cause A: Indicator is not working properly.

Action A: Verify the state of the mercury degasser by looking at the status display window. If the status display does not agree with the indicator, the indicator may not be working properly. Contact your Micromeritics service representative.

If the status display window shows that the mercury has overflowed (the alarm state), see [Handling Mercury Overflow on page 11 - 21](#). If the status display shows that the mercury degasser is either filled or partially filled, see [Drain the Low Pressure System on page 11 - 10](#).

Cause B: Mercury is not drained. Status display shows either filled or partially filled.

Action B: See [Drain the Low Pressure System on page 11 - 10](#).

Mercury warning buzzer sounds in pulses. (Mercury overflow).

Cause: Mercury detected by overflow sensor in low pressure port system.

Action: See [Handling Mercury Overflow on page 11 - 21](#).

Mercury warning buzzer sounds with continuous tone.

Cause: Mercury has been improperly drawn into the mercury trap.

Action: Drain excess mercury into the reservoir. See [Handling Mercury Overfill on page 11 - 21](#). Remove the plug extending down from the mercury trap. The plug is reached through the front door. Facing the analyzer, the plug is in the upper back right corner. See [Drain Spilled Mercury Dish on page 11 - 11](#). Position a container beneath the trap before removing the plug.

The high pressure system failed to attain the specified pressure.

Cause: Low fluid level in the hydraulic pump.

Action: Add fluid to the hydraulic pump. See [Maintain Hydraulic Pump Fluid Level on page 11 - 23](#).

High pressure system failed to retain a reasonably constant pressure.

Cause: Leakage around the high pressure chamber cap.

Action: Replace the cup seals and backup rings on the high pressure chamber cap. See [Replace Chamber Cup Seals on page 11 - 21](#).

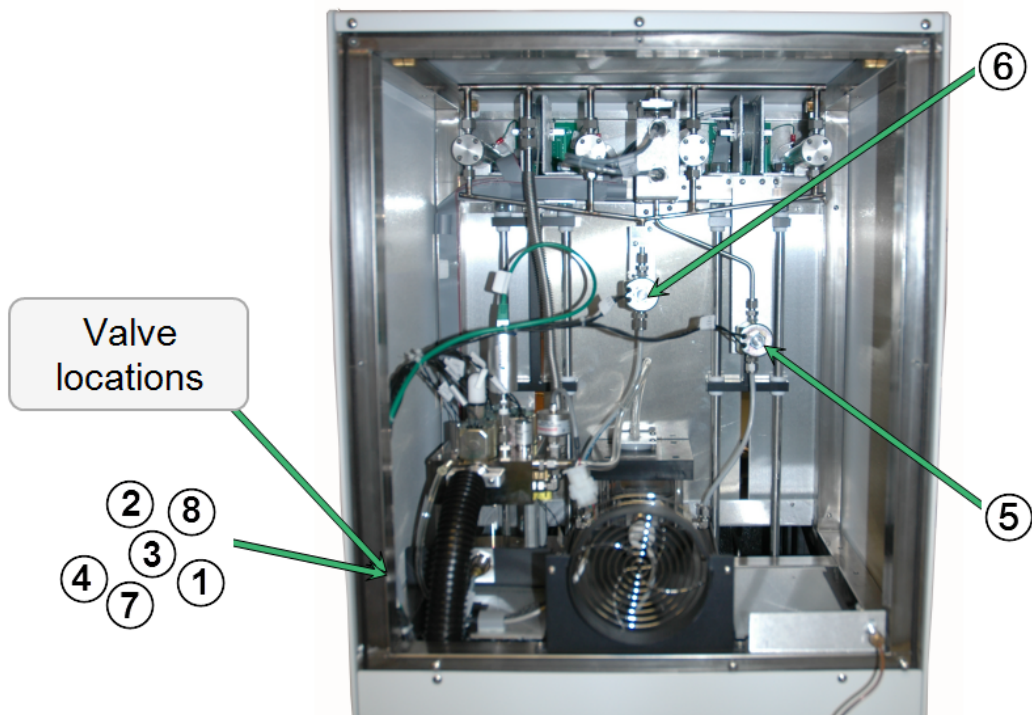
Cannot attain low pressure data points above atmospheric pressure.

Cause: Gas supply pressure too low.

Action: Verify gas supply regulator is set to 40 to 45 psig (276 to 310 kPa) and that there is sufficient gas in the supply bottle.

SYSTEM COMPONENTS

LOW PRESSURE SYSTEM COMPONENTS

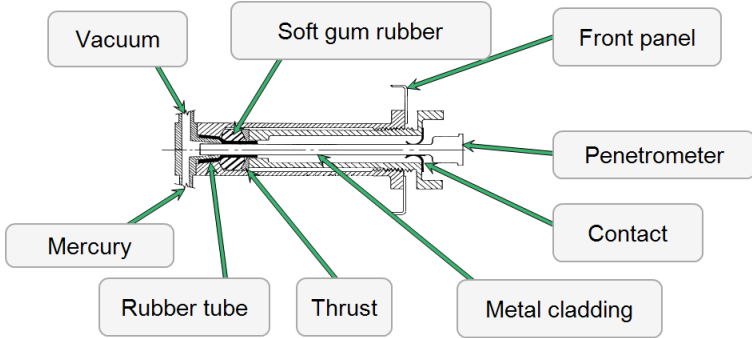


Rear View

Low Pressure System Valves

Item #	Component	Description
1	Servo isolation valve	Ensures no leaking occurs through the servo. It is open when the servo is in use and closed when the servo is off.
2	Fast evacuation valve	Used at the end of the evacuation routine to achieve the best vacuum. When this valve is open, the sample stations are directly connected to the vacuum system.
3	Vacuum valve	<p>The vacuum valve is opened to evacuate the back of the servo valve during the ramping part of the evacuation sequence. This allows the servo valve to control the evacuation rate.</p> <p>When the pressure is low enough, the evacuation through the reservoir proceeds. This is accomplished by alternately opening Valve 8 (to allow gas from the sample to move to the reservoir) and Valve 3 (to evacuate the reservoir for the next step).</p>

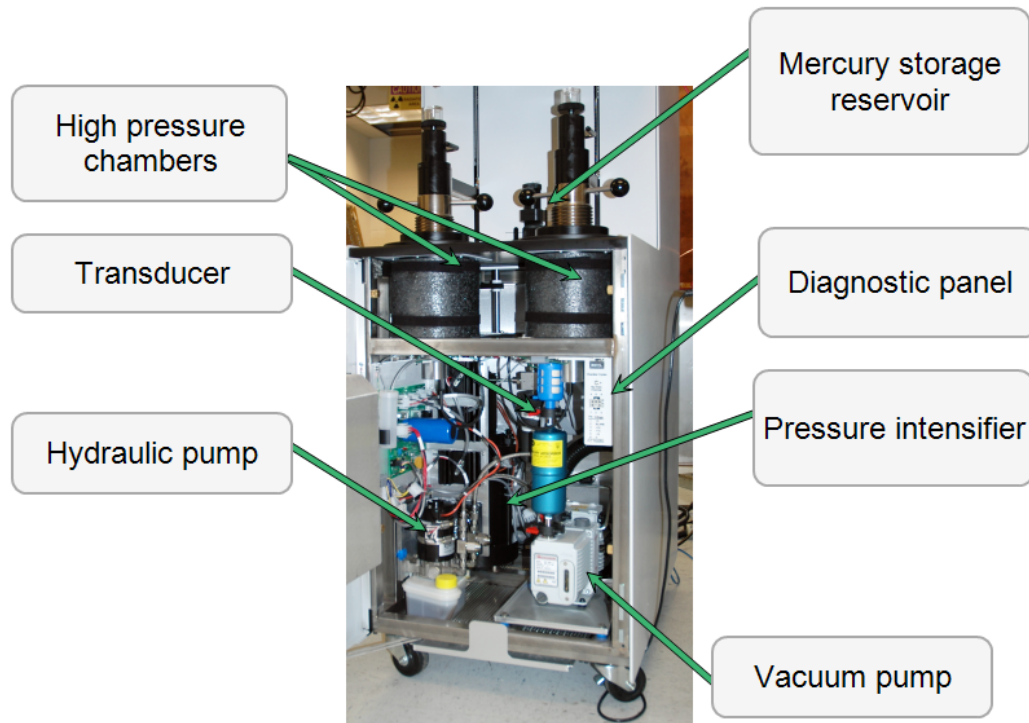
Low Pressure System Valves (continued)

Item #	Component	Description
4	Gas inlet valve	Allows pressurization at the back of the servo valve. The servo can then be used to dose pressures from 0.2 psia to 50 psia onto the low pressure stations. Dry nitrogen or argon is the preferred gas. Do not use air unless it is dried to remove water vapor.
5	Mercury fill valve	Mercury flows into the degasser then into the low pressure stations and is controlled by the fill valve. The drain valve allows mercury to flow back into the reservoir.
6	Drain valves	See Mercury fill valve [5].
7	Mercury reservoir evacuation valve	Connects the vacuum line to the mercury reservoir.
8	Evacuation Reservoir valve	See Vacuum valve [3].
Low pressure ports		<p>A sample encased in a penetrometer is first evacuated, filled with mercury, then pressurized to between 15 and 50 psia.</p>  <p>Penetrometer stems inserted into a station are sealed for vacuum and against mercury leakage by compression of a soft, gum rubber cylinder near the tip. The rubber is compressed by turning the large knob or pressure collar that protrudes from the front. The inner rim of this knob serves also as the mounting mechanism for a capacitance transducer after the knob has been tightened.</p>
Mercury storage reservoir		Holds 7 to 10 pounds of mercury and is located behind the front panel. The level of mercury is visible through a small window in the front panel. Before beginning a low pressure analysis, the application verifies that the level of mercury in the reservoir is adequate.
Mercury degasser		<p>Removes trapped gases as mercury passes to low pressure stations then into the penetrometer.</p> <p>HG Up indicator. Illuminates when mercury level fills the penetrometers. The sensor completes an electric circuit and flow</p>

Low Pressure System Valves (continued)

Item #	Component	Description
		<p>is stopped by closure of the mercury fill solenoid valve.</p> <p>Drained indicator. Illuminates when the mercury drain valve is closed and the level of mercury in the degasser is below the level in the low pressure stations.</p> <p>Alarm buzzer. If the level of mercury rises above the normal filling level, the sensor causes both the mercury fill valve and the mercury drain valve to close and a buzzer alarm sounds. See Handling Mercury Overfill on page 11 - 21.</p>
Mercury trap		<p>Serves as a large volume reservoir for mercury in the event that operator error or a malfunction allows mercury to travel toward the vacuum system. If not stopped, mercury can severely damage the analyzer. Should this occur, a high-pitched, continuous alarm sounds if more than 6 mm (0.25 in.) of mercury accumulates in the trap. The fault must be corrected and the mercury removed before proceeding.</p>

HIGH PRESSURE SYSTEM COMPONENTS

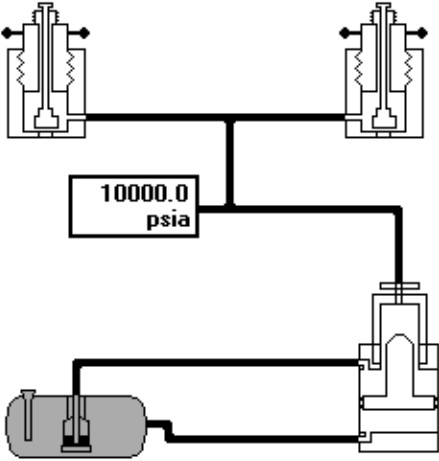


Front View

High Pressure System Components

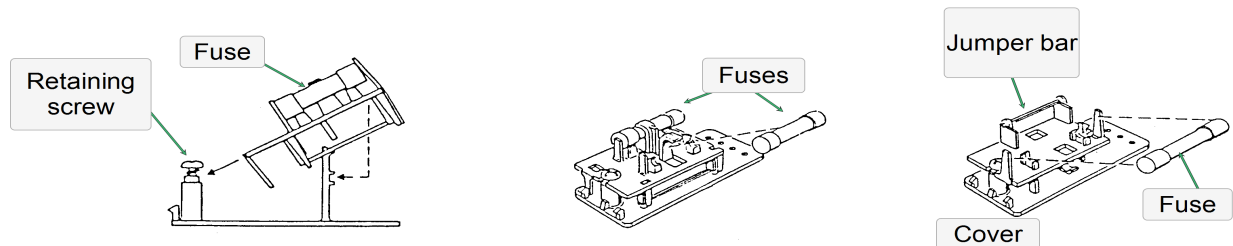
Component	Description
High pressure chamber	The two high-pressure chambers are closed by the chamber plug assemblies. These are sealed into the chambers with large threads and tightened by turning two short handles. The closure components are counter-sprung so that they are readily raised and lowered. On top of each closure is a manual valve for venting or purging air that may be trapped when a high pressure chamber is closed.

High Pressure System Components (continued)

Component	Description
	
Hydraulic pump	<p>The basic high-pressure generating unit is a hydraulic pump. The pump has a hydraulic fluid reservoir and a pressure gauge. The relief valve limits the maximum pressure to 2800 psia. The pump and drive motor rotation are reversible to allow the pressure adjustment.</p> <p>Pump speed and direction are controlled by the application which prevents overshooting of the target pressure while minimizing time required to attain a target pressure.</p>
Pressure intensifier	<p>The required psia is generated by a double- action, dual piston intensifier connected to the hydraulic pump. Two limit switches are located near the upper and lower limits of piston travel. The first of these switches alerts the operating program; the second interrupts the power to the motor.</p>
Transducer	<p>High pressure measurements are made with one pressure transducer. The signal from this transducer is processed by electronic circuitry to yield two pressure output signals.</p>

CHANGE THE FUSES

Fuse the input power line according to local safety practices. The input power connector can be used with either a single-fuse arrangement or a double-fuse arrangement.



The power cord should be disconnected from the analyzer before removing the cover from the power input connector. Failure to disconnect the power cord could result in electrical shock.

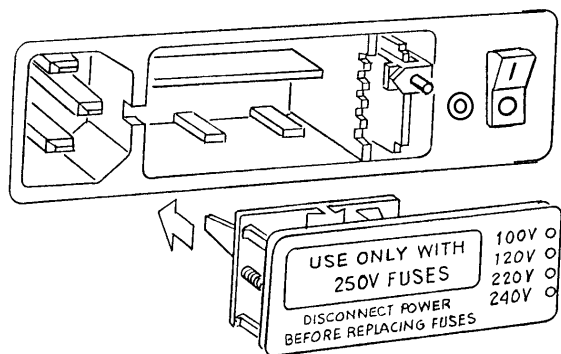
- If the single-fuse arrangement is needed, position the fuse block so that the side with the single-fuse slot and the jumper bar is away from the cover.
- If the double-fuse arrangement is needed, position the fuse block so that the side with the double-fuse slots is away from the cover.
 1. Reposition the fuse block, if necessary:
 - a. Remove the fuse block retaining screw.
 - b. Lift the fuse block from the cover.
 - c. Rotate the fuse block.
 - d. Mount the fuse block to the cover.
 - e. Replace the retaining screw.
 2. Insert appropriate fuse for the input power source. Refer to the chart below for the appropriate fuse rating.



The fuse used in the analyzer must be identical in type and rating to that specified. Use of other fuses could result in electrical shock and/or damage to the unit.

Power Source	Fuse
100-120 VAC	3AG 6.25 Amp Slow-Blow
200-240 VAC	5 x 20 mm 5.0 Amp Slow-Blow

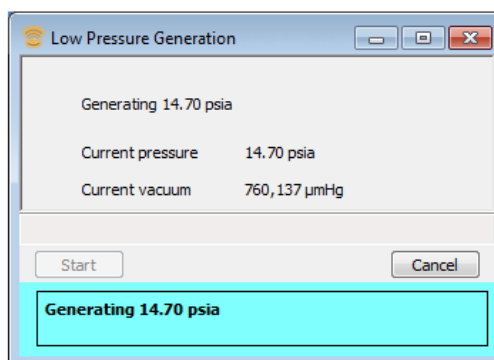
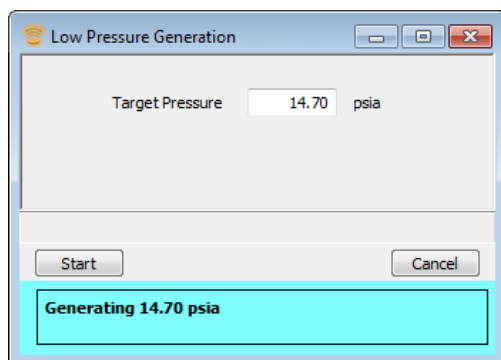
3. Insert fuse block and cover assembly into input power connector and snap it into place. After the fuse block and cover assembly are in place, the position of the indicator pin shows the input power selected.



4. Connect the power cord to the analyzer and plug into an appropriate power source.

DRAIN THE LOW PRESSURE SYSTEM

Unit [n] > Generate Low Pressure



1. Specify a pressure of 10 psia. After the system reaches 10 psia and returns to *Idle* status, open the reservoir evacuation valve (#7) for 20 seconds.

Ensure the fill cap is sealed before proceeding. See [Maintain Mercury Level on page 11 - 17](#).

2. Open the mercury drain valve until the mercury drains.
3. Close the mercury drain valve.
4. Close the mercury reservoir evacuation valve.
5. To return the system to atmosphere, go to ***Unit [n] > Generate Low Pressure*** and enter a pressure of 15 psia.



If the system still indicates that the mercury is not drained (the status display, the mercury drained indicator or both), see [Handling Mercury Overfill on page 11 - 21](#).

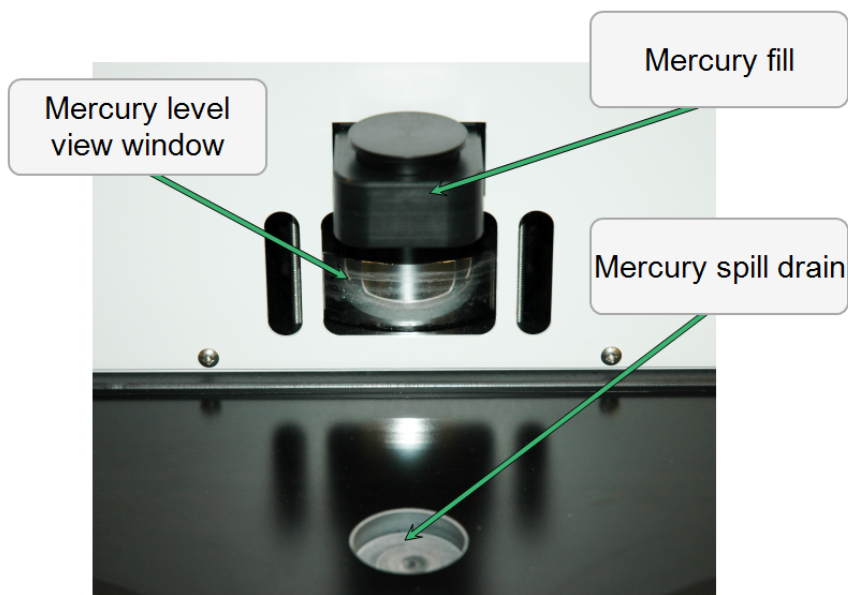
DRAIN SPILLED MERCURY DISH

A dish for collecting mercury is located just behind the high pressure chambers. Pour approximately 1.0 to 2.0 cc of oil into the dish to prevent the mercury from vaporizing.

If mercury accumulates in the dish, remove it by removing the cover and extracting the mercury with the syringe accessory.



Approximately 3 mm (1/8 in.) of oil should remain in the container to prevent the escape of mercury vapors.

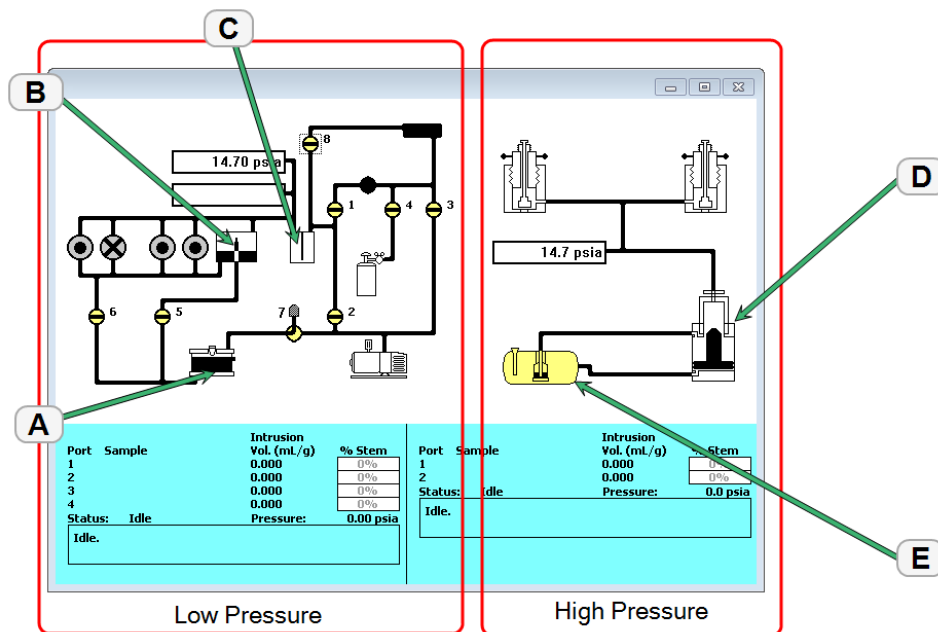


ENABLE MANUAL CONTROL

Unit [n] > Enable Manual Control

Use to enable the manual control of certain system valves and elevator components on the analyzer schematic. When this option is enabled, a checkmark appears to the left of **Unit [n] > Enable Manual Control**.

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







Schematic Components Table

Schematic Components	Description
1	Servo isolation valve
2	Fast evacuation valve
3	Vacuum valve
4	Gas inlet valve
5	Mercury fill
6	Mercury drain
7	Mercury reservoir evacuation
8	Evacuation reservoir




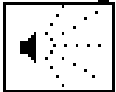
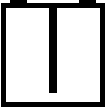
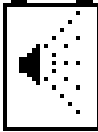


Schematic Components Table (continued)

Schematic Components	Description
A	Mercury reservoir
B	Mercury degasser
C	Mercury trap
D	Intensifier
E	Hydraulic pump
Pressure	<ul style="list-style-type: none"> Displays the current pressure in the system. The pressure is shown in the units selected in Options > Units. The reading shown is from either the vacuum gauge or the 50 psia transducer, depending upon which is currently in range.
Status	Provides the sample file name and sequence number, the intrusion volume, and the percent of the penetrometer stem already used.

Analyzer Schematic Icon Table

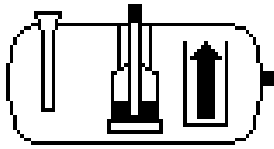
Icon or Symbol	Description
	Open Valve. Green indicates an open valve.
	Closed Valve. Yellow indicates a closed valve.
	Servo Valve. Closed.
	Servo Valve. Open.

Low Pressure Schematic Icons

<p>Mercury Degasser. Displays the mercury level.</p>			
 <p>Drained</p>	 <p>Partially Filled</p>	 <p>Filled</p>	 <p>Overfilled. This alarm displays with a red background. This is an alarm state.</p>
<p>Mercury Trap. Displays the state of the mercury trap.</p>			
 <p>Empty</p>	 <p>Contains more than 6 mm of mercury. This is an alarm state.</p>		
<p>Mercury Reservoir. Displays the level of mercury in the reservoir.</p>			
 <p>If the mercury level is low, see Maintain Mercury Level on page 11 - 17.</p>		 <p>Level is OK.</p>	

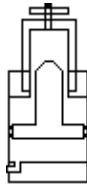
High Pressure Schematic

Hydraulic Pump

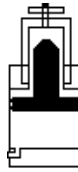


When the pump is operating, the target pressure displays below the icon. A green icon indicates the pump is ON. A yellow icon indicates the pump is OFF. To set the target pressure when the icon is yellow, either double-click the pump icon or right click the icon and select *Set*.

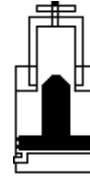
Intensifier. Displays the state of the intensifier limit switches.



Midway



Top



Bottom

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.

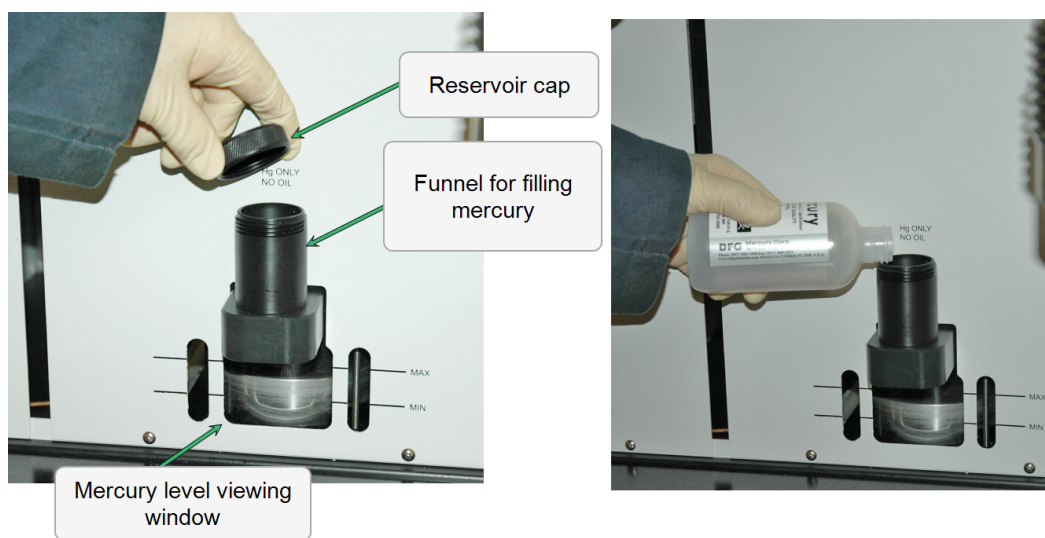
Maintenance Required	Frequency
Banana plug	Check every 600 samples or 3 months. Replace as needed.
Chamber plug seals	Replace every 600 samples or 3 months.
High pressure chambers	Check and clean every 600 samples or 3 months.
High pressure fluid	Check every 600 samples or 3 months.
High pressure fluid level	Check prior to performing a high pressure analysis. Fill as needed.
Hydraulic pump fluid	Check every 6 months. Fill as needed. Change annually.
Low pressure ports	Grease every 600 samples or 3 months.
Mercury dish	Check daily for spills. Service as needed.
Mercury level	Service daily.
Mercury vapor filter	Replace annually.
Moisture in system	Remove as needed.
Vacuum pump exhaust filter	Replace every 600 samples or 3 months.
Vacuum pump fluid	Change every 1200 samples or 3 months.
Vacuum pump fluid level	Check monthly. Fill as needed. Refer to the vacuum pump user manual for instructions.
Valves	Check for leaks and clean as needed.

MAINTAIN MERCURY LEVEL

Each analysis may extract from 3 to 15 mL of mercury from the reservoir depending on the penetrometer and sample size used. When running a low pressure analysis, a pop-up message displays on the *Low Pressure Analysis* window when the level of mercury drops below the minimum level. The mercury level should be within 0.5 to 1.0 in. (1 to 3 cm) below the top of the mercury viewing window. It must never reach above the viewing window. To avoid delays, check the mercury level, adding mercury when necessary, at the beginning of each day.



Ensure the use of triple distilled mercury that is at least 99.999% pure.



9600 Model Shown

The analyzer requires approximately 5 lbs of mercury (minimum) to begin analyses. Do not use more than 10 lbs.

A spilled-mercury container, located in the center of the analyzer work surface behind the high pressure chambers, is provided so that any accidentally spilled mercury can be immediately brushed into it. Pour approximately 1.0 to 2.0 cc of oil into the container to prevent the mercury from vaporizing.

1. Remove the mercury reservoir cap.
2. Slowly pour the mercury into the reservoir to within 0.5 to 1.0 in. (1 to 3 cm) from the top of the viewing window.
3. Replace the reservoir cap.



Overfilling may interfere with the vacuum system. Reservoir cap should be finger tight only.



Ensure that the mercury filling funnel is tightly secured. If it is necessary to tighten the funnel, use an appropriate tool. If the funnel is not tightened, there could be a gas leak and the vacuum will not pull properly.

MAINTAIN HIGH PRESSURE FLUID LEVEL

The high pressure fluid level in the high pressure chamber should be checked when preparing for a high pressure analysis. The fluid level should be up to the ledge when a penetrometer is installed. Add high pressure fluid as needed.



9600 Model Shown

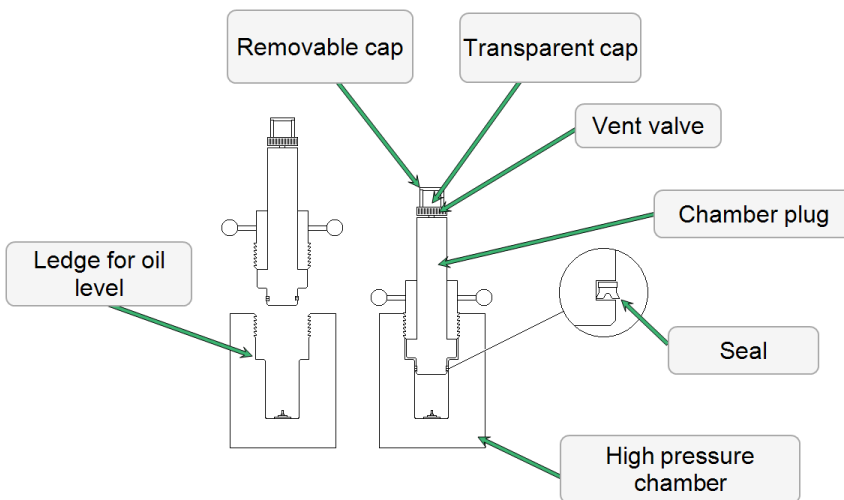
SEAL THE HIGH PRESSURE CHAMBER



High pressure fluid leaking past the chamber seal indicates one of three problems:

- Too much fluid. Remove fluid, clean the seal and try again.
- Damaged seal. Replace the seal, then try again.
- Fluid in the threads. Clean and reseal.

The chamber plug seals the high pressure chamber. Proper sealing does not require the use of excessive force. The outside diameter of the elastic seal on the plug is slightly larger than the inner diameter of the pressure chamber. Lowering the chamber plug into the pressure chamber presses the seal against the chamber wall, sealing the chamber. Then, as pressure increases during an analysis, the outer lip of the seal is forced more tightly against the chamber wall, preventing leakage.



1. Push the plug into the chamber until it contacts the chamber shoulder. Several threads of the plug will remain exposed.
2. Ensure the vent valve is partially open (unscrewed). Slowly turn the plug clockwise into the chamber to force air from the chamber. Continue turning until high pressure fluid (or air bubbles and fluid) appears in the transparent cup on top of the vent valve.



Tightening the plug too quickly may cause unwanted intrusion caused by pressure created when the chamber is closed.

3. The cup should not be completely full. If the cup is too full, slowly open the chamber and recheck the fluid level. Fluid may need to be removed. See [Maintain High Pressure Fluid Level on the previous page](#) for instructions on how to remove excessive fluid. Repeat the previous steps to release trapped air.

- Large air bubbles may be caught in the chamber. Slowly loosen and tighten the plug approximately 1/2 turn several times. This should cause any air bubbles to rise through the transparent cup to the surface. Any remaining tiny bubbles will not affect the analysis.
- If no fluid is visible in the cup when the plug is fully tightened, open the chamber and recheck the fluid level. It is likely that fluid may need to be added. Repeat the previous steps to release trapped air.



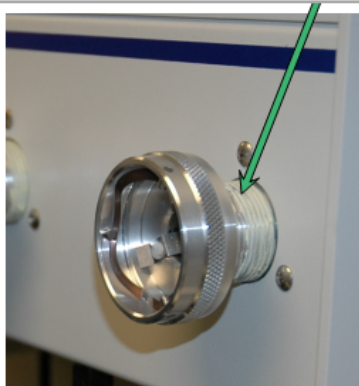
Small amounts of high pressure fluid can be added to or removed from the cup on top of the vent valve without opening the chamber. To do so, remove the cap and use appropriate tools, such as a syringe.

GREASE LOW PRESSURE PORTS

The screw threads visible behind each low pressure port retaining knob should be greased monthly.

1. Remove the capacitance detectors by turning counter-clockwise.
2. Remove the plastic caps.
3. Unscrew the retaining knobs until the threads disengage.

Turn retaining knob counter-clockwise to expose threads behind knob



4. Lubricate the retaining knob threads and mating surfaces with a medium consistency grease such as white lithium grease.
5. Screw the retaining knobs back into the low pressure ports.
6. Replace the plastic caps and capacitance detectors.

HANDLING MERCURY OVERFILL

1. On the Manual Control window, open the reservoir evacuation valve. Evacuate for twenty seconds.

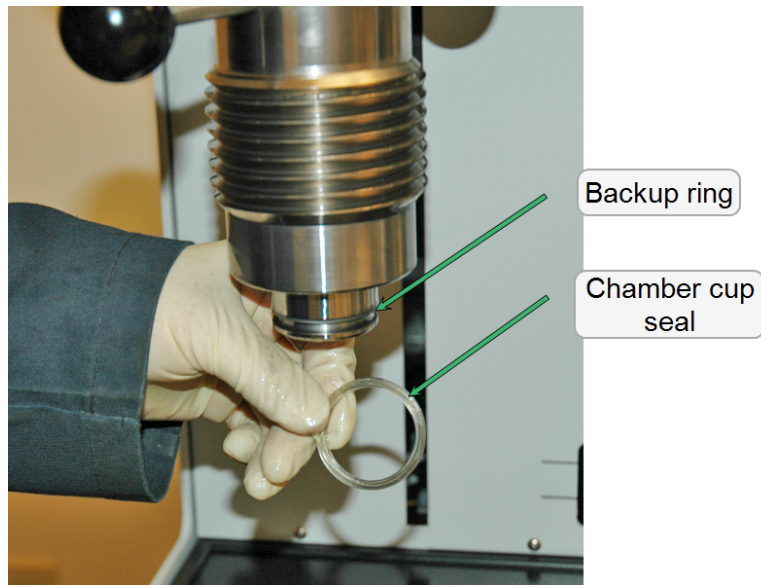
The *Hg reservoir evacuated* indicator should be displayed on the analyzer schematic. If it is not shown, do not proceed to the next step. Instead, verify that the mercury reservoir fill cap is properly sealed on top of the mercury filling funnel (see [Maintain Mercury Level on page 11 - 17](#)). If the *Hg reservoir evacuated* indicator still is not displayed, contact a Micromeritics Service Representative.

2. Open the gas inlet valve. Close the gas inlet valve when at least 15 psia or atmosphere is attained.
3. Open the mercury drain valve.
4. Open the front panel of the analyzer and locate the **Override** switch on the small control panel.
5. Press the switch. This allows mercury to drain back into the reservoir.
6. Release the switch as soon as the pulsed buzzing stops. Close the mercury drain valve.
7. Close the reservoir evacuation valve. If the pulsing tone remains, call the Micromeritics Service Department. The analyzer may be powered off.

REPLACE CHAMBER CUP SEALS

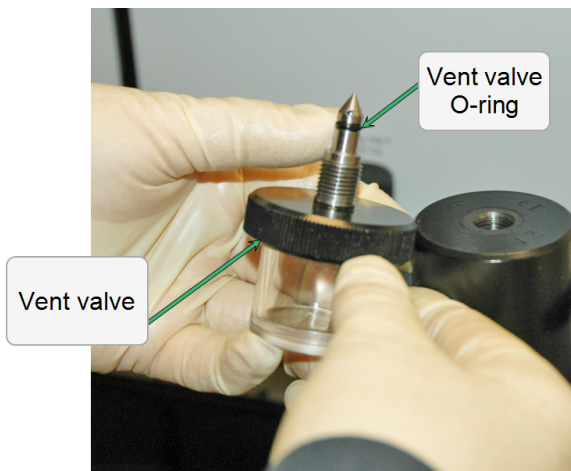
The chamber plug is sealed into the high pressure chamber by means of a cup seal and a backup ring. There is also an O-ring in the vent valve. See [Replace the Vent Valve O-ring on the next page](#). These rings should be checked monthly and replaced when the backup ring extrudes, small slivers come from the cup seal or backup ring, or pinched areas appear on the cup seal. The rings may also need replacing when oil appears around the top of the chamber or when it is difficult to maintain pressure in the high pressure chamber

1. Remove the chamber cup seal and backup ring without scratching the surrounding surfaces.



2. Replace the backup ring on the lower portion of the chamber plug with the backup ring uppermost and with the groove of the cup seal downward.
3. Replace the O-ring in the vent valve.

REPLACE THE VENT VALVE O-RING



1. Remove the vent valve from the top of the high pressure chamber by turning the vent valve counter-clockwise.
2. Gently remove the O-ring on the vent valve and replace with a new one.

CHANGE HIGH PRESSURE FLUID AND CLEAN HIGH PRESSURE CHAMBER

The high pressure fluid should be changed after every 600 samples or 3 months to ensure accurate results. It should also be changed if mercury is spilled into a high pressure chamber. Small drops of mercury in the bottom of the chamber can cause erroneous results.

1. Remove the high pressure fluid from the high pressure chambers using the syringe provided.
2. Clean the high pressure chambers using a clean cloth dampened with IPA.
3. When the chambers are completely dry, add new high pressure fluid.

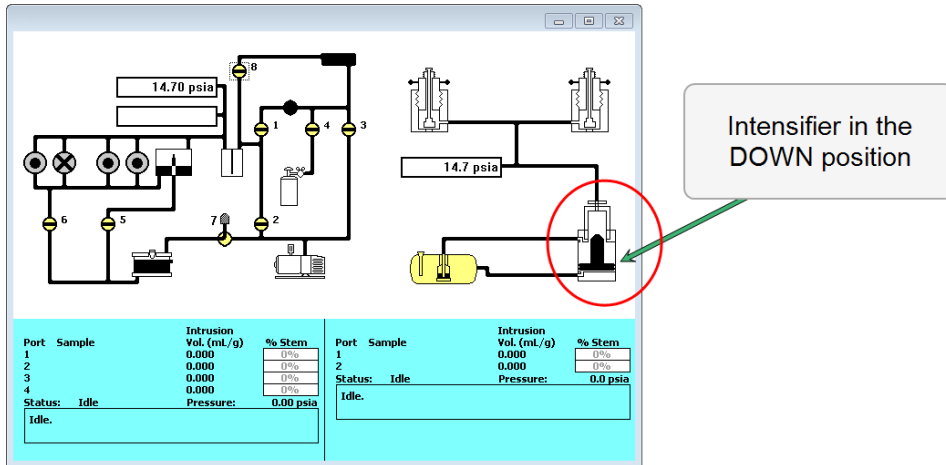
MAINTAIN HYDRAULIC PUMP FLUID LEVEL

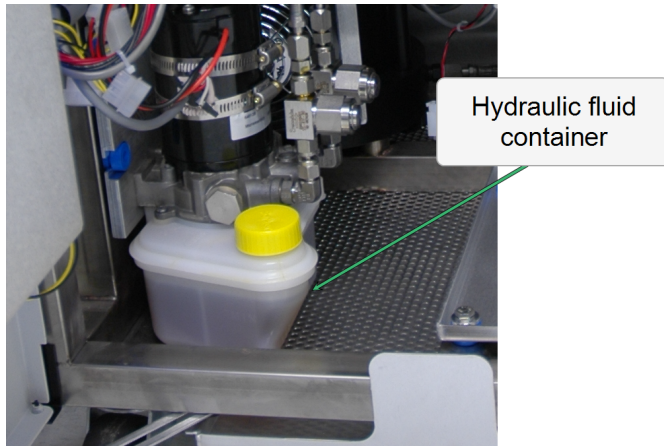


When changing the hydraulic pump fluid, use only Mobil-1 50W-50 Synthetic Motor Oil (Micromeritics part number 004-16011-00).

The hydraulic pump fluid container is located in the lower left corner inside the analyzer's front door.

Check the fluid level when the intensifier on the schematic is in the down position. Add fluid as necessary to keep the fluid level near the top of the container.





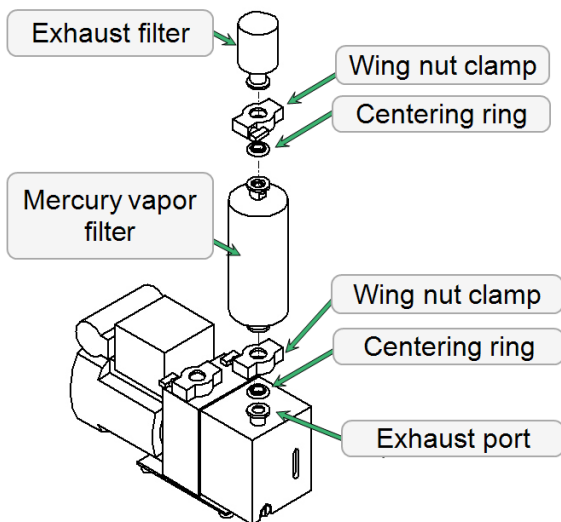
Filling the fluid container when the intensifier is in the up position can cause the fluid to overflow when the intensifier is lowered.

To add fluid to the container, remove the cap to the fluid container and add fluid as necessary. Replace the cap when done.

VACUUM PUMP

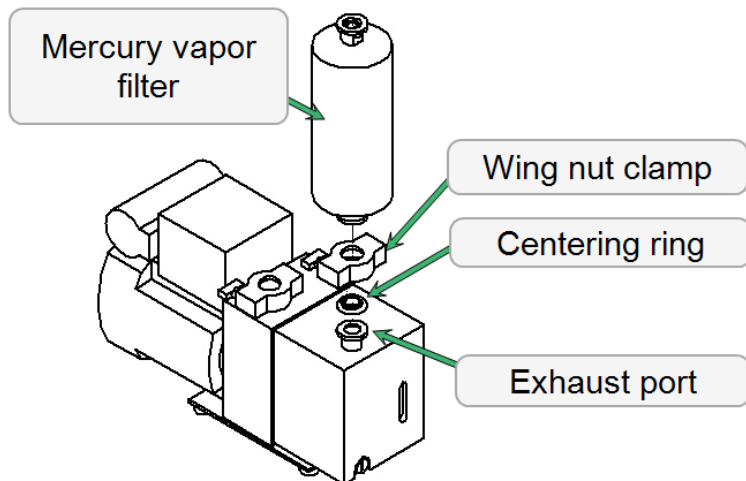
REPLACE VACUUM PUMP EXHAUST FILTER

The exhaust filter is attached to the mercury vapor filter on the vacuum pump.



1. Loosen the wing nut that secures the clamp between the exhaust filter and the mercury vapor filter by turning the nut counterclockwise. Open the clamp and remove it, the centering ring, and the exhaust filter.
2. Inspect the centering ring for wear and replace if necessary.
3. Align the centering ring between the replacement exhaust filter and the mercury vapor filter.
4. Open the clamp and place it around the lip of the exhaust filter, mercury vapor filter, and centering ring.
5. Close the clamp and secure it by turning the wing nut clockwise. Tight the wing nut finger tight.

REPLACE THE MERCURY VAPOR FILTER



Mercury vapor filters are used on vacuum pumps to minimize the release of mercury vapors.



The mercury vapor filter should be replaced annually when preventive maintenance is performed. When the mercury exhaust filter is replaced, go to **Unit [n] > Show Dashboard**. Right click the *Days until Hg filter replacement is due* box and click **Reset**. The counter resets to 365 days until the next filter change is due. Service Mode and password are required to make this change.

1. Loosen the wing nut that secures the clamp between the exhaust port and the mercury vapor filter by turning the nut counter-clockwise.
2. Open the clamp and remove it.
 - a. Remove the protective cover if this is an initial installation.
 - b. If this is not an initial installation, remove and inspect the centering ring for wear and replace if necessary.
3. Align the centering ring between the mercury vapor filter and the exhaust port.
4. Open the clamp and place it around the lip of the exhaust port, mercury vapor filter, and centering ring.
5. Close the clamp and secure it by turning the wing nut clockwise. Tighten the wing nut finger tight.

CHANGE OR ADD VACUUM PUMP OIL

Drain the oil while the pump is warm and disconnected from the power source.

The pump must be removed from the analyzer to change the oil. Use oil supplied by Micromeritics or refer to the vacuum pump manual for other acceptable oils.

1. Unplug the vacuum pump from the power source.
2. Remove the exhaust filter by loosening the wing nut on the clamp between the vacuum pump exhaust filter and the mercury vapor filter.
3. Remove the centering ring and place to the side.
4. Remove the vacuum hose from the vacuum pump by loosening the wing nut on the clamp between the vacuum pump and the vacuum hose.
5. Remove the centering ring and place to the side.
6. Remove the pump from inside the analyzer.
7. Remove the vacuum pump from inside the analyzer .
8. at the top of the oil vapor trap. Swing the clamp open and remove the trap from the hose.
9. Grasp the handle on top of the vacuum pump and place the pump on a work surface.
10. Place a waste container under the drain spout.



11. Remove the drain plug and drain the oil into the waste container.
12. Replace the drain plug.
13. Remove the drain plug from the oil-fill port on top of the pump.



14. Slowly add oil to the port until the level is midway between the indicator lines in the oil-level window when the pump is running.



Do not allow oil to rise above the midway position when the pump is running. Doing so may cause oil to splash into the oil filter causing contamination.

15. Check the washer or O-ring used at the oil-filling port and replace if necessary.
16. Insert the oil-fill plug and turn clockwise to tighten.
17. Return the vacuum pump to the original position inside the analyzer.
18. Reconnect the vacuum pump hose.
19. Reconnect the exhaust filter to the top of the mercury vapor filter.
20. Reconnect the power cord to the power source.
21. Turn the vacuum pump on and recheck the oil level.
22. Allow the pump to run a few hours (overnight if possible) to eliminate air and moisture from the fresh fluid and to produce efficient vacuum operations.

CHECK VALVES FOR LEAKS

Valve leakage due to sample particles or oxidized mercury deposits on valve seats can cause difficulty in attaining adequate vacuum conditions. To test for leakage:

1. Open valves 1, 2, 3, and 8.
2. If the pressure does not reach 100 μm in less than five minutes, the system has a leak. If this occurs, clean the valves. See [Clean Valves below](#).

CLEAN VALVES

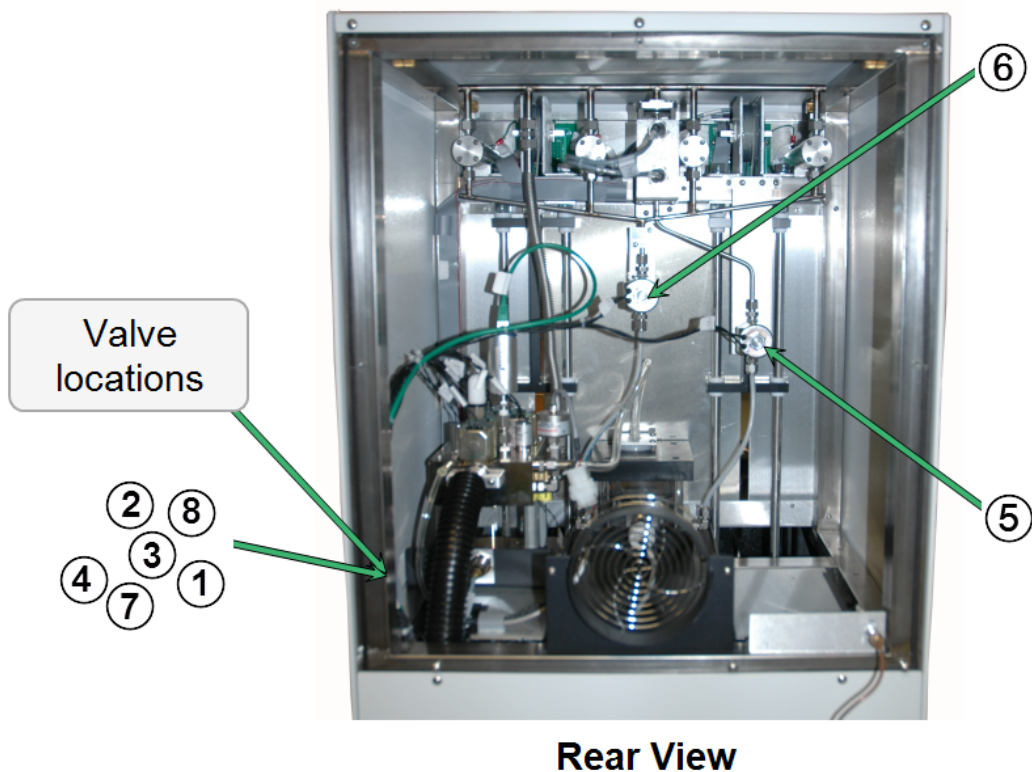


Ensure all mercury is below drain valves. Evacuate the reservoir and open the drain and fill valves with the low pressure manifold at atmospheric pressure. Failure to do so could result in a mercury spill.



Power off the analyzer before removing the rear panel. Failure to do so could result in personal injury.

1. Disconnect the power cord.
2. Remove the upper rear panel by removing the retaining screws.



- Remove the 11/16 in. nut retaining the valve actuating coil from the mercury fill [5] and drain [6] valves. Pull the coils off.

Valve Locations

Valve Location	Description
1	Servo isolation valve
2	Fast evacuation valve
3	Vacuum valve
4	Gas inlet valve
5	Mercury fill valve
6	Mercury drain valve
7	Mercury reservoir evacuation valve
8	Evacuation reservoir valve

- Place a container below the valves to capture any retained mercury.
- Remove the plunger housing from each using the special spanner wrench from the accessory kit.
- Clean the plunger and housing with IPA and expose the valve seat. Use a pipe cleaner to clean the valve seat. Ensure that no lint remains on the sealing surface.

7. Re-assemble the valves.
8. The slow / medium, medium, and fast evacuation valves, and the gas inlet solenoid valve are much less likely to collect debris. They can be cleaned (as outlined above) if cleaning the mercury valves did not solve the vacuum difficulty.

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.

REMOVE MOISTURE FROM THE ANALYZER

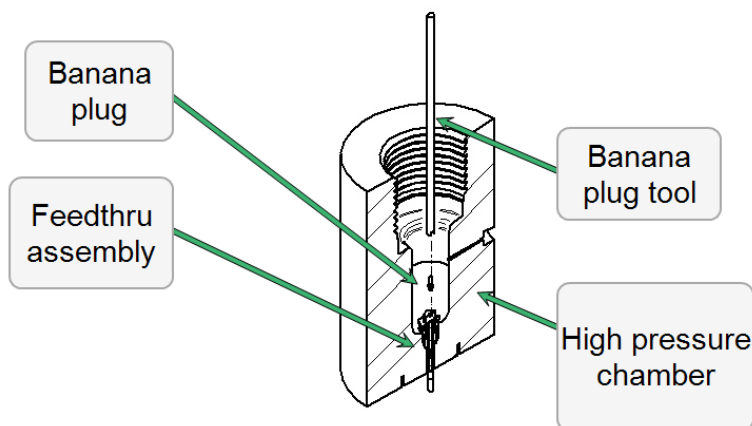
A difficult-to-detect vacuum problem arises if moisture is allowed to collect in the system. A probable accumulation point is the restricting frits that control flow rates. The best way to remove moisture is to evacuate the analyzer at full rate for several days. If the indicated vacuum continues to decrease slowly over this period, accumulated moisture is the probable cause of the vacuum difficulty.

To prevent the accumulation of moisture in the future, do not evacuate samples which hold excessive water vapor. This is best done by precleaning samples in a drying oven.

REPLACE THE BANANA PLUG

HIGH PRESSURE CHAMBER

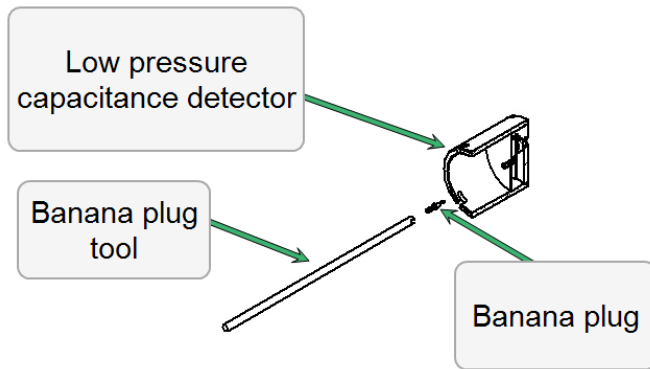
1. Remove the fluid from the high pressure chamber using the syringe provided.
2. Remove any mercury droplets found in the high pressure chamber. Clean the high pressure chamber using a clean dry cloth.
3. Locate the feedthru assembly in the bottom of the high pressure chamber. Remove the banana plug from the feedthru assembly using the banana plug tool provided.



4. Insert a new banana plug into the banana plug tool, ensuring the flat sides of the hex fit down into the slot.
5. Screw the banana plug into the feedthru assembly. Do not cross-thread or overtighten the banana plug.
6. Refill the high pressure chamber with clean high pressure fluid.

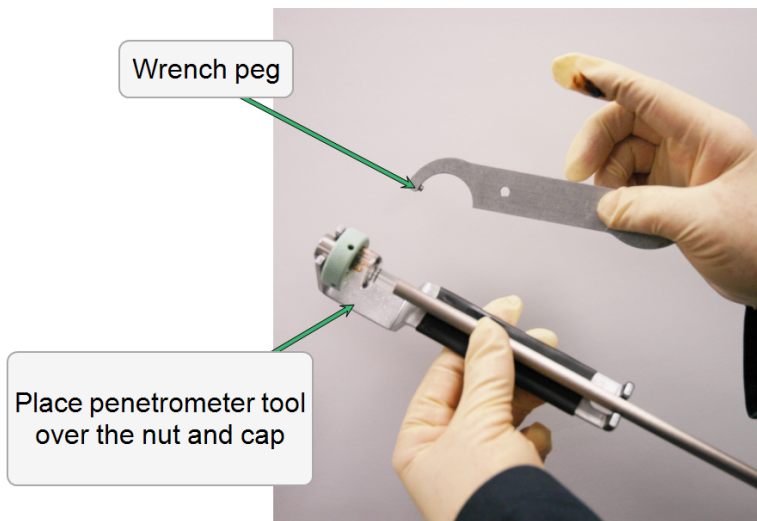
LOW PRESSURE CAPACITANCE DETECTOR

1. Remove the low pressure capacitance detector from the low pressure station.
2. Remove the banana plug from the low pressure capacitance detector using the banana plug tool provided.
3. Insert a new banana plug into the banana plug tool, making sure the flat sides of the hex fit down into the slot.



4. Screw the banana plug into the low pressure capacitance detector.

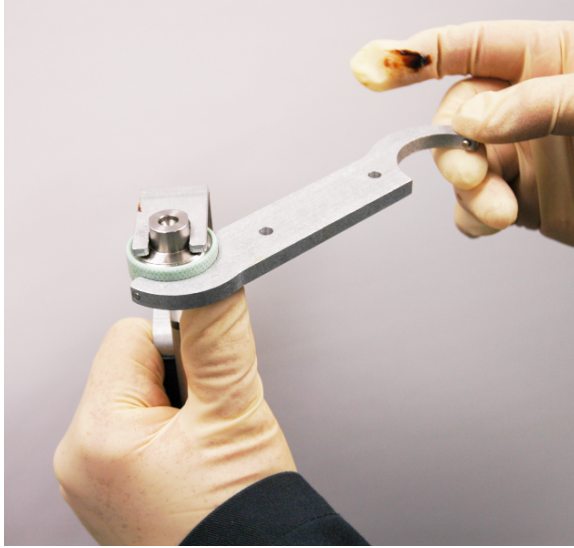
PENETROMETER NUT



REMOVE THE PENETROMETER NUT

If the penitrometer nut cannot be easily opened by hand, it may have become sealed too tightly during analysis. Use the penitrometer wrench to remove it.

1. Place the penitrometer tool over the nut.
2. Place the wrench peg into a hole in the penitrometer nut. Use the penitrometer tool to stabilize the cap while tightening the nut counter-clockwise.
3. Position the wrench so the peg on the wrench fits into the notch on the nut.



4. While holding the penetrometer tool, turn the wrench clockwise to loosen and remove the nut.

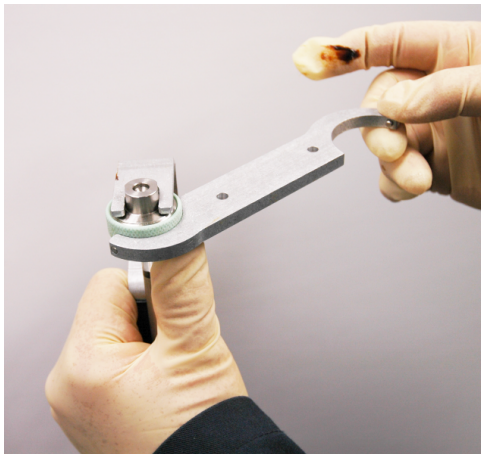
TIGHTEN THE PENETROMETER NUT

1. Apply grease to the penetrometer bulb and remove any excess grease.
2. Hold the penetrometer upright (sample bulb opening facing upward) and place a seal on the bulb opening.
3. Turn the seal one half turn to smooth the grease and seat the seal.



Take care not to move the seal after seating onto the penetrometer bulb. The grease may extrude too much and cause an air leak.

4. Place the penetrometer tool over the nut.



4. Place the wrench peg into a hole in the penetrometer nut. Use the penetrometer tool to stabilize the cap while tightening the nut counter-clockwise.

POWER ANALYZER ON AND OFF



DO NOT connect or disconnect cables when the analyzer is powered ON.

Power ON the equipment in the following order:



The vacuum line from the low pressure system and the vacuum pump filter must be installed before applying power to the vacuum pump. Refer to your vacuum pump manual.

1. Analyzer
2. Computer, monitor, and printer

Power OFF the equipment in the following order:

1. Exit the analysis program. Failure to do so could result in loss of data.
2. Computer, monitor, and printer
3. Analyzer

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.

A BLANK AND SAMPLE COMPRESSION CORRECTIONS

Baseline errors in AutoPore V data are errors that occur even when no sample is placed in the sample bulb and when a zero intrusion or extrusion volume of mercury would be expected as the pressure is increased to its maximum pressure and then decreased again. The material which follows relates the causes of these errors and discusses ways to minimize and compensate for them when maximum accuracy is required.

BASELINE ERRORS

When the AutoPore applies pressure to the mercury, penetrometer, and surrounding high pressure oil, compression occurs.

Compressibility effects account for a substantial portion of the baseline errors. The penetrometer bulb and capillary are made of glass which decreases in linear dimensions by about 0.8% and in volume by 2.3% at 60,000 psia. If the mercury were incompressible, a typical penetrometer having a 400 microliter capillary and a 5 milliliter bulb would experience a rise of mercury in the capillary of about 124 microliters or 31% of the capillary. Fortunately, mercury compresses also, but slightly more than glass such that the capillary actually falls some as the pressure is increased. The compressibility amounts to about 150 microliters in this example leaving a net fall of 26 microliters or about 6% of the capillary. The oil which surrounds the penetrometer and transmits the pressure to the mercury compresses at more than 10 times the rate of the mercury and occupies only 3/4 the original volume at 60,000 psia. Some of the oil is in the electric field of the capacitor, especially around the sample bulb and its connection to the exterior. The dielectric constant of the oil increases with its density. This contributes an increasing capacitance which cancels some of the decrease due to the net fall of mercury with compression.

Other effects caused by compression arise from the plastic insulators which are used on the penetrometer bulb base to prevent an electrical short circuit. Not only does the plastic compress almost as much as the oil, but it lags behind and only slowly assumes its final density. This is especially pronounced upon release of pressure where the plastic may continue to increase in dimensions for almost an hour. It also tends to increase the dielectric constant and capacitance with increasing pressure. The pressure vessel expands as the internal pressure is increased and, like the plastic, requires considerable time to stabilize. The resulting changes in spacing from the sample bulb to the walls and bottom causes a decrease in capacitance. Micromeritics has minimized this effect by making the initial spacings as large as is practical.

Another effect, and the one most difficult to predict, arises from the similarity of the penetrometer to a thermometer. This would not be troublesome if its temperature could be maintained constant, but compression of the surrounding oil causes a temperature rise of nearly 50 °C in the oil and a smaller change in the glass and mercury. How quickly this heat is transferred to the mercury depends upon how rapidly the pressure is being increased, the relative amounts of oil and mercury present, and how recently the vessel has been previously cycled and the metal and oil warmed relative to the penetrometer. Release of the pressure causes the inverse effect, chilling the oil and setting up a reversal of the heat flow. The thermal gradient across the glass of the penetrometer may be considerable such that little benefit may be derived from precisely equalizing the temperature coefficients of the mercury and glass. As might be expected this problem is worst when the sample bulb is large and the capillary volume small. Choosing the right penetrometer helps minimize this effect. Make sure the sample nearly matches the size of the sample bulb and that the capillary volume is large enough to satisfy intrusion.

APPROACHES FOR ERROR COMPENSATION

Situations arise where the typical errors of about 1.0% of capillary volume are significant or where the errors exceed this level due to unfavorable sample characteristics. Most commonly, this happens when one of the following is encountered: 1) The amount of sample available is so limited that the intrusion volume is only a small fraction of the smallest diameter capillary; 2) adequate sample is available but the porosity is so low that a limited amount of the smallest capillary is used even though the largest sample bulb is filled; 3) the sample is of small or moderate porosity and its compressibility or thermal properties differ considerably from those of mercury; 4) accuracy and reproducibility specifications have been imposed at levels tighter than the typically expected levels for mercury porosimetry. In such cases “blank corrections” may be used to advantage.

Micromeritics' AutoPore provides four different ways to apply blank corrections. The first, and simplest, is by use of stored formulas based upon averages of large numbers of blank runs on mercury-filled penetrometers under varying rates of pressure build and release. No provisions are made for entering compressibility data or thermal data since these numbers are seldom known and the formulas would become very complex. Typical examples of blank runs are shown in Figures 1, 3 and 7. Typical examples of formula blank correction of data are shown in Figures 2 and 6. It is very important that trial blank runs be made when applying these formulas to ensure that a reasonable degree of correction is actually attained under the running conditions being used.

The second technique is apt to be much more useful. It permits the user to run a blank run, store the results using the exact run conditions and penetrometer type to be used for the real sample, and subtract this result from other runs. Examples of correction by subtracting a blank run file are shown in Figures 4 and 8.

The third technique provides the highest degree of compensation possible and can be attained when the exact penetrometer to be used later is loaded with a nonporous sample of the same mass and material as the porous sample to be run later. When analyzed, the non-porous sample will expose all the aforementioned compressibility effects which can then be subtracted from the porous sample run. This third technique has the advantage of compensating for differences in compressibility and thermal effects between mercury and the sample material. Care should be exercised that the interval between runs, oil temperature, and penetrometer temperature, and any other initial conditions are made as nearly identical as possible. Figure 9 is a typical baseline run so obtained. Figure 10 is a subsequent blank run corrected using the Figure 9 data and shows the actual degree of correction attained.

Besides running blank runs, correction files may be created by manually entering the data. This fourth technique allows entry of the average of several blank runs, assuring a representative correction.

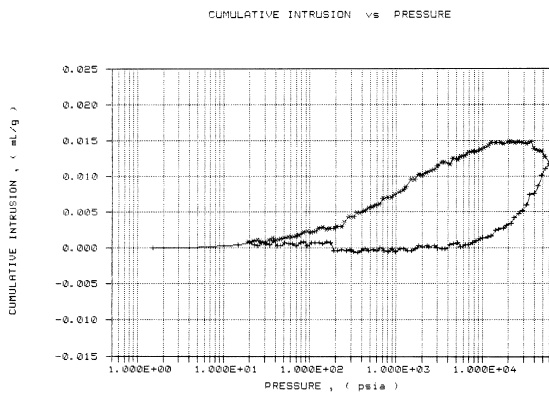


Figure 1

A blank run on a 5-mL powder penetrometer with a 1.1-mL stem volume. The rise in the initial depressurization data is primarily caused by thermal effects. As the hydraulic fluid is allowed to expand, it cools. this in turn cools the mercury in the penetrometer, causing it to contract and recede in the stem, giving the appearance of positive intrusion during depressurization.

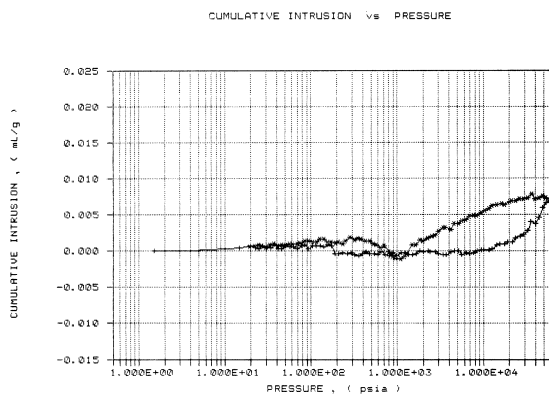


Figure 2

The difference between the blank data in Figure 1 and the formula blank correction for a run under the same conditions. The formula cancels some of the error, but does an imperfect job in this case.

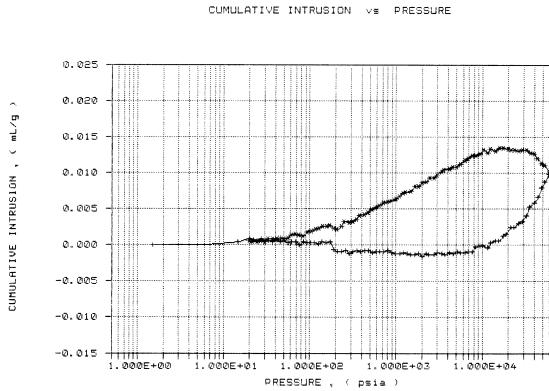


Figure 3

Another blank data set taken under identical conditions to those for Figure 1. The similarity between the two blank data sets is an indication of the excellent repeatability of blank runs.

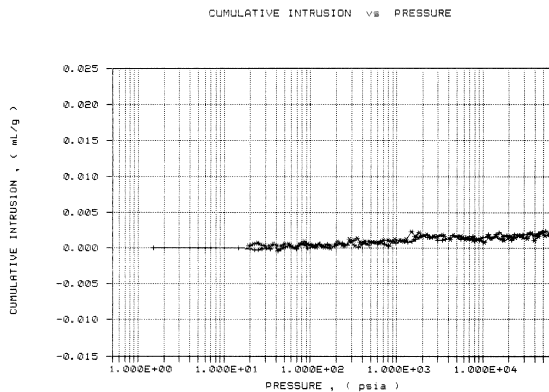


Figure 4

The difference between the blank data from Figure 1 and the blank data from Figure 3. This demonstrates that blank data collection and subtraction is a powerful method for accurately removing blank error from sample data.

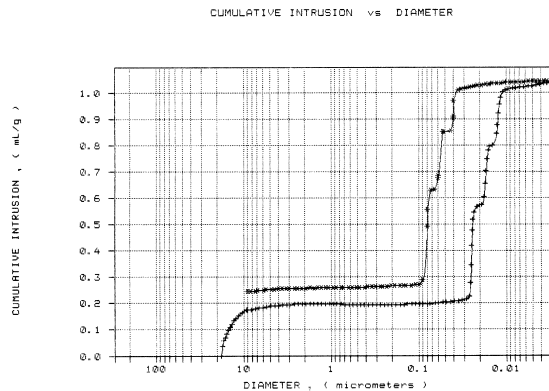


Figure 5

Uncorrected data from analysis of a sample of controlled pore glass made of a mixture of three pore sizes. Note the three distinct regions of intrusion between 0.03 and 0.01 micrometers on the pressurization curve, and the corresponding extrusion regions. The apparent intrusion at sizes above 10 micrometers is due to interparticle filling. The apparent intrusion between 0.01 and 0.003 micrometers, and the “loop” in the extrusion curve from 0.04 to 0.003 micrometers, are due to a combination of sample compression and blank error. There is no actual intrusion in this region.

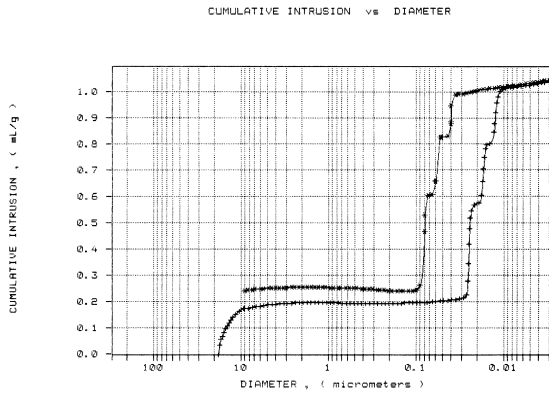


Figure 6

The data from Figure 5 with the formula blank correction applied. Note that the rise at the top due to blank error has been removed, but the apparent intrusion due to sample compression remains. This is because the formula makes no attempt to account for sample compression.

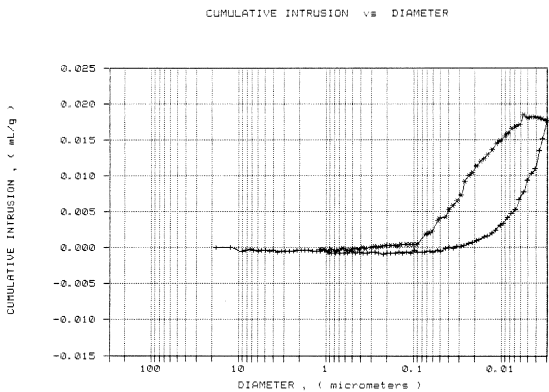


Figure 7

A blank run with the same type of penetrometer under the same conditions as the sample in Figure 5. It is dominated by the initial increase between pressurization and depressurization, primarily due to thermal effects.

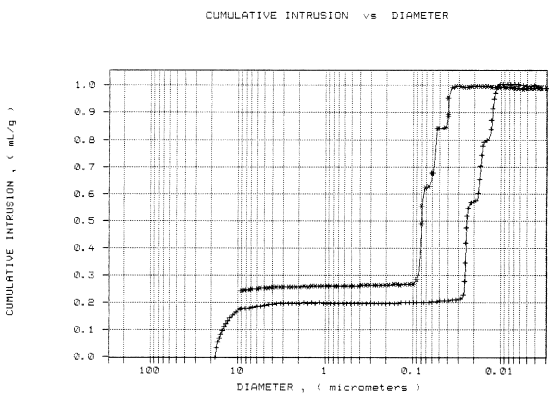


Figure 8

The sample data from Figure 5 corrected by subtracting the blank data from Figure 7. Note that practically all of the blank error and compression data have been removed, leaving only the filling curve and the actual intrusion. The sample compression is effectively canceled because the compression coefficient of mercury is close to that of the controlled pore glass used as sample. Many solid materials have compression coefficient fairly close to that of mercury, making this a very effective means of blank correction in many cases.

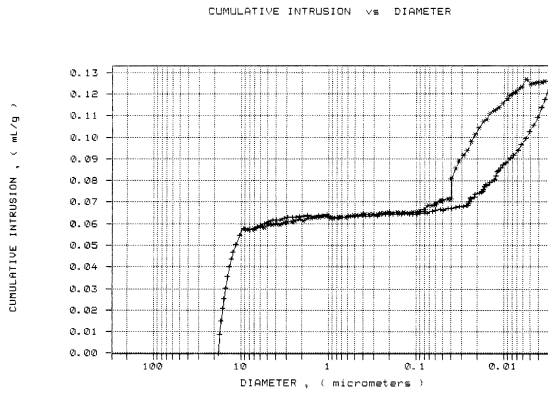


Figure 9

Uncorrected data from an essentially nonporous sample of the same type of glass shown in Figure 5. The mass of sample used was approximately equal to the mass of porous sample analyzed, so that the same volume was occupied. Note the filling curve and the blank error “loop.” The slight incline of the intermediate plateau and the angle of the “loop” are due to compression of the sample.

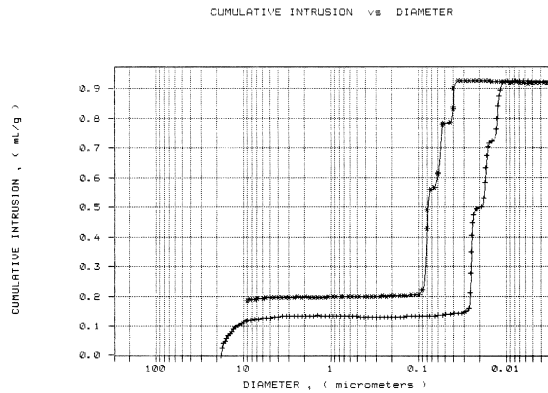


Figure 10

The difference between the porous sample data of Figure 5 and the nonporous sample data of Figure 9. Some of the filling curve has been removed, as well as all blank error and sample compression effect, leaving an accurate picture of the actual intrusion. This is the preferred method of blank correction, especially for materials with compression coefficients substantially different from that of mercury, and where maximum accuracy is desired.

B CHOOSE PROPER PUMP-DOWN RATES FOR UNFAMILIAR SAMPLE MATERIALS

There are a number of characteristics of an unfamiliar sample which can aid you in making a proper decision as to how aggressively the sample may be pumped down. Some of these characteristics are listed below. The assumption is always made that the sample material has been first dried in a shallow pan at 150 °C or higher for one hour or in a vacuum oven.

Samples, whether in the form of fine powders, granules, or even larger pieces, require extreme caution if one or more of the following characteristics are noted or known to be the case:

- a fine dust is raised upon stirring or shaking and the sample shows little sign of quickly settling
- the sample is a finely powdered organic material
- the sample is known to be microporous or mesoporous with pores of less than 100 Angstrom width
- the sample is known or suspected to be a carbon
- the sample is known or suspected to be a zeolite
- the sample is known or suspected to be a fluid cracking catalyst
- the sample has significant fine particle content below 10 mm
- the sample leaves behind a visible deposit of fine particles when transferred from a weighing pan

Such sample materials should be evacuated at initial pump down rates of about 0.5 psia/minute or more slowly if of marked fineness until a pressure of 0.1 psia is reached. The pulsing of the second pump down path should be maintained until 100 mmHg is reached before the final shift to the third and most direct pump down path takes place. The penetrometer must be filled only about 1/3 full so that there is a margin of clearance between the stem bore and the sample bed.

Sample materials which are coarse, medium, or dense powders, or are composed of obviously non-shedding chunks, pellets, or extrudates, can be pumped down at rates of 1 to 2 psia per minute until a pressure of 0.25 psia is reached. The pulsing of the second pump down path should be maintained until 250 mmHg is reached before the third pump down path takes over.

Sample materials that obviously present little risk such as monolithic chunks or very coarse, dense granules usually can be pumped down at near maximum rates. For these, specify 5 psia per minute initial pump down followed by a change to the second path at 0.5 psia and finally a transition to the direct path at 500 mmHg.

In most cases, you should choose a conservative rate of evacuation to be on the safe side. When large numbers of samples of a material are to be run, the potential time savings may make it worthwhile to investigate as to whether faster pump down rates might be possible without risk. To do so, leave the capacitance transducer off the penetrometer base so that you may observe the sample during a trial pump down performed at slightly higher than the usual speed. Twenty percent increases in pump-down rates and pressures of path changeovers are reasonable trial increases.

Monitor closely during all portions of the pump-down sequence, especially as changes from one path to another occur. Should the sample bed or any particles begin to boil or move, immediately stop the pump down and return the sample to atmospheric pressure; reduce the pump down rate to a slower rate and try again. If the pump down was successful, then another twenty percent speed increase might be attempted. Continue until a suitable rate is found. Note that this procedure is not without risk and must be done very carefully.

The amount of time for which to continue the extended pump down after the lowest target on the third path has been met will depend upon the structure of the sample and what volatile materials may be present within it. Large chunks of porous materials such as sandstone or green ceramics or concrete may present a considerable diffusion barrier for gases and vapors traveling to their surface and may require extended exposure times to vacuum to sufficiently clear the internal pores of this obstructive matter which would prevent fully measuring the true porosity. Experience and prior knowledge of the sample can be used for guidance.

Additionally, a manual mode test can be done in which you pump the sample down to the target pressure, and then close it off from further vacuum and monitor the rate and ultimate value of the pressure rebound that results from the tardy release of gas or vapor. The amount of time before the rebound ceases or remains within tolerable limits will serve to guide you in choosing an extended evacuation time for future automatic runs of the sample material.

C PROPER HANDLING OF MERCURY

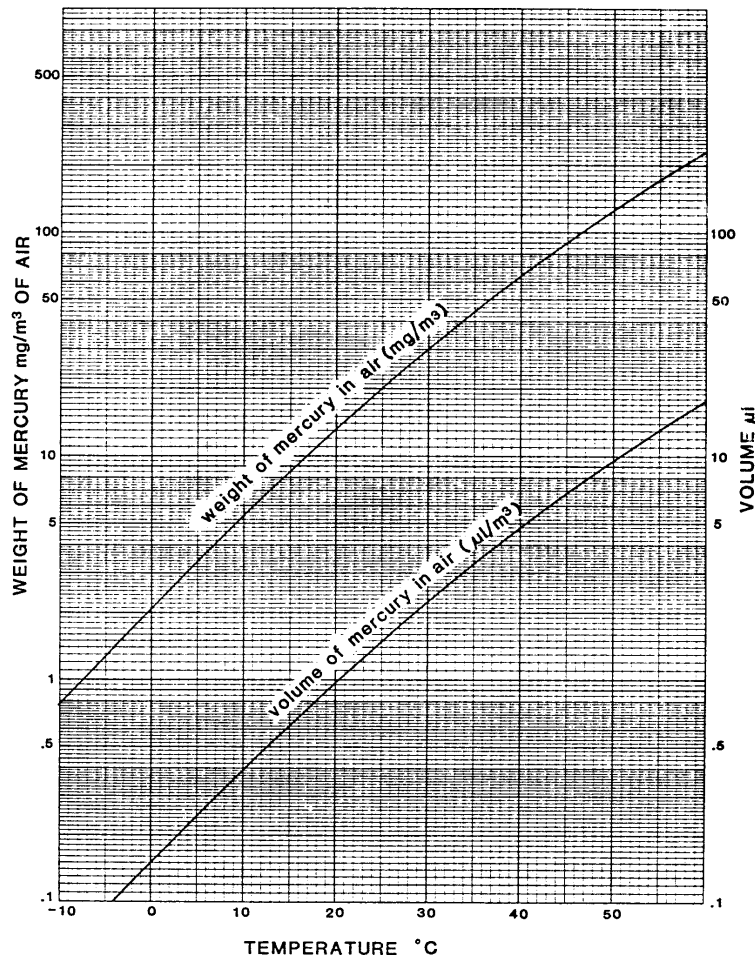
Because of its low melting point (-38.87 °C), mercury (Hg) is slightly volatile at ordinary room temperatures and its vapor may pose a health hazard if allowed to accumulate in the work space. Although mercury can enter the body through the skin, lungs or digestive system, breathing air laden with high concentrations of mercury vapor is the most common cause of mercury poisoning. Chronic poisoning caused by long-term exposure to low levels of mercury is occasionally found among those working with mercury. Mining, chemical, electrical, dentistry materials, pharmaceutical, explosive, porcelain, photography, printing, battery, paint, engraving, jewelry, cosmetics and color are some of the industries that use mercury in their manufacturing or processing.

Governmental agencies, i.e., National Institute for Occupational Safety and Health (NIOSH), Environmental Protection Agency (EPA), etc., and some industries have set criteria and recommended standards to protect the health and safety of workers exposed to mercury. A Threshold Limit Value (TLV) of 0.05 mgHg/cubic meter of air, recommended by the American Conference of Governmental Industrial Hygienists, was among the first hygienic guides for controlling exposure of mercury in the U.S. Values well below this level can easily be met through proper ventilation, prompt and thorough cleanup of spills, good personal hygiene and safe storage when working with mercury.

Health hazards from mercury can be prevented by limiting the average concentration of mercury to values below the TLV in an 8-hour workday. This is achieved through proper ventilation in the work area where mercury is handled; for example, a local exhaust ventilation system can be designed and maintained to prevent the accumulation or recirculation of mercury vapor, dust and fume; all handling of mercury can be confined to a hood, etc. Appropriate protective respiratory devices can be used when mercury exposure continues to exceed the recommended standard. To ensure TLV levels are met, governmental agencies suggest environmental levels of inorganic mercury be monitored every six months: breathing-zone samples are collected to permit calculation of a time-weighted average exposure for every operator. When any time-weighted average exposure is at or above the TLV, immediate steps are required to reduce exposure below the standard.

Maintaining low temperature where mercury is used will help limit mercury concentration. Vapor pressure of mercury goes up exponentially with temperature, for example, 20 °C: $P = 1.20 \times 10^{-3}$ mmHg. As temperature increases from 20 to 40 °C, the partial pressure of mercury vapor increases fivefold.

Proper clean-up of mercury spills and disposal of mercury-contaminated articles will limit exposure. In a poorly ventilated, closed area, where mercury spills have not been properly and thoroughly cleaned, mercury concentration in air can become significantly elevated above the TLV of 0.05 mg per cubic meter of air. The following figure shows that the equilibrium concentration of mercury at a room temperature of 25 °C reaches a level of 20 mg per cubic meter of air. This is 400 times the TLV, resulting in a dangerous work environment. Surveys in labs where mercury is routinely used reveal the presence of mercury in porous surfaces, in pools under cabinets or floors, and inside drawers and lab equipment. This accumulation can be attributed to the lack of an effective clean-up procedure for both large and small spills.



Mercury spills should be cleaned immediately and thoroughly by mechanical, chemical or other appropriate means. Micromeritics uses and recommends that you use plastic or rubber gloves and a small vacuum pump equipped with a mercury vapor absorbing filter on the exhaust and a vacuum probe with a mercury trap on the inlet for efficient pick-up of small mercury particles in cleaning mercury spills. Afterwards, the spill area should be swabbed with a mercury decontaminants¹⁾ and allowed to dry.

The health status of those working with mercury should be monitored regularly, with emphasis placed on good personal hygiene to prevent contamination of hands, mouth, clothing or food. Hand washing facilities, including hot and cold running water, soap, hand cream, and towels should be made available adjacent to mercury work areas. Clothing contaminated with mercury should be stored in vapor-proof containers pending removal for laundering.

¹⁾ Mercury decontaminants may be purchased from Fisher Scientific (800/766-7000) or Lab Safety Supply (800/356-0783). They also may be available from your local laboratory supplier.

Open containers for storage of mercury in the work area should be covered with an aqueous or an oil layer and kept at ambient temperatures to prevent vaporization. Because of permeability of polyethylene or plastic bottles to mercury vapor, thick glass bottles, stainless steel or cast iron containers are recommended for storing mercury. To avoid dangerous chemical reactions, mercury should not be stored with acetylene, fulminic acid, ammonia and oxalic acid.

PROPER USE OF MERCURY AS A TOOL IN PORE STRUCTURE ANALYSES

Micromeritics' Mercury Intrusion Porosimeters obtain accurate and reproducible pore structure analyses using mercury. Mercury is ideal as an intrusion liquid in the porosimetry method because it does not wet nor react with most materials. By measuring the amount of mercury intruded into the pores of a powdered or solid sample, the porosimeters give valuable data from which pore size, volume and distribution, as well as apparent densities, pore surface area and particle size can be determined.

All of Micromeritics' porosimetry instruments are designed with safety in mind. They come equipped with built-in spill and vapor safeguards that minimize operator exposure to mercury. They also are designed so that you may connect them to a ventilation system that pulls ambient air over the counter, through the instrument and out a duct at the rear. A built-in tray work area allows the operator to easily wipe exposed mercury to a dish where it is covered with oil. Our product literature on porosimetry supplies detailed site recommendations to assure safe operation.

Mercury vapor levels well below the accepted safe level can easily be achieved through regular monitoring, diligent handling and proper clean-up practices.

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D THEORY

Mercury porosimetry is based on the capillary law governing liquid penetration into small pores. This law, in the case of a non-wetting liquid like mercury and cylindrical pores, is expressed by the Washburn equation

$$D = - \left(\frac{1}{P} \right) 4\gamma \cos\phi$$

where D is pore diameter, P the applied pressure, γ the surface tension, and ϕ the contact angle, all in consistent units. The volume of mercury V penetrating the pores is measured directly as a function of applied pressure. This P - V information serves as a unique characterization of pore structure.

Pores are rarely cylindrical, hence the above equation constitutes a special model. Such a model may not best represent pores in actual materials, but its use is generally accepted as the practical means for treating what, otherwise, would be a most complex problem.

The surface tension of mercury varies with purity; its usually accepted value and the value recommended here is 485 dynes/cm. The contact angle between mercury and the solid containing the pores varies somewhat with solid composition. A value of 130 degrees is recommended in the absence of specific information to the contrary.

Mercury extruding from pores upon reduction of pressure is in general accord with the above equation, but indicated pore diameters are always offset toward larger diameters. This results from equivalent volumes of mercury extruding at pressures lower than those at which the pores were intruded. It is also commonly observed that actual pores always trap mercury. The first phenomena is usually attributed to receding contact angles being less than advancing ones. The second is likely due to pore irregularities giving rise to enlarged chambers and “inkwell” structures. These phenomena give rise to hysteresis phenomena, i.e., distinct intrusion and extrusion P - V curves.

Log in to your [customer portal](#) to access the Calculations document for a discussion on Pore Surface Area Computation.

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E USE OF THE MAXIMUM INTRUSION VOLUME OPTION

Using the maximum intrusion volume option allows routine analyses with fewer points in a pressure table while maintaining good resolution. However, use of the maximum intrusion volume requires some knowledge of the total pore volume of the sample to be analyzed. You should use about 2% of the sample's total pore volume as the maximum intrusion volume. This would give about fifty points for the intrusion pore spectrum and should be adequate to completely characterize most samples. The AutoPore IV will automatically add a pore spectrum point any time it sees an increment of intrusion equal to the maximum intrusion volume specified.

Care should be taken not to use too small a maximum intrusion volume. Use of a value less than 0.4% of the total intrusion volume will cause too many points to be taken at lower pressures. The total of 1000 data points will be exhausted and the analysis will terminate prematurely.

Use of too small a maximum intrusion volume can also cause points to be taken too close together on the pressure axis. If this causes pressures to be taken within the target pressure tolerance of each other, an apparent pressure decrease may be reported during the intrusion sequence. A reported pressure drop greater than 10 psi or 0.5% of the target pressure will be interpreted as the end of the intrusion segment. Reported summary data (such as total intrusion volume) will be reported at this point, rather than at the maximum pressure as intended. Data for graphs other than cumulative intrusion volume will also be terminated at this point.

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F WORKSHEETS

Worksheets in this section may be copied as needed.



See also:

- [*Analysis Conditions Worksheet on the next page*](#)
- [*Penetrometer Properties Worksheet on page F - 3*](#)
- [*Pressure Table and Tabular Data Worksheet on page F - 4*](#)
- [*Report Options Worksheet on page F - 5*](#)
- [*Sample Data Worksheet on page F - 6*](#)
- [*Volumetric Reference Capsule Worksheet on page F - 7*](#)

ANALYSIS CONDITIONS WORKSHEET

File Name: _____

Analysis conditions identifier	
Low pressure equilibration	<input type="checkbox"/> Time <input type="checkbox"/> Rate
Max. intrusion volume	
Evacuation	Initially evacuate at _____ Switch to medium at _____ Switch to fast at _____ Evacuation target _____ Continue evacuating for _____
High pressure equilibration	<input type="checkbox"/> Time <input type="checkbox"/> Rate <input type="checkbox"/> Pressure-controlled scan <input type="checkbox"/> Intrusion-controlled scan
Max. intrusion volume	

Mercury Properties

Advancing contact angle	
Receding contact angle	
Hg surface tension	
Hg density	

PENETROMETER PROPERTIES WORKSHEET

File Name: _____

Penetrometer Number (etched on penetrometer)	
Mass. Weigh the loaded penetrometer. Subtract the mass of the sample. Enter the difference.	
Volume	
Constant	
Stem Volume	
Max. head pressure	
Correction method <input type="checkbox"/> Blank <input type="checkbox"/> Formula <input type="checkbox"/> None	If <i>Blank</i> , give file name:

REPORT OPTIONS WORKSHEET

File name	
Calculation range	
<input type="checkbox"/> Pressure <input type="checkbox"/> Pore size	
Reports	
Overlays	
Sample 1	Sample 5
Sample 2	Sample 6
Sample 3	Sample 7
Sample 4	Sample 8

SAMPLE DATA WORKSHEET

_____ Basic presentation sample file

_____ Advanced presentation sample file

	Port 1	Port 2	Port 3	Port 4
Sample file name or identifier <i>(optional)</i>				
Penetrometer number <i>(optional - etched on penetrometer)</i>				
Sample mass(g) . Enter this value on the <i>Sample Description</i> tab.				
Sample + penetrometer mass . Load the penetrometer with sample and weigh.				
<p>Penetrometer mass. Subtract the <i>sample mass</i> from the <i>Sample + penetrometer mass</i> and enter the difference.</p> <ul style="list-style-type: none"> • If using Basic presentation, enter this value on the <i>Sample Information</i> window. • If using Advanced presentation, enter this value on the <i>Penetrometer Properties</i> window. 				
High pressure port number <i>(optional)</i> . Enter the number of the high pressure port where the sample is loaded.				
Assembly mass . The mass of the <i>sample + penetrometer + mercury</i> . (Weigh the penetrometer after the low pressure analysis.) Enter this value on the <i>Start High Pressure Analysis</i> window.				

VOLUMETRIC REFERENCE CAPSULE WORKSHEET

Calculate the volumetric reference capsule volume three times, then record the average of these calculations. When creating the penetrometer properties file, enter the average volume.

Penetrometer Number _____ Date: _____
By: _____

First Calibration of Reference Capsule Volume:

1. Mass of capsule filled with mercury	_____	g
2. Mass of sealed, empty capsule	_____	g
3. Mass of mercury (line 1 minus line 2)	_____	g
Room Temp = _____ °C	Density of mercury * = _____	g/ml
4. Volume of capsule (line 3 ÷ density of mercury)	_____	ml

Second Calibration of Reference Capsule Volume:

1. Mass of capsule filled with mercury	_____	g
2. Mass of sealed, empty capsule	_____	g
3. Mass of mercury (line 1 minus line 2)	_____	g
Room Temp = _____ °C	Density of mercury * = _____	g/ml
4. Volume of capsule (line 3 ÷ density of mercury)	_____	ml

Third Calibration of Reference Capsule Volume:

1. Mass of capsule filled with mercury	_____	g
2. Mass of sealed, empty capsule	_____	g
3. Mass of mercury (line 1 minus line 2)	_____	g
Room Temp = _____ °C	Density of mercury * = _____	g/ml
4. Volume of capsule (line 3 ÷ density of mercury)	_____	ml

Average Reference Capsule Volume:

	ml
--	----

* See [Density of Mercury Table on the next page](#) for values.

DENSITY OF MERCURY TABLE

Density of Mercury Table

°C	g/ml	°C	g/ml	°C	g/ml	°C	g/ml
18.0	13.5512	23.2	13.5384	25.2	13.5335	27.2	13.5286
19.0	13.5487	23.4	13.5379	25.4	13.5330	27.4	13.5281
20.0	13.5462	23.6	13.5374	25.6	13.5325	27.6	13.5276
21.0	13.5438	23.8	13.5369	25.8	13.5320	27.8	13.5271
22.0	13.5413	24.0	13.5364	26.0	13.5315	28.0	13.5266
22.2	13.5408	24.2	13.5359	26.2	13.5310	29.0	13.5242
22.4	13.5403	24.4	13.5354	26.4	13.5305	30.0	13.5217
22.6	13.5399	24.6	13.5350	26.6	13.5301	31.0	13.5193
22.8	13.5394	24.8	13.5345	26.8	13.5296	32.0	13.5168
23.0	13.5389	25.0	13.5340	27.0	13.5291	33.0	13.5144

G EXPORTED DATA EXAMPLE

Sample Information

Sample Information

Method: HP REF CAPSULE (LARGE)
Sample: HP REF CAPSULE (LARGE)
Operator:
Submitter:
Type of data: Automatically collected
Instrument type: 9600
Original instrument type: 9600
Comments:
Penetrometer mass: 1.0000 g
Assembly mass: 1.0000 g

Material Properties

Material: Garnet
BET surface area: 200.0000 m²/g
Use user entered density: No
Use user entered conductivity formation factor: No
Use user entered pressure threshold: No
Linear compressibility: -2.7400e-007 1/psia
Quadratic compressibility: 2.8500e-013 1/psia²

Penetrometer Properties

Penetrometer: ##### - 0 cc Bulb, 0.000 cc MMV, solid
Penetrometer mass: 1.0000 g
Volume: 1.0000 mL
Constant: 10.790 μ L/pF
Stem volume: 0.4120 mL
Max. head pressure: 4.680 psia
Correction method: None

Analysis Conditions

Analysis Conditions: HP REF CAPSULE (LARGE)

Mercury Properties

Advancing contact angle: 130.000 °
 Receding contact angle: 130.000 °
 Surface tension: 485.000 dynes/cm
 Density type: Entered
 Mercury Density vs. Temperature: 13.5335 g/mL
 Linear compressibility: -2.7400e-007 1/psia
 Quadratic compressibility: 2.8500e-013 1/psia²

Evacuation Options

Sample type: other
 Initially evacuate at: 10.0 psia/min
 Switch to medium at: Use pressure transducer
 0.50 psia
 Switch to fast at: 900 µmHg
 Evacuation target: 50 µmHg
 Continue evacuating for: 5 min

Low Pressure

Filling pressure: 10.000 psia
 Equilibration time: 2 s

High Pressure

Equilibration time: 20 s
 Hold at maximum pressure: No

Reverberi Options

Autocalculate Reverberi pressures: No

Pressure Table

	Pressure Increment (psia)	Points per Decade	Ending Pressure (psia)	Maximum Intrusion (mL/g)	Pressure Scan Rate (min/decade)	Intrusion Scan Rate (mL/g·s)
1			11.00		5.0	0.00100
2			15.00		5.0	0.00100
3			20.00		5.0	0.00100
4			23.00		5.0	0.00100
* 5			25.00		5.0	0.00100
6			30.00	0.050	5.0	0.00100
7	2.50		50.00	0.050	5.0	0.00100
8			100.00	0.050	5.0	0.00100
9	100.00		400.00	0.050	5.0	0.00100

Report Options

```

Report Options:      HP REF CAPSULE (LARGE)
Show report title:  Yes
                   HP REFERENCE CAPSULE TEST (LARGE)
Show bitmap:       Yes
  Height:          0.250 in
  Width:           2.000 in
Overlay Files:     None
Specification Files: None
Reference Files:   None
    
```

Intrusion Data Options

```

Report negative intrusion: No
Smooth differentials:     Yes
Adjust intrusion for compressibility: No
    
```

Reports

```

Summary:           Yes
Tabular Report:    Yes
Cum. Vol. vs Size: No
Inc. Vol. vs Size: No
Cum. Area vs Size: No
Cum. Vol. vs Pressure: Yes
Inc. Vol. vs Pressure: No
Diff. Vol. vs Size: 1 No
Log Diff. Vol. vs Size: No
Diff. Ref. % Vol. vs Size: No
Out Spec. % Vol. vs Pressure: No
Diff. Vol. vs Size: 2 No
Mayer-Stowe:      No
Pore Structure:   No
Material Compressibility: No
Cavity to Throat Size Ratio: No
Fractal Dimension: No
Reverberi:        No
Advanced:          No
Options:           Yes
Sample Log:        No
Validation:        No
    
```

Summary Report

```

Total intrusion volume: Yes
Total pore area:         Yes
Median pore Diameter:   Yes
Average pore Diameter:  Yes
Bulk density at 0.10 psia: Yes
Apparent (skeletal) density at 61,000.00 psia: Yes
Porosity:                Yes
Show graph:              No
Show pressure table:     No
Show pore size table:    No
Show percent intrusion table: No
Show peak table:         No
    
```

Pore Structure Summary

Permeability: No
 Threshold pressure: No
 Characteristic length: No
 Conductivity formation factor: No
 Tortuosity factor: No
 Tortuosity: No
 Percolation fractal dimension: No
 Backbone fractal dimension: No

Physical Properties Summary

Mayer-Stowe interstitial porosity: No
 Mayer-Stowe breakthrough pressure ratio: No
 Material compressibility coefficients: No
 Calculated porosity: No

Tabular Report

Column 1: Pressure
 Column 2: Mean Pore Size
 Column 3: Cumulative Volume
 Column 4: Incremental volume
 Column 5: None
 Column 6: None
 Tabular Data Definition: Collected Data

Cum. Vol. vs Pressure

Plot points: Yes
 Plot curve: Yes
 Show as histogram: No

 X-Axis
 Variable: Pressure
 Scale: Logarithmic
 Autoscale: Yes
 Secondary X axis: None

 Y-Axis
 Variable: Cumulative Intrusion
 Overlay:
 Plot intrusion: First
 Plot extrusion: First
 Autoscale: Yes

H COMPUTE VOLUMETRIC COMPRESSIBILITY OF A SAMPLE MATERIAL

Ideally, you should choose a sample material that is completely non-porous; if this is not the case, then you should choose the pressure range over which the compressibility test is conducted such that no pore filling occurs within it. Closed pores may not always cause volume changes during testing but they may alter the results due to stress concentrations around them or because of their effects upon measured density. Closed pores may also abruptly fail and even become open during testing and cause invalid compressibility results to be reported. In some cases, such as the testing of plastic foams at low pressures, the presence of closed pores may be acceptable and expected.

The sample mass and sample density must be known and available to a resolution and accuracy at least three significant digits (preferably better) to permit accurate computation of the initial volume of the sample material. Alternatively, an accurate geometric volume of a material such as one containing closed pores (such as plastic foam) may be supplied. Before data reduction can be performed, you must have available a “blank run” file consisting (at least ideally) of a run made with the same penetrometer and accessory hardware that is to be used in the compressibility test and (again ideally) on the same instrument ports as will be used in the compressibility run. The pressure range of the blank run must, at a minimum, fully encompass the planned range to be used in the compressibility measurement and have a minimum of seven uniformly spaced (linear basis) data points inside the planned computation range and with the beginning and ending data points within 5% (pressure) of the planned computation range end points. It is also permissible for the “blank run” to consist of a manually entered data file.

The first and second order isostatic pressure coefficients of volumetric compressibility for mercury over the pressure range from zero psia to 60,000 psia must be known and available. All standard input information such as sample material identity, equilibration times, evacuation information, penetrometer constants, etc. that would be required for standard runs is required for a compressibility run. Note that sample volume, bulk volume / density, and skeletal volume / density as measured during the mercury porosimetry run are, in general, far too imprecise to yield good results if used in the compressibility computations. For this reason, you must enter very accurate material density and sample mass values to be used in computing an accurate initial sample volume or, alternatively, directly enter a measured initial sample material volume.

The pressure table entered must contain at least seven pressure points uniformly spaced (on a linear basis), with these points coinciding as closely as possible to those in the blank run which is to be used along with the data in the final computation. As indicated above, the pressure values of the end points achieved during the run must be within 5%

Blank Page

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