AUTOPORE V SERIES

MERCURY INTRUSION POROSIMETER

MICROMERITICS

OPERATOR MANUAL

962-42800-01
Feb 2015
(Rev B)

Trademarks

Alconox is a registered trademark of the Alconox Company.

AutoPore is a registered trademark of Micromeritics Instrument Corporation.

Micromeritics is a registered trademark of Micromeritics Instrument Corporation.

Microsoft, Windows, and Windows 7 are registered trademarks of Microsoft Corporation.

Python is a registered trademark of Python Software Foundation.

Micromeritics is a registered trademark of Micromeritics Instrument Corporation.

Swagelok is a registered trademark of Swagelok.

This application may contain a binary form of the Info-ZIP tool to create .zip files. That source code is provided under the following license:

This software is provided "as is," without warranty of any kind, express or implied. In no event shall Info-ZIP or its contributors be held liable for any direct, indirect, incidental, special or consequential damages arising out of the use of or inability to use this software.

Permission is granted to anyone to use this software for any purpose, including commercial applications, and to alter it and redistribute it freely, subject to the following restrictions:

1. Redistributions of source code must retain the above copyright notice, definition, disclaimer, and this list of conditions.
2. Redistributions in binary form must reproduce the above copyright notice, definition, disclaimer, and this list of conditions in documentation and / or other materials provided with the distribution.
3. Altered versions--including, but not limited to, ports to new operating systems, existing ports with new graphical interfaces, and dynamic, shared, or static library versions--must be plainly marked as such and must not be misrepresented as being the original source. Such altered versions also must not be misrepresented as being Info-ZIP releases--including, but not limited to, labeling of the altered versions with the names "Info-ZIP" (or any variation thereof, including, but not limited to, different capitalizations), "Pocket UnZip," "WiZ" or "MacZip" without the explicit permission of Info-ZIP. Such altered versions are further prohibited from misrepresentative use of the Zip-Bugs or Info-ZIP e-mail addresses or of the Info-ZIP URL(s).
4. Info-ZIP retains the right to use the names "Info-ZIP," "Zip," "UnZip," "WiZ," "Pocket UnZip," "Pocket Zip," and "MacZip" for its own source and binary releases.

Copyright

WARRANTY

MICROMERITICS INSTRUMENT CORPORATION warrants for one year from the date of shipment each instrument it manufactures to be free from defects in material and workmanship impairing its usefulness under normal use and service conditions except as noted herein.

Our liability under this warranty is limited to repair, servicing and adjustment, free of charge at our plant, of any instrument or defective parts when returned prepaid to us and which our examination discloses to have been defective. The purchaser is responsible for all transportation charges involving the shipment of materials for warranty repairs. Failure of any instrument or product due to operator error, improper installation, unauthorized repair or alteration, failure of utilities, or environmental contamination will not constitute a warranty claim. The materials of construction used in MICROMERITICS instruments and other products were chosen after extensive testing and experience for their reliability and durability. However, these materials cannot be totally guaranteed against wear and / or decomposition by chemical action (corrosion) as a result of normal use.

Repair parts are warranted to be free from defects in material and workmanship for 90 days from the date of shipment.

No instrument or product shall be returned to MICROMERITICS prior to notification of alleged defect and authorization to return the instrument or product. All repairs or replacements are made subject to factory inspection of returned parts.

MICROMERITICS shall be released from all obligations under its warranty in the event repairs or modifications are made by persons other than its own authorized service personnel unless such work is authorized in writing by MICROMERITICS.

The obligations of this warranty will be limited under the following conditions:

1. Certain products sold by MICROMERITICS are the products of reputable manufacturers, sold under their respective brand names or trade names. We, therefore, make no express or implied warranty as to such products. We shall use our best efforts to obtain from the manufacturer, in accordance with his customary practice, the repair or replacement of such of his products that may prove defective in workmanship or materials. Service charges made by such manufacturer are the responsibility of the ultimate purchaser. This states our entire liability in respect to such products, except as an authorized person of MICROMERITICS may otherwise agree to in writing.
2. If an instrument or product is found defective during the warranty period, replacement parts may, at the discretion of MICROMERITICS, be sent to be installed by the purchaser, e.g., printed circuit boards, check valves, seals, etc.
3. Expendable items, e.g., sample tubes, detector source lamps, indicator lamps, fuses, valve plugs (rotor) and stems, seals and O-rings, ferrules, etc., are excluded from this warranty except for manufacturing defects. Such items which perform satisfactorily during the first 45 days after the date of shipment are assumed to be free of manufacturing defects.

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

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

CONTACT US

Micromeritics Instrument Corporation

4356 Communications Drive

Norcross, GA / USA / 30093-2901

Phone: 1-770-662-3636

Fax: 1-770-662-3696

www.Micromeritics.com

U.S. Inquiries

U.S. Sales Phone: 1-770-662-3636

USSales@Micromeritics.com

Quotes, Orders, and Customer Satisfaction

Customer Service Phone: 1-770-662-3636

Orders@Micromeritics.com

U.S. Instrument Service Department

U.S. Service Department Phone: 1-770-662-3666

ABOUT THIS MANUAL

The following formats may be used in this manual.

NOTE - Notes contain important information pertinent to the subject matter.

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

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

Field Labels and Screen Titles

Labels and Buttons	Description
Buttons (in the application)	Buttons in the application are represented as bold font and blue letters — such as: **Save**, **Edit**, and **Replace All**.
Buttons (on the equipment)	Buttons on the equipment are represented as bold font and black letters — such as: **On**or **Off**.
Field Labels	Field Labels are represented as italicized words — such as: *Sample, Automatically Collected,* and *Analysis Conditions*.
Keyboard Commands	Keyboard commands are represented as bold font and black letters — such as: **F2** and **Alt+F4**.
Menu Instructions	Menu instructions are represented as bold and italicized words — such as: ***File > New Sample*** and ***Reports > Start Report***.
Screen Tabs	Screen Tabs are represented as italicized words — such as: *Sample Description*, *Analysis Conditions*, and *Report Options*.
Screen Titles	Screen Titles are represented as italicized words — such as: *Analysis Adsorptive Properties*, *Free Space*, and *Sample Tube*.

Blank Page

Table of Contents

1 About the AutoPore V

The mercury porosimetry analysis technique is based on the intrusion of mercury into a porous structure under stringently controlled pressures. Besides offering speed, accuracy, and a wide measurement range, mercury porosimetry permits the calculation of numerous sample properties such as pore size distributions, total pore volume, total pore surface area, median pore diameter and sample densities (bulk and skeletal).

The AutoPore V Series Mercury Porosimeters can determine a broader pore size distribution (500 to 0.003 micrometers) more quickly and accurately than other methods. This instrument also features enhanced safety features and offers new data reduction and reporting choices that provide more information about pore geometry and the fluid transport characteristics of your material.

FRONT PANEL

Front Components

Component	Description
High Pressure Ports 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 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.
Hg Up	Illuminates when the amount of mercury in the low pressure system is sufficient to fill the penetrometers.
Hg Drained	Illuminates when mercury has been drained from the low pressure system.
Power	Illuminates when power is supplied to the instrument.

REAR PANEL

Rear Components

Component	Description
Aux RS-232 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	Turns the analyzer on and off. It also provides electrical overload protection.
Voltage Selector	Used to set the analyzer for the correct incoming AC line voltage.

OPERATIONAL SAFETY FEATURES

The AutoPore V comes with multiple safety features:

- **Mercury Drip Trays**

  Safe collection of mercury for disposal in the case of compromised penetrometer seals or operator error resulting in broken penetrometers

- **Mercury Temperature Sensor**

  Automatic measurement of mercury temperature allows automatic calculation of mercury density used for penetrometer calibrations. The ability to set a temperature limit in manual mode allows the display of a warning message if the temperature is exceeded.

- **Improved Mercury Funnel Design**

  Attached screw cap and funnel-shaped opening eliminates mercury contamination and possible drip spillage associated with separate detached filling funnel. Attached screw cap prevents loose cap and possible vapor release.

- **Triple Fail Safe-One Penetrometer Safety Caps**

  In case of operator error, this device prevents penetrometer or rod being released from port unintentionally

- **Triple Fail Safe-Two Interlock on Locking Cap**

Verifies that capacitance detector is installed on low pressure port, automatically suspends run if cap is not in place. This prevents the penetrometer from being accidentally released while under pressure. If error is corrected, suspend run can be resumed.

- **Triple Fail Safe-Three System Pressure Vent on manifold**

  Works in concert with cap interlock to automatically vent system pressure if above ambient pressure and error condition is detected.

- **Mercury Vapor Detection Device**

  Hand held device to check localized mercury vapor levels that exceed defined safety limits. Portable device allows point checks at the instrument or any location within lab exposed to mercury.

- **Mercury Vapor Capture Filter**

  Affixed to vacuum pump, this filter prevents release of mercury vapors. This is a superior solution to cold trap dewars, particularly if the cryogen level is insufficiently maintained.

- **Software Control for Fine Powder Samples**

  Prevents fine powder from accidental aspiration into low pressure ports during analysis.

POWER THE ANALYZER ON-OFF

POWER ON THE ANALYZER

> **CAUTION** 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. Place the analyzer's power switch in the ON position. The *Power* indicator on the upper front panel is illuminated.
2. Turn on the video monitor, printer, and computer.
3. To start the application, double click the analyzer icon or select the AutoPore program from the Windows Start menu.

POWER OFF THE ANALYZER

> **CAUTION** Ensure that the application is closed prior to powering off the analyzer. Failure to do so could result in loss of data.

1. Go to ***File > Exit***. If the application is closed with analyses in progress, the analyses continue and data are collected. Reports that are queued will print.
2. Place the computer, monitor, and printer ON / OFF switches in the OFF position.
3. Place the analyzer power switch in the OFF position.

THE BASIC WORKFLOW

Before a sample can be analyzed, it must be:

1. Weighed and the mass recorded, and
2. A sample information 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 install it in a high pressure port. The high pressure analysis collects data at pressures indicated in the high pressure portion of the pressure table up to maximum pressure specified.

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's stem is measured by determining the penetrometer's electrical capacitance. Capacitance is the amount of electrical charge stored per volt of electricity applied. The penetrometer's 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's capacitance. The analysis application converts measurements of the penetrometer's capacitance into data points showing the volume of mercury intruding into the sample's pores.

"Theory" on page F - 1 provides a thorough discussion of the theory of porosimetry.

SPECIFICATIONS

Characteristic	Specification
Electrical	
Voltage	100/120/220/240 VAC ± 10%
Frequency	50/60 Hz
Power	500 VA maximum
Gas	
Nitrogen or other clean, dry gas at 50 psig (345 kPa)	
High Pressure Model 9605	
Measurement	From atmospheric pressure to 33,000 psia
Resolution	0.165 psia from 3,300 psia to 33,000 psia
Transducer Accuracy	± .1% of full scale (transducer manufacturer's specifications)
Transducer Hysteresis	.05% of full scale
Pore Diameter Range	6 to 0.005 µm
Servo Control Precision	0.5% of target, min 5 psia, no overshoot
High Pressure Model 9620	
Measurement	From atmospheric pressure to 60,000 psia
Resolution	0.03 psia from atmospheric pressure to 60,000 psia
Transducer Accuracy	± .1% of full scale (transducer manufacturer's specifications)
Transducer Hysteresis	.05% of full scale
Pore Diameter Range	6 to 0.003 µm
Servo Control Precision	0.5% of target, min 5 psia, no overshoot
Intrusion	
Resolution	Better than 0.1 all
Accuracy	± 1% of maximum penetrometer stem volume
Low Pressure	
Measurement	0.2 to 50 psia (345 kPa)
Resolution	0.00025 psi
Pore Diameter Range	500 to 3.6 µm
Transducer Accuracy	± 1% of full scale (transducer manufacturer’s specifications)
Servo Control Precision	1% of target, min. .05 psia, no overshoot
Penetrometers	
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)

Characteristic	Specification
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	
Windows 7 Professional or higher operating system is recommended for the best user experience. If the computer is to be connected to a network, a second Ethernet port on the computer must be used for that purpose.	
All users of the application will need Read / Write permission to all directories and subdirectories where the application is installed.	
In keeping with a policy of ongoing product improvement, specifications are subject to change without notice.	

2 About the Software

The analyzer software allows other computer programs to run while an automatic operation is in progress. The *Help* menu provides access to this operator manual and tutorials on using the software.

The MicroActive feature offers a Windows interface with an easy way to collect, organize, archive, and reduce raw data and store sample information files for later use. Scalable and editable graphs, and cut-and-paste graphics, are easily generated. Customized reports can be generated to screen, paper, or exported for use in other programs. There are two report functions:

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

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

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 CD. It is used to:

- Reinstall the software version [*n*]
- Add an analyzer
- Move an analyzer
- Remove an analyzer
- Change analyzer setup
- Reinstall calibration files for an analyzer
- Import an analyzer from a previous installation on this PC

To access the *Setup* program:

1. Insert the *Setup* CD into the CD drive.
2. Locate and double click the *Setup.EXE* file.

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

SOFTWARE UPDATES

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

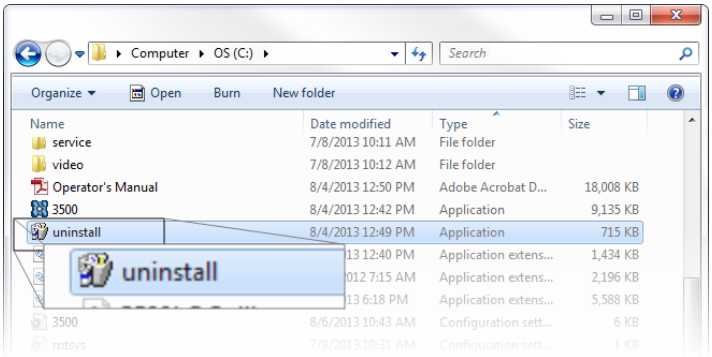
- a later version than the current installation
- the same version as the current installation
- an earlier version than the current installation

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

UNINSTALL THE SOFTWARE

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

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



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.
Unit [*n*]	Use to perform analyses, calibrations, and other analyzer operations. A *Unit [n]* 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, 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 operator manual, online instructional tutorials, 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 Log Entry	Use to enter information to appear in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Autoscale checkbox	When enabled on report parameters windows, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y- ranges will be set so that all the data is shown. If *Autoscale* is not selected, the entered range is used.
Axis Range	On report parameters windows, the *From* / *To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and / or y-axes.
Bar Code	Enter bar code reader information if a bar code reader is connected to the computer's USB port. If a bar code reader is not used, this alphanumeric field can be used to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Browse	Searches for a file. Select a file from the *Name* column or from the library, then click **Open**. Alternatively, double click the file name to open (or import) the file.
Cancel	Discards any changes or cancels the current process.
Close	Closes the active window.
Close All	Closes all active windows. If changes were made and not yet saved, a prompt displays for each changed file providing the option to save the file.
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Delete	When working with report parameters, **Delete** removes the selected report. Deleted reports will have to be regenerated if deleted in error.
Destination group box	• **Preview.** Previews the predefined report on the screen. • **Print.** Sends the report to the default printer. • **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected. • **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report sys-

Common Fields and Buttons Table (continued)

Field or Button	Description
	tem (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
Edit	When working with report parameters, highlight the item in the *Selected Reports* list box and click **Edit** to modify the report details.
Exit	If a file is open with unsaved changes, a prompt displays providing the option to save the changes and exit or to exit the application without saving the changes.
Export	Exports intrusion data in a sample information file as a .REP, .TXT or .XLS file. When saved to a file, the data can be imported into other applications.
File name text box	Select a file from either the *Name* column or from the library. The file name displays in the *File name* text box. Click **Open** or double click the file name to open the file. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
From / To text boxes	When working with report parameters windows, enter the *From* and *To* range for x- and / or y-axes.
List	Provides the option to create a list of sample or report options file information, for example, file name, date / time the file was created or last edited, file identification and file status.
Name column	A list of files in the selected directory or library.
Next	Click to move to the next window or next step.
OK	Saves and closes the active window.
Open	Opens the selected file. Alternatively, double click the file name in the *Name* column to open the file.
Prev	Click to move to the previous window.
Preview	Previews predefined reports. Click the tabs at the top of the window to preview each selected report. When an analysis has not been run on a sample, this button is disabled.
Print	Sends the report to the selected destination (screen, printer or file).
Remove	Click to remove an item from the list.
Replace	Click to select another file where the values will replace the current file's values.
Replace All	Click to select another .SMP file where the values will replace all values for the active Sample Information file. The original file will remain unchanged.
Report	Click to display a window to specify report output options. • **Start Date.** Displays a calendar to select the start date for the report.

Common Fields and Buttons Table (continued)

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

OPTION PRESENTATION

Options > Option Presentation

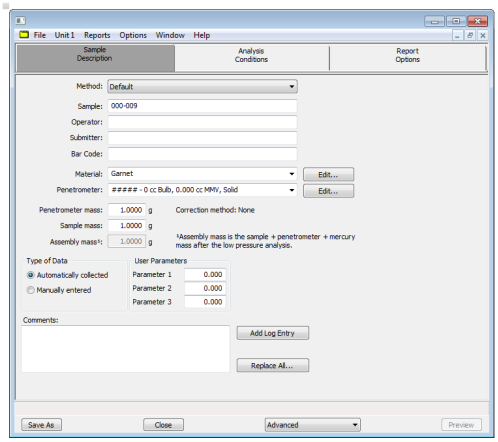
Use to change the way sample files and parameter files display: *Advanced*, *Basic*, or *Restricted*. Each display option shows sample information and options differently.

Presentation Display Table

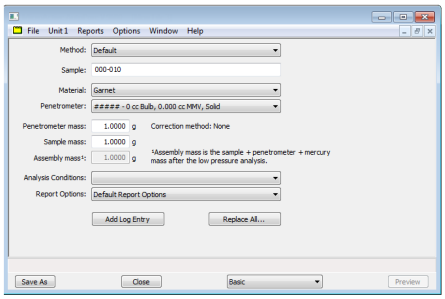
Presentation Display	Description
Advanced	Displays all parts of sample information and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been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

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

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



Advanced presentation option



Basic / Restricted presentation option

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

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 information files that are currently being used for analysis.
Entered	Sample information files containing manually entered data.
HP Complete	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 *HP Complete* 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 *HP Complete* 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 information 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
Materials properties	.MTP	Param Directory
Methods	.MTH	Methods Directory
Penetrometer properties	.PEN	Param Directory
Report options	.RPO	Param Directory
Sample file	.SMP	Data Directory
Thickness curve	.THK	Param Directory
The following file types are available when printing or exporting reports:		
Report	.REP	

File Type, Extension, and Location Table (continued)

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

APPLICATION SHORTCUTS

MENU SHORTCUTS

Shortcut menus are available for:

- the analyzer schematic when manual control is enabled
- onscreen graphs and tabular reports.

KEYBOARD SHORTCUTS

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

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

If the 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 *File* menu.
Alt + F4	Exits the program. If files are open with unsaved changes, a prompt to save changes displays.
Alt + H	Opens the *Help* menu.
Alt + I	Opens the *Options* menu.
Alt + R	Opens the *Reports* menu.
Alt + W	Opens the *Window* menu.
Shift + F9	Opens the shortcut menu of (1) selected component on analyzer schematic when manual control is enabled or (2) onscreen reports.
Ctrl + N	Opens a new sample file.
Ctrl + O	Opens the *File Selector*.
Ctrl + P	Opens the *File Selector* to start a report from a selected .SMP file.
Ctrl + S	Saves the open file.
F1	Opens the online help operator manual.

Keyboard Shortcut Table (continued)

Field or Button	Description
F2	Displays the *File Selector* window.
F3	When in the *File Selector* window, opens the file search box.
F4	When in the *File Selector* window, opens the address bar.
F6	Cascades open windows.
F7	Tiles open windows.
F8	Opens the *File Selector* to start a report from a selected .SMP file.
F9	Closes all open reports.

LIBRARIES

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

1. To locate and open a sample information file, go to ***File > Open***.
2. Click the *Sample Information* library folder on the left navigation bar.
3. Select the .SMP file on the pane on the right side of the window, then click **Open**.

MANAGE LIBRARIES

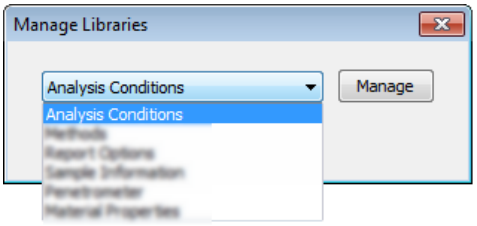
Options > Manage Libraries

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

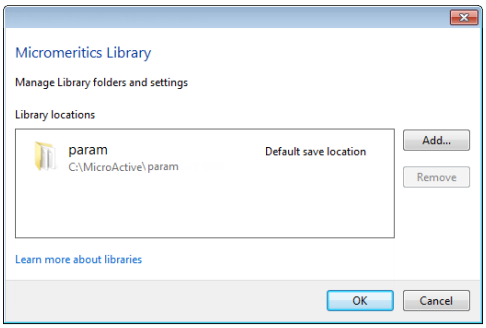
The library gathers sample and parameter files that are stored in multiple locations — such as folders on a C: drive, a network location, a connected external hard drive, or a connected USB flash drive — providing instant access at once to all of those files. Even though libraries do not store actual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

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

1. To manage folders in a library, go to ***Options > Manage Libraries***. Select the library to modify from the drop-down list, then click **Manage**.



- To add a folder to the library, click **Add** to browse and locate a folder.



- Select the folder, then click **Include folder**.
- To remove a folder, select the folder from the library locations box, then click **Remove.**

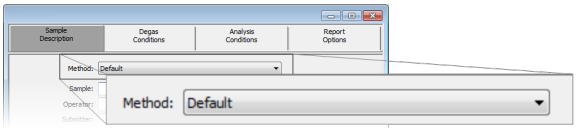
> When removing a folder from a library, the folder and its contents are not deleted from the original file storage location. However, when deleting files or folders from within a library, they are deleted from their original file storage location.

2. Click **OK** when done.

METHODS

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

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

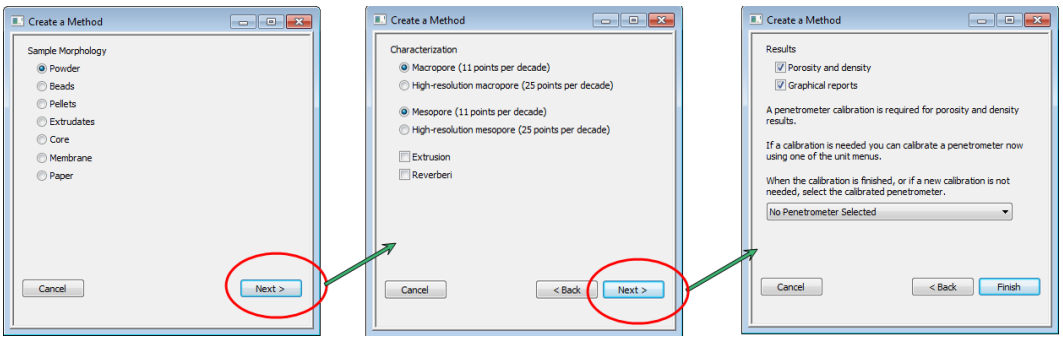


Default Method Files

Default Method Selected	Default File Modified
Mercury Porosimetry	9600.SMP

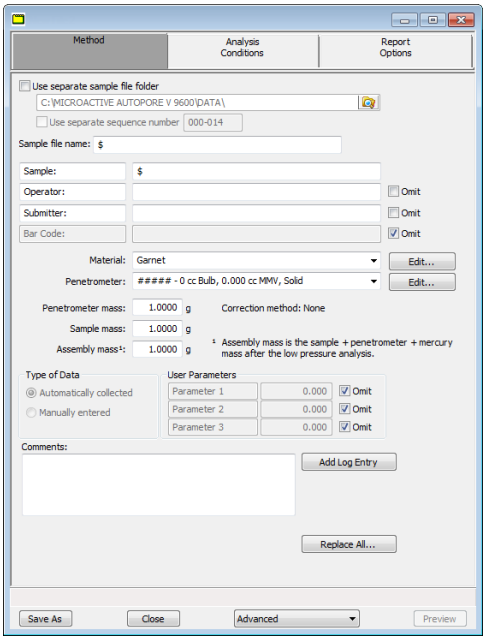
CREATE A NEW METHOD

1. On the *Create a Method* window, select the *Sample Morphology* to be used as a default method, then click **Next**.



2. Select a default *Sample Morphology*, then click **Next**.
3. Select the default *Characterization* options, then click **Next**.
4. Select the defaults *Results*:
   - **Porosity and density.** If checked, a penetrometer calibration is required. If a calibration is needed now, go to ***Unit [n] > Calibrate Penetrometer***. See *"Calibrate the Penetrometer" on page 9 - 2*.
   - **Graphical Reports.** If checked, the graphical reports are selected on the *Reports Options* tab.
5. Select the calibrated penetrometer in the drop-down list. Click **Finish**.

6. On the *Method* tab, if files created using this method are to be saved in a file folder other than the default, select *Use separate sample file folder*, then click the **Browse** icon to select a folder. The **Browse** icon is enabled only when *Use separate sample file folder* is selected. Select the new folder, then click **OK** on the *Browse for Folder* window.



7. If the file sequence numbers for this method will differ from other methods, select *Use separate sequence number*. This open is enabled only when the *Use separate sample file folder* is selected.
8. In the *Sequence Number* text box, specify an optional default alphanumeric file sequence string. This field must contain a minimum of three numbers. As files are created, this number is incrementally sequenced as a part of the file name.
9. In the *Sample file name* text box, enter an optional default file name. This information will be appended to the sequence number as a part of the file name. The **$** symbol must remain in this field.
10. In the *Sample* field text box, enter a format for the default sample identification. Include the **$** symbol to automatically include the contents of the *Sequence Number* field as part of the sample identification.
11. Enter default *Operator*, *Submitter*, and *Bar Code* identification information in the respective text boxes.

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

12. To specify a default sample material, click **Edit** to the right of the *Material* drop-down list.
13. In the *Penetrometer* drop-down list, select a penetrometer. If the required penetrometer does not appear in the list, click **Edit** and enter the description and other parameters for this penetrometer. Then go to ***File > Save As > Penetrometer*** to save these values for the next time this penetrometer is used.
14. Enter the *Penetrometer mass*, the *Sample mass*, and the *Assembly mass*.
15. In the *Type of Data* group box, indicate if the data is to be automatically collected by the system or manually entered by the operator.
16. Enter any pertinent information about the sample information file in the *Comments* text box.
17. Click **Add Log Entry** to enter notes for the analyzer log report. Create entries that cannot be recorded automatically through the software.
18. To auto-populate fields from another .SMP file, click **Replace All**, then select a .SMP file that contains the preferred parameters. Select the file, then click **Load**.
19. Click **Save As**. Select *Methods* in the library and enter a file name for the method in the *File name* text box.
20. Click **Save**.

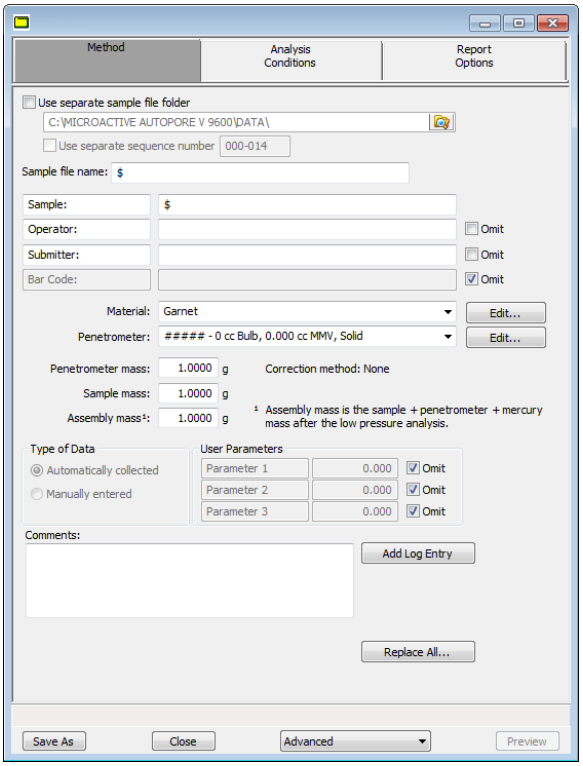
EDIT THE DEFAULT METHOD

Options > Default Method

See *"Create a New Method" on page 2 - 14*.

EDIT A METHOD

File > Open > [Method]



1. In the *File Selector*, select a .MTH file and click **Open**.
2. On the *Method* tab, if files created using this method are to be saved in a file folder other than the default, select *Use separate sample file folder*, then click the **Browse** icon to select a folder. The **Browse** icon is enabled only when *Use separate sample file folder* is selected. Select the new folder, then click **OK** on the *Browse for Folder* window.
3. If the file sequence numbers for this method will differ from other methods, select *Use separate sequence number*. This open is enabled only when the *Use separate sample file folder* is selected.
4. In the *Sequence Number* text box, specify an optional default alphanumeric file sequence string. This field must contain a minimum of three numbers. As files are created, this number is incrementally sequenced as a part of the file name.
5. In the *Sample file name* text box, enter an optional default file name. This information will be appended to the sequence number as a part of the file name. The **$** symbol must remain in this field.
6. In the *Sample* field text box, enter a format for the default sample identification. Include the **$** symbol to automatically include the contents of the *Sequence Number* field as part of the sample identification.

7. Enter default *Operator*, *Submitter*, and *Bar Code* identification information in the respective text boxes.

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

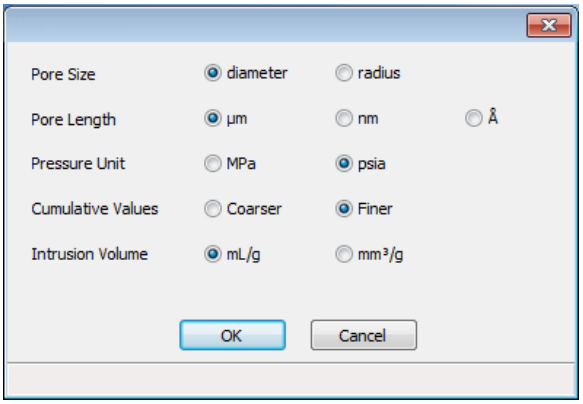
8. In the *Penetrometer* drop-down list, select a penetrometer. If the required penetrometer does not appear in the list, click **Edit** and enter the description and other parameters for this penetrometer. Then go to ***File > Save As > Penetrometer*** to save these values for the next time this penetrometer is used.
9. In the *Mass* group box, indicate if mass is to be manually entered by the operator (*Enter*) or calculated by the system (*Calculate*).
10. In the *Type of Data* group box, indicate if the data is to be automatically collected by the system or manually entered by the operator.
11. The optional user-defined fields in the *User Parameters* group box may be used to enter and track information from another analyzer or source, along with other statistical process control (SPC) data.
12. Use the *Comments* text box to enter notes about the Method.
13. After completing the *Sample Description* tab select the parameter tabs across the top portion of the window to edit other sample information file parameters. The saved parameter settings become the defaults for new sample files when this method is selected.
14. Click **Save**, then click **Close**.

CONFIGURE THE ANALYZER

SPECIFY UNIT SELECTIONS

Options > Units

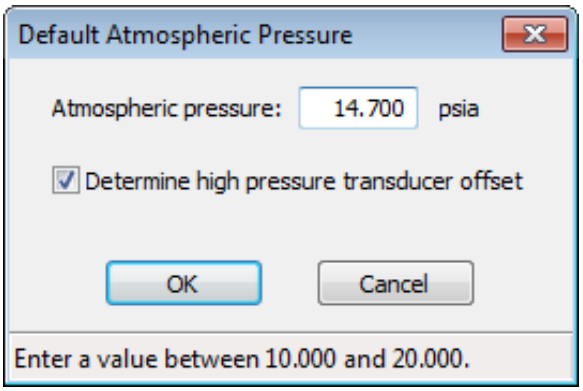
Use to specify how data should appear on the application windows and reports. This menu option is not available if using *Restricted* presentation option.



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).

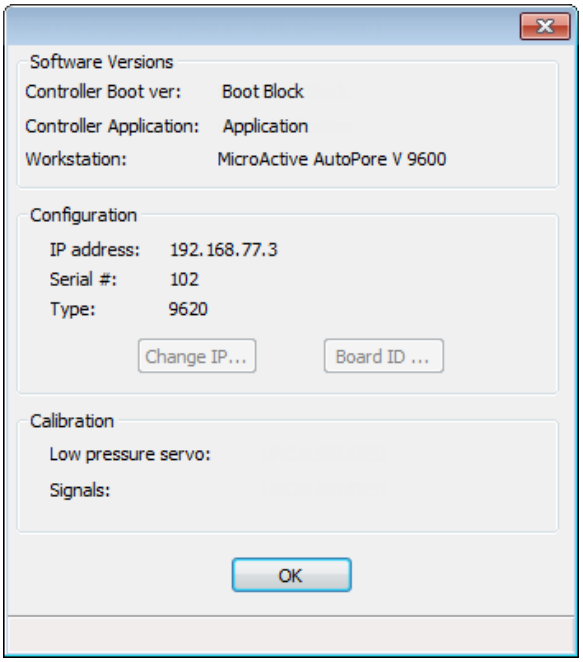
Select *Determine high pressure transducer offset* to enable the system to determine automatically the high pressure transducer offset.

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

UNIT CONFIGURATION

Unit [n] > Unit Configuration

Use to display hardware / software configurations.



Unit Configuration Fields and Buttons Table

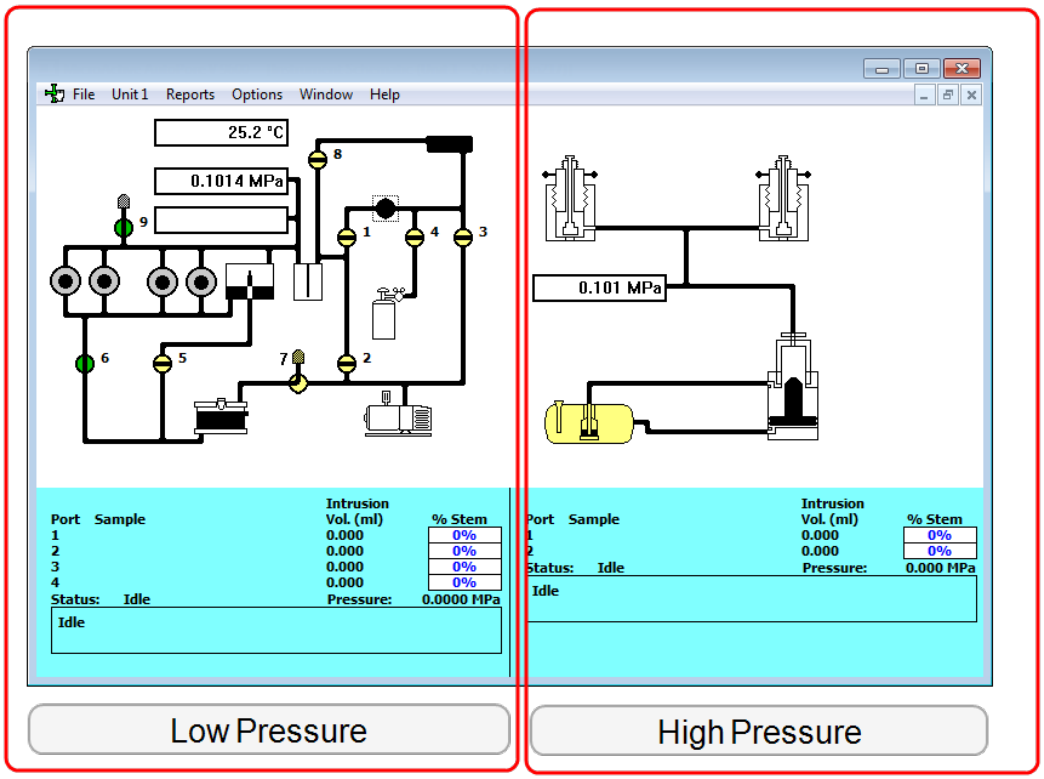
Field or Button	Description
Calibration *group box*	Displays the dates of the last high and low pressure servo calibrations and signals.
Configuration *group box*	Displays the IP address used by the analysis program and the serial number of the selected analyzer. • **Change IP**. Click to display the *Unit IP Setup* window. The IP address and Subnet mask assigned during installation display. Do not edit these fields unless instructed by a Micromeritics service representative. • **Board ID**. Click to read the board ID. These parameters cannot be edited.
Software Versions *group box*	Displays the software versions of the MIC BIOS, controller, and analysis program.

ANALYZER 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.

Analyzer Schematic Icon Table (continued)

Icon or Symbol	Description
	Servo Valve. Open.

Low Pressure Schematic Icons

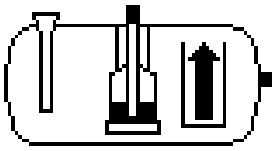
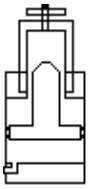
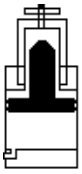
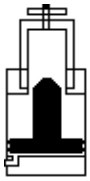
Mercury Degasser. Displays the mercury level.			
Drained	Partially Filled	Filled	Overfilled. This alarm displays with a red background. This is an alarm state.

Mercury Trap. Displays the state of the mercury trap.	
Empty	Contains more than 6 mm of mercury. This is an alarm state.

Mercury Reservoir. Displays the level of mercury in the reservoir.	
If the mercury level is low, see *"Maintain Mercury Level" on page 11 - 15*.	Level is OK.

Hg Reservoir Vacuum Switch	
Hg Reservoir Evacuated	When illuminated, indicates that mercury reservoir has been evacuated.

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**

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.

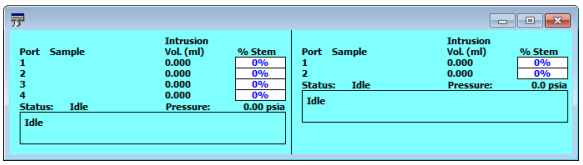
Schematic Shortcuts Table

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

SHOW STATUS

Unit [n] > Show Status

Use to show the current status for each port.



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

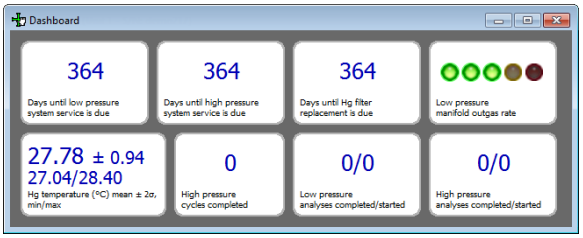
SHOW DASHBOARD

Unit [n] > Show Dashboard

The dashboard displays the following:

- Number of 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 ± 2σ, 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.



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

Dashboard Gauges and Descriptions Table (continued)

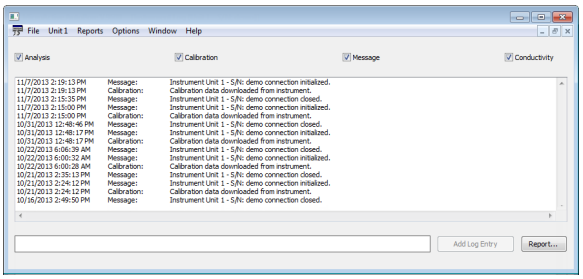
Field or Button	Description
	negative numbers display if maintenance is past due.
Hg temperature	Displays the statistics of the manifold temperature reading. The mean, the value at two standard deviations, the minimum, and the maximum display.
High pressure analyses completed / started	Displays N/M where 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	Provides the qualitative indication of the outgas rate in the dosing manifold. LED images constitute a bidirectional bar graph of the outgas rate. • 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.

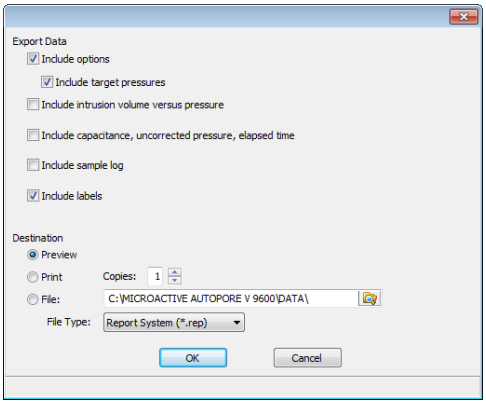
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

EXPORT FILES

File > Export

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

1. Select one or more files from the library. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.
2. Click **Export**.
3. In the *Export Options* window, select the type of data to include in the export file.



Types of data that can be included:

- Options
  - Target pressures
- Intrusion volume versus pressure
- Capacitance, uncorrected pressure, elapsed time
- Sample log
- Labels

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

- **Preview.** Previews the predefined report on the screen.
- **Print.** Sends the report to the default printer.
- **Copies.** Select the number of copies to print. This field is only enabled when *Print* is selected.

- **File.** Select the destination directory. Enter a new file name in the *File name* field, or accept the default. Select to save the file as a report system (.REP), a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.

5. Click **OK**.

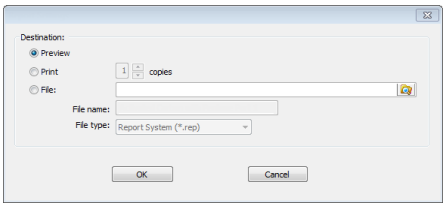
LIST FILES

File > List

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

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

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



4. Click **OK**.

3 About Sample Files

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

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

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

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

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

Create Sample Files

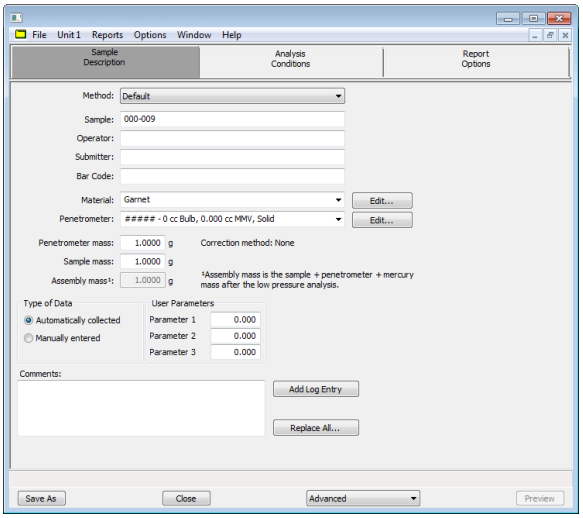
Create Sample Files in Advanced Presentation Option

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

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

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

1. Go to ***Options > Option Presentation > Advanced*** and ensure *Advanced* has a checkmark.
2. To create a new sample file, go to ***File > New Sample***, or go to ***File > Open*** and select a sample file.
3. Select a method from the *Method* drop-down list.



4. Select a method from the *Method* drop-down list.
5. Enter a sample description in the *Sample* text box.
6. Enter *Operator*, *Submitter*, and *Bar Code* identification information in the respective text boxes. This information will display on the *Sample Description* tab of new sample information files. This option may not display (or may have a different field label) if modified in the method from which the sample file was created, either through ***Options > Default Method*** or ***File > Open > Method***.
7. To select the sample material, click the *Material* drop-down arrow. Alternatively, click **Browse** and locate the file.
8. In the *Penetrometer* drop-down list, select a penetrometer. If the required penetrometer does not appear in the list, click **Edit** and enter the description and other parameters for this tube. Then go to ***File > Save As > Penetrometer File*** to save these values for the next time this penetrometer is used.
9. The *Penetrometer Mass* field auto-populates with the value from the selected *Penetrometer Properties* file. If the value shown is not correct, enter the correct value.
10. Enter the *Sample Mass*. The *Assembly Mass* entry is disabled until a low pressure analysis has been completed on the sample.
11. In the *Type of Data* group box, indicate if the data is to be automatically collected by the system or manually entered by the operator.
    - **Automatically collected.** Select for all sample runs where the data are collected.
    - **Manually entered**. Select when another sample has been run on a different analyzer or different model analyzer so that data can be analyzed or used for comparison. If *Manually entered* is selected, the data are entered in the Intrusion interactive report.
11. The optional user-defined fields in the *User Parameters* group box may be used to enter and track information from another analyzer or source, along with other statistical process control (SPC) data.

12. Enter any pertinent information about the sample information file in the *Comments* text box. Entered comments are displayed in the report header.
13. Click **Add Log Entry** to enter notes for the analyzer log report. Create entries that cannot be recorded automatically through the software.
14. To auto-populate fields from another .SMP file, click **Replace All**, then select a .SMP file that contains the preferred parameters. Select the file, then click **Load**.
15. After completing the *Sample Description* tab, click the other parameter tabs to edit more sample information file parameters.See *"About Parameter Files" on page 4 - 1*.
16. Click **Save As** to save as a different file name.The file can also be saved as a different file type to save part of the sample file as a new parameter set such as Analysis Conditions, Report Options, etc.

Sample File Fields and Buttons Table

Field or Button	Description
Assembly mass	This field is entered after the low pressure analysis is completed.. Sample + penetrometer + mercury mass after the low pressure analysis.
Comments	Enter comments about the sample or analysis. Comments display in the report header.
Material	Select the material to be analyzed from the drop-down list. See *"Material Properties" on page 4 - 9*.
Method	Select a method from the drop-down list. See *"Methods" on page 2 - 14*.
Operator	Enter operator identification information. This field label may have been renamed or may not display if modified in ***Options > Default Methods.***
Penetrometer	Select a penetrometer file from the drop-down list, or click **Edit** to modify or create a new Penetrometer file. See *"Penetrometer Properties" on page 4 - 10*.
Sample	Enter a sample description.
Submitter	Enter submitter identification information. This text box may have been renamed or may not display if modified in ***Options > Default Methods.***

Sample File Fields and Buttons Table (continued)

Field or Button	Description
Type of Data group box	• **Automatically collected.** Select if the type of data will be automatically collected by the system while an analysis is running. • **Manually entered.** Use to enter data manually that was collected from another source. If *Manually entered* is selected, the Intrusion Report becomes available in the *Basic/Advanced* drop-down list for pasting or importing data into the file. See *"Manually Enter Data" on page 3 - 10*.
User Parameters group box	These fields are primarily used for the SPC (Statistical Process Control) reporting to specify sample characteristics or its manufacturing process but may be used for other data by entering specific analysis conditions or sample criteria. The entered parameters display on the *Summary Report*. This option may not display (or may have a different field label) if modified in the method from which the sample file was created, either through ***Options > Default Method*** or ***File > Open > Method***.

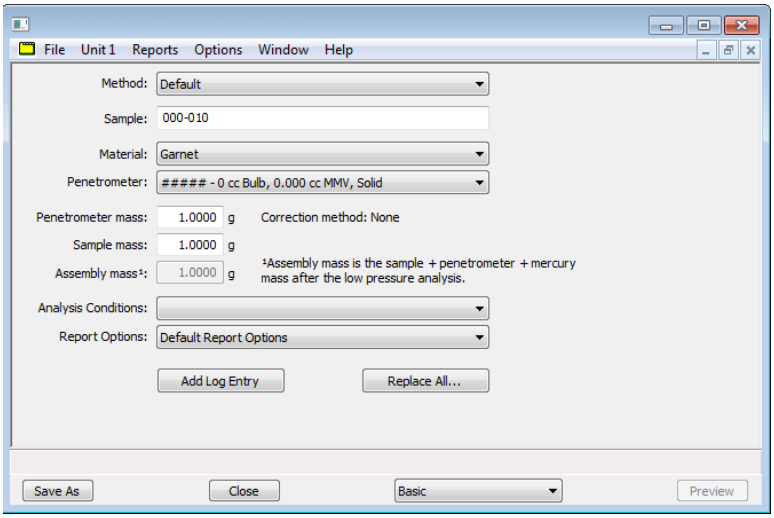
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

CREATE SAMPLE FILES IN BASIC PRESENTATION OPTION

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

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

1. Go to ***Options > Option Presentation > Basic*** (or ***Restricted***).
2. To create a new sample file, go to ***File > New Sample***, or go to ***File > Open*** and select a sample file.



3. Select a method from the *Method* drop-down list.
4. In the *Sample* field, enter a sample description.
5. Select a sample material from the *Material* drop-down list. Alternatively, click **Browse** and locate the file.
6. Select a penetrometer from the *Penetrometer* drop-down list. Alternatively, click **Browse** and locate the file.
7. The *Penetrometer Mass* field auto-populates with the value from the selected *Penetrometer Properties* file. If the value shown is not correct, enter the correct value.
8. Enter the *Sample Mass*. The *Assembly Mass* entry is disabled until a low pressure analysis has been completed on the sample.
9. Click the down arrows to select default parameter files for *Analysis conditions*, and *Report options*.
10. To auto-populate fields from another .SMP file, click **Replace All** and select a .SMP file that contains the necessary parameters. Select the file and click **Replace**.

11. Click **Add Log Entry** to enter notes for the analyzer log report. Create entries that cannot be recorded automatically through the software, for example, when the port filter was changed.
12. Click **Save**, then click **Close**.The file can be retrieved later from the *Sample Information* folder in the library.

CREATE SAMPLE FILES IN RESTRICTED PRESENTATION OPTION

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

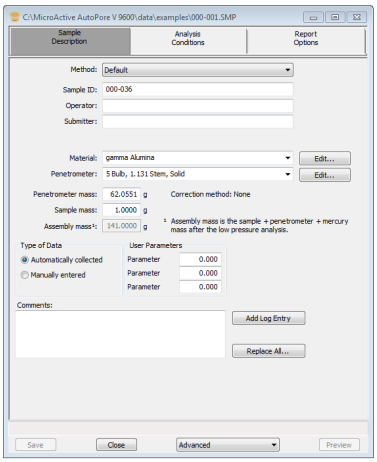
OPEN A SAMPLE FILE

File > Open > [.SMP File]

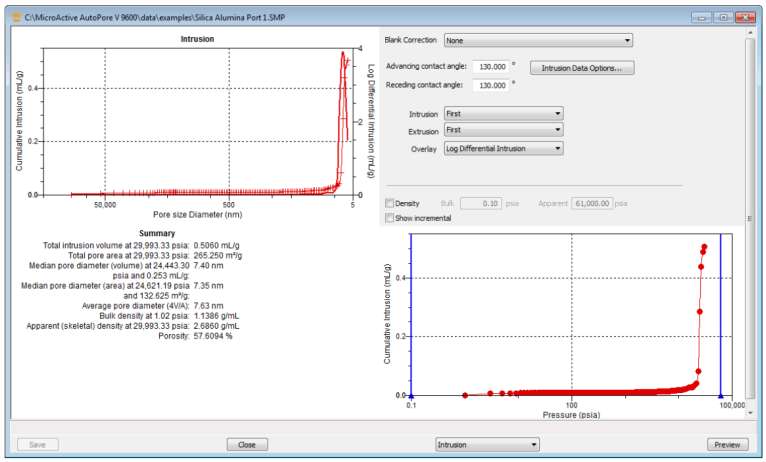
- When working with an existing file, it is recommended that a copy of the file be used rather than the original.
- Columns on the *File Selector* window can be sorted by clicking the column header. To sort the file list by status, click the *Mic Status* column header.

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

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



Example of tabbed file editor



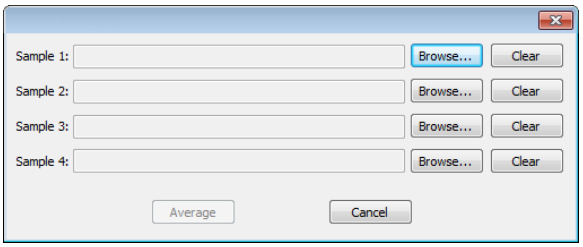
Example of MicroActive Report window

SAMPLE AVERAGING

File > Average

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

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



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

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

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 a sample file. A sample file must be created even if there is no sample or if the sample is non-porous.
2. Go to ***File > Open*** and enter the new sample file name.
3. Click **Open**.
4. Create the sample file. Enter zero for the sample mass.
5. Perform the low and high pressure analysis. See *"Perform a Low Pressure Analysis" on page 5 - 9* and *"Perform a High Pressure Analysis" on page 5 - 14*.

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 - 14*.

FORMULA CORRECTION

Through large numbers of blank runs using a variety of pressure tables and penetrometers filled with mercury, Micromeritics has developed a formula for correction of each penetrometer type. 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.

If using the formula correction, perform several trial blank runs to ensure that the formula provides an acceptable degree of correction for the sample type(s) and analysis conditions.

To achieve the most accurate formula correction, accurate values for Sample mass, Assembly mass, and Penetrometer mass must be entered. The calibrated volume of the penetrometer must also be provided. Either an Assembly mass or a Penetrometer volume of 1.0 disables the mercury compression form of formula correction.

COMBINE A PENETROMETER CALIBRATION AND BLANK CORRECTION FILE CREATION

1. Calibrate the penetrometer. See ***"Calibrate the Penetrometer" on page 9 - 2***. Enter the analysis conditions and pressure table to be used when samples are analyzed with this blank correction file.
2. After the low pressure run, do not empty the penetrometer. Weigh it and install it in the high pressure port.
3. Create blank correction files by completing the high pressure run. See ***"Create Blank Correction File" on the previous page***.
4. Create a new *Penetrometer Properties* file for this penetrometer. See ***"Penetrometer Properties" on page 4 - 10***. From the file selection list, choose the default file for this type of penetrometer. Change the identifier to include the penetrometer's 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

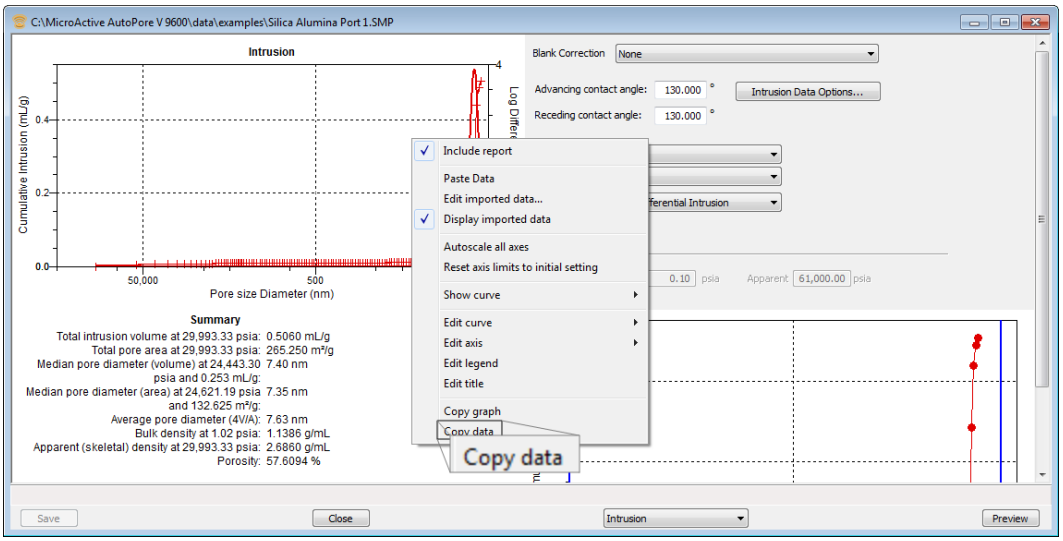
The images shown in this section may differ slightly from yours. However, the process is the same unless otherwise noted.

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

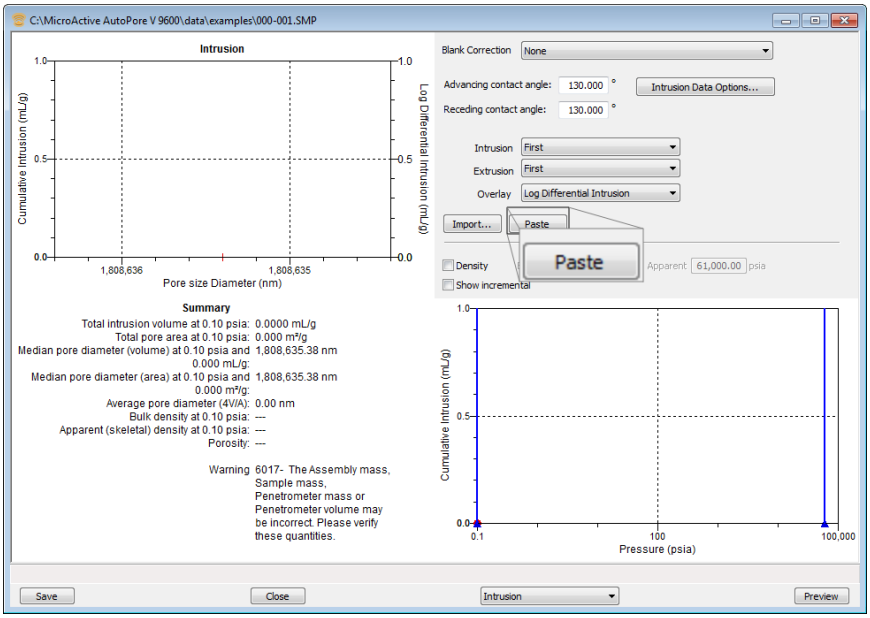
- Copy and paste onto the graph area of the interactive window
- Import data into the interactive window

COPY AND PASTE MANUALLY ENTERED DATA

1. Go to ***File > Open [.SMP file]***, then select the sample information file with a *Complete* status that contains the data to be copied and pasted.
2. Click **Open**. The file will open to the interactive reports window.
3. Right click in the graph area of the interactive reports window, then select *Copy Data*. This will copy the data from the active file to the clipboard.



4. Go to ***File > New Sample***, then open a new sample information file. To save the file as a new file name, go to ***File > Save As***, then enter a new file name in the *File name* text box.
5. On the *Sample Description* tab, select *Manually entered* in the *Type of Data* group box.
6. Click the *Advanced* down arrow at the bottom of the window, then select *Intrusion* .
7. Resize the interactive window to display the **Paste** button.



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

IMPORT MANUALLY ENTERED DATA

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

ASCII text file format rules

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

For Mercury Porosimetry:

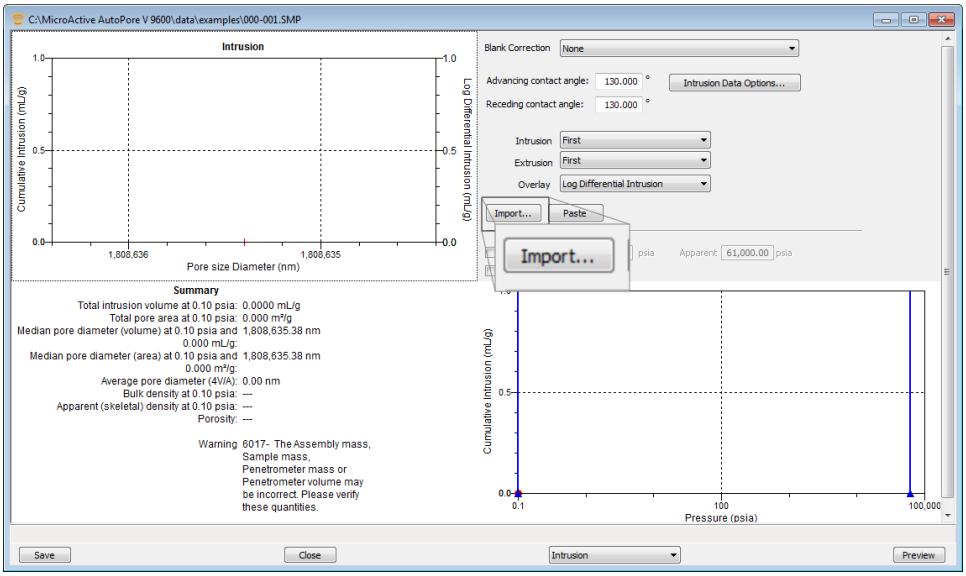
- Pressure (psi)
- Pressure (MPa)
- Pressure (kPa)
- Pressure (Pa)
- Pressure (bar)
- Pressure (mbar)
- Pressure (Atm)\
- Intrusion (cm^3)
- Intrusion (cm^{263})
- Intrusion (ml)
- Intrusion (mm^3)
- Intrusion (mm^{263})

Sample Mercury Porosimetry ASCII text file

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

To import the ASCII text file:

1. Go to ***File > New Sample***, then open a new sample information file. To save the file as a new file name, go to ***File > Save As***, then enter a new file name in the *File name* text box.
2. Click the down arrow at the bottom of the window and select *Advanced*.
3. On the *Sample Description* tab, select *Manually entered* in the *Type of Data* group box.
4. Click the *Advanced* down arrow at the bottom of the window, then select *Intrusion.*.
5. Resize the interactive window until the **Import** button displays.



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

Blank Page

4 About Parameter Files

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

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

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

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

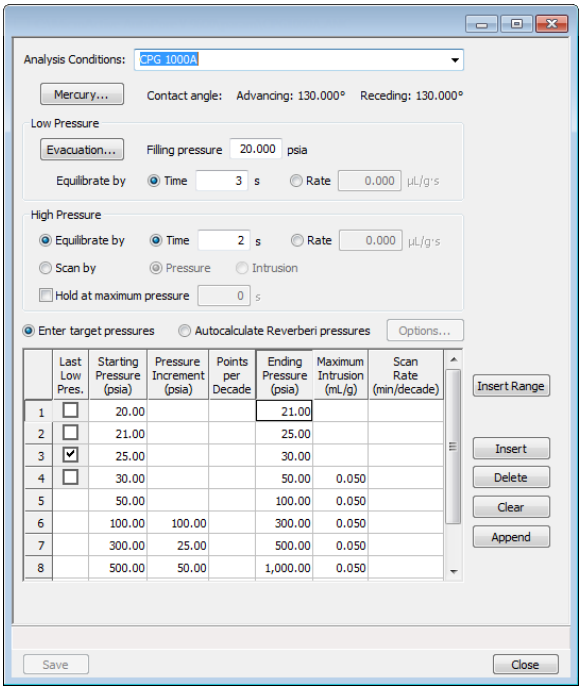
Analysis Conditions

File > Open > [.ANC File]

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

Analysis conditions specify the data used to guide an analysis.

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



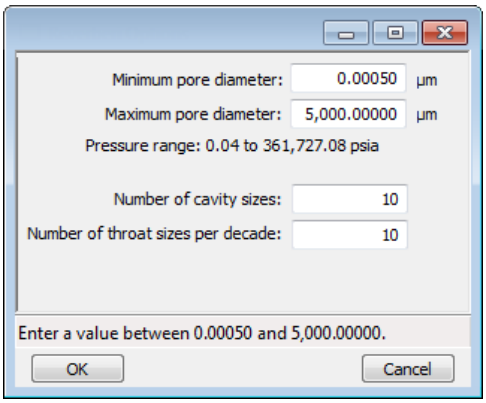
2. To overwrite analysis conditions with parameters from another *Analysis Conditions* file, click the *Analysis Conditions* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file.
3. To edit the *Contact angle* parameters, click **Mercury** to enter mercury properties.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

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

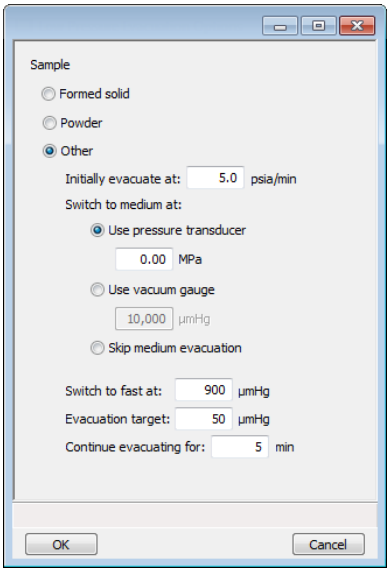
If the *Last low Pressure* checkbox is selected, the *Ending Pressure* for this row is the *Last Low Pressure Point*. The *Last Low Pressure* column must have only one checkbox selected. Only rows before the first row with an *Ending Pressure* greater than 50 psia are eligible and will have a checkbox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

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

Analysis Conditions Fields and Buttons Table

Button	Use to Specify...
Autocalculate Reverberi pressures	Select to have the application automatically use the Reverberi pressures. Selecting this option disables the pressures table. Click **Options** to enter additional Reverberi options. 
Enter target pressures	Select to manually enter pressures into the table.
High Pressure group box	**Equilibrate by.** Select the option for equilibration based on elapsed *Time* (in seconds) or decrease in *Rate* of intrusion (or extension) in mL/g per second. **Scan by:** • **Pressure**. The instrument goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment ends at the specified pressure. The pressure is programmed to increase or decrease at a rate to give a constant time per decade of pressure. Along the way, the instrument takes intrusion points at the specified number of points per decade, ending at the specified ending pressure. Also, any points in the pressure table, as well as points separated by the maximum intrusion volume, are collected. • **Intrusion**. The instrument goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment

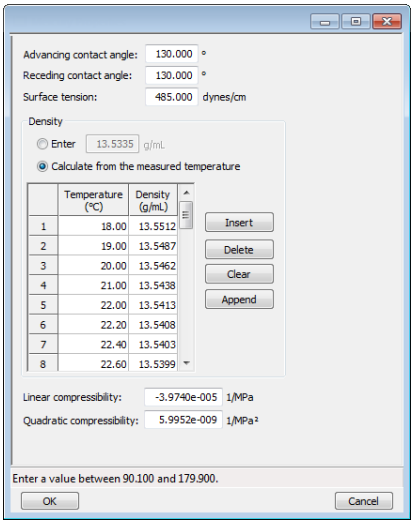
Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	ends at the specified pressure The pressure rate is the maximum achievable safe rate (up to 0.5 min/decade) and is programmed to increase or decrease at a rate to give a constant intrusion/extrusion rate. The instrument takes intrusion points at the specified number of points per decade, ending at the specified ending pressure. Also collected are points in the pressure table, as well as points separated by the maximum intrusion volume. If both high pressure ports are in use, they may have different intrusion rates. In this case, the left port (port 1) determines when data are collected. The right port collects data at the same times as the left. This allows a differential analysis to be performed in this mode. • **Hold at maximum pressure.** Enter additional amount of time to remain at the maximum pressure in a pressure table intrusion segment before beginning extrusion..
Low Pressure group box	**Evacuation**. Select if the sample is a *Formed solid*, *Powder*, or *Other*. If *Other* is selected, the remaining fields are enabled. This window is also available from ***Unit [n] > Evacuate Low Pressure***.  • **Initially evacuate at**. Enter the initial maximum evacuation rate. • **Switch to medium at**. Enter the method and pressure the system must reach before medium evacuation begins. ○ Use pressure transducer

Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	◦ User vacuum gauge ◦ Skip medium evacuation • **Switch to fast at.** Enter the pressure the system must reach before fast evacuation begins. • **Evacuation target.** Enter the evacuation pressure. • **Continue evacuating for**. Enter the evacuation duration. **Filling pressure.** The penetrometer is filled with this pressure prior to data collection. It is recommended to set the filling pressure slightly lower than the first low pressure point on the pressure table. A filling pressure of at least 0.5 psia is recommended. Because mercury generates pressure and because fill pressures less than 0.5 psia can fail to fill the corner radii and gaps between the glass and sample in the penetrometer, using a lower pressures may reduce the accuracy of data. If the filling pressure is higher than any point in the table, an error message occurs. Delete the pressures lower than the filling pressure or change the filling pressure. **Equilibrate by.** Select the option for equilibration based on elapsed *Time* (in seconds) or decrease in *Rate* of intrusion (or extension) in mL/g per second.
Mercury	Enter the mercury properties. Mercury properties may change with variations in temperature.

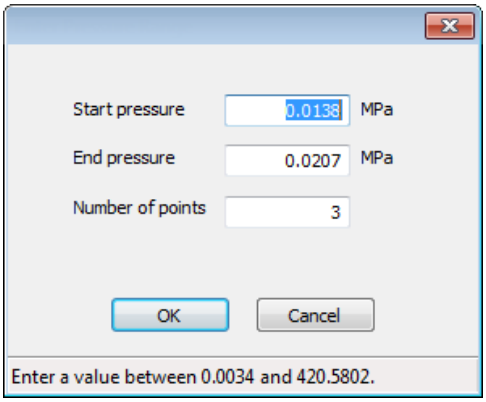
Analysis Conditions Fields and Buttons Table (continued)

Button	Use to Specify...
	 **Advancing contact angle.** Enter the advancing (intrusion) contact angle. **Receding contact angle**. Enter the receding (extrusion) contact angle. **Surface tension**. Enter the surface tension of mercury. **Density.** • **Enter**. Select to manually enter a density. • **Calculate from the measured temperature**. Select to use the entries in the table. • **Linear compressibility**. Enter the linear compressibility coefficient. • **Quadratic compressibility**. Enter the quadratic compressibility coefficient.
Table Options	**Last low pressure**. Select the rows to indicate the last low pressure. **Starting Pressure.** This column is not editable. The data are taken from the *Ending Pressure* of the preceding row and the *Filling Pressure* for the first row. **Pressure Increment**. Enter the pressure increment for this segment if a sequence of linearly spaced pressures is preferred. Either the *Pressure Increment* or *Points Per Decade* can be specified. If one is entered, the other is automatically set to zero and displayed as blank. By default, both columns are blank (zero) when a new row is inserted or appended.

Analysis Conditions Fields and Buttons Table (continued)

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

Analysis Conditions Fields and Buttons Table (continued)

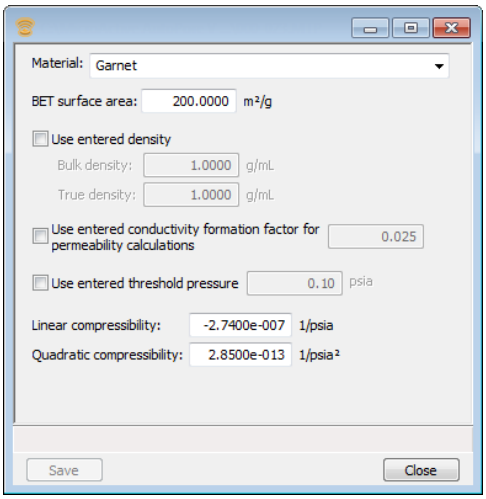
Button	Use to Specify...
	
	For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

MATERIAL PROPERTIES

File > Open > [.MTP File]

Material properties specify the properties of the material to be used in an analysis.

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



2. In the *Material* drop down, select the material being analyzed. To overwrite material properties with parameters from another *Material Properties* file, click the *Material Properties* down arrow and select a file from the list. Alternatively, click **Browse** and locate the file.
3. Enter the *BET surface area.*
4. If using the entered density, select the *Use entered density* checkbox and enter the bulk and / or true density in the appropriate text box.
5. If using the entered conductivity formation factor for permeability calculations, select the *Use entered conductivity formation factor for permeability calculations* check box and enter the factor in the text box.
6. If using the entered threshold pressure, select the *Use entered threshold pressure* checkbox and enter the pressure in the text box.

7. Enter the *Linear compressibility* and *Quadratic compressibility* pressures in the appropriate text boxes. These values can be copied from the *Compressibility Report* after a sample of the material has been analyzed.
8. Click **OK** to save the changes or click **Cancel** to close the window.

Material Properties Fields and Buttons Table

Field or Button	Description
BET Surface Area	Enter the BET surface area.
Linear compressibility	Enter the linear compressibility coefficient.
Quadratic compressibility	Enter the quadratic compressibility coefficient.
Use entered conductivity formation factor...	Select if using the entered conductivity formation factor for permeability calculations and enter the factor in the text box.
Use entered density	Select if using the entered density and enter the bulk and /or true density in the appropriate text boxes.
Use entered threshold pressure	Select if using the entered threshold pressure and enter the threshold pressure in the text box.

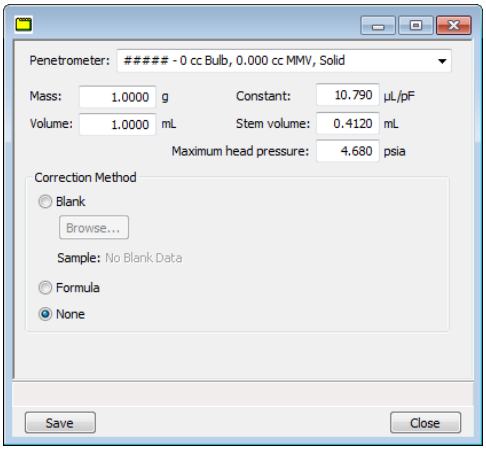
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

PENETROMETER PROPERTIES

File > Open > [.PEN File]

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

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



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

Penetrometer Properties Fields and Buttons Table

Field or Button	Description
Maximum head pressure	Enter the maximum head pressure. See *"Select a Penetrometer" on page 5 - 1*.
Constant	Enter the penetrometer constant provided with the penetrometer. Verify the field contents if the **Replace** option has been used.
Correction Method	**Blank**. If using the blank correction method, click **Browse** to select a sample file. See *"Create Blank Correction File" on page 3 - 8*. **Formula**. See *"Formula Correction" on page 3 - 9*. **None**.
Mass	Mass of the empty, assembled penetrometer (excluding the spacer).
Penetrometer	Enter identifying information for this file.

Penetrometer Properties Fields and Buttons Table (continued)

Field or Button	Description
Stem volume	Enter the penetrometer stem volume provided with the penetrometer.
Volume	Enter the volume of the penetrometer. This is required to calculate density or when using the blank correction formula. See *"Calibrate the Penetrometer" on page 9 - 2*.

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

REPORT OPTIONS

File > Open > [.RPO File]

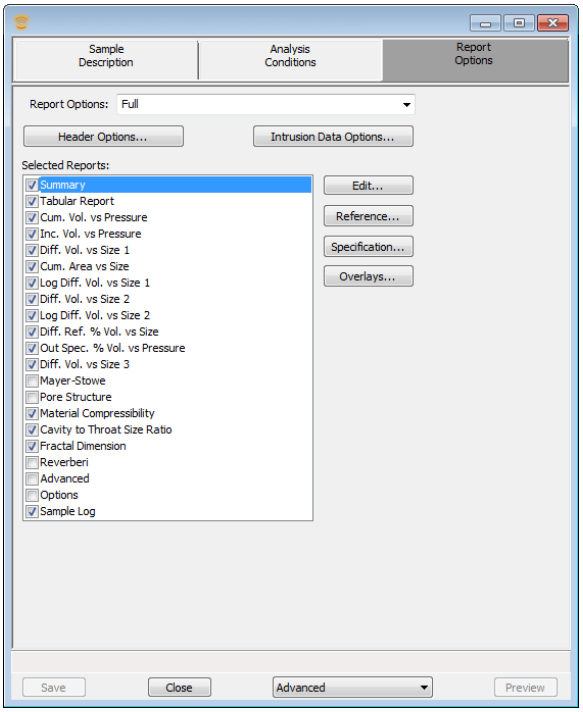
(or click the *Report Options* tab when in *Advanced* format)

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, column headings, and components of thickness curve equations. These files may contain tabular reports, plots, or both, as well as user-defined report tables.

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

Report Options files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis. See *"Overlay Multiple Graph Options" on page 6 - 22*.

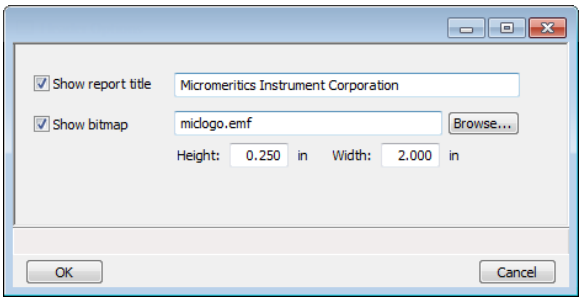
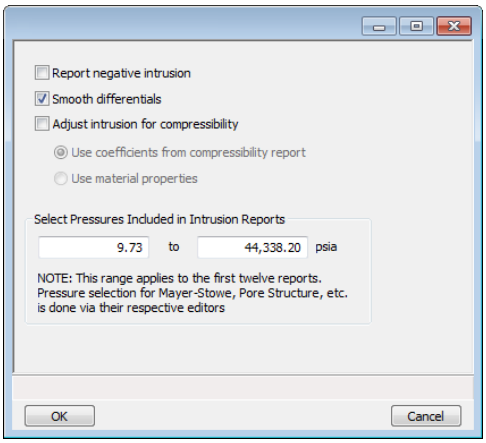
1. Go to ***File > Open***.
   - Select the appropriate library folder for the parameter file type, then select a file name in the list or enter it in the *File Name* field, then click **Open**, or
   - Select the appropriate file type from the drop-down list on the lower right portion of the window, then select a file name in the list or enter it in the *File Name* field, then click **Open.**
2. To overwrite report options with parameters from another *Report Options* file, on the *Report Options* tab, click the *Report Options* down arrow, then select a file from the list. Alternatively, click **Browse** and locate the file.



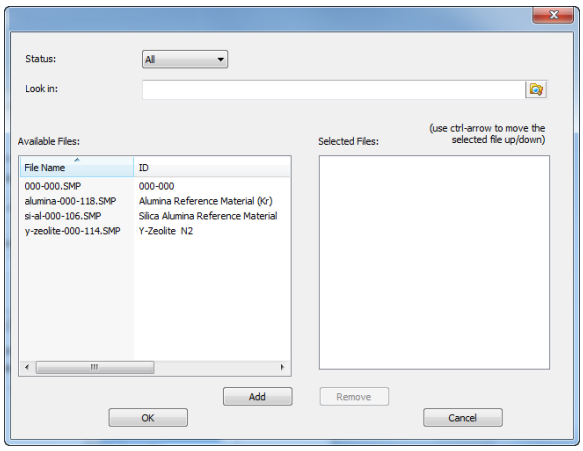
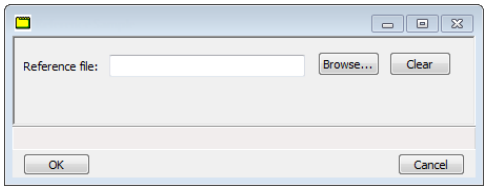
3. To have a report title display on the report header, select **Header Options**, then enter a title to appear on the report header.
4. To have a graphic display on the report header, select *Show graphic* to insert a graphic in the report header. Click **Browse** to locate a .BMP or a .EMF file. Specify the graphic size in the *Height* and *Width* text fields.
5. The *Selected Reports* list box displays the reports that may be generated.
   - Select checkboxes to the left of the reports to include in this file.
   - To specify report options, highlight the report in the *Selected Reports* list box, then click **Edit**. Make changes as necessary. Click **OK**.

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

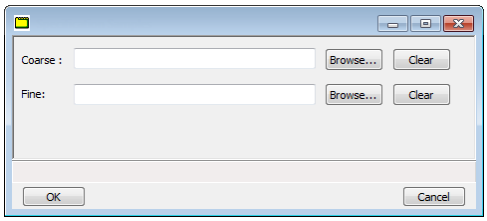
Report Options Fields and Buttons Table

Field or Button	Description
Edit	Edit graph options. • **Plot Points.** Select to plot points on the graph.. • **Plot Curve.** Select to plot curves on the graph.. • **Show Histogram.** Select to show the graph as a histogram. When selected, the *Plot Points* and *Plot Curve* selections are disabled.
Header Options	 **Show report title**. Enter a report title to appear on the report header. **Show bitmap.** Displays the selected graphic on the report header. Click **Browse** to locate the graphic in either .BMP or .EMF format. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Intrusion Data Options	 **Adjust intrusion for compressibility:** • **Use coefficients from compressibility report.** Select to have the application use the coefficients from the *Material Compressibility* report rather than from the *Material Properties*.

Report Options Fields and Buttons Table (continued)

Field or Button	Description
	• **Use materials properties.** Select to have the application use the parameters from *Material Properties* rather than from the *Material Compressibility* report. **Report negative intrusion.** Select to report small incorrect polarities (negative intrusions or positive extrusions) which may indicate the presence of noise, improper blank correction, or instrument malfunction. **Select Pressures Included in Intrusion Reports.** Enter range of pressure. This range applies to the first twelve reports on the options list. **Smooth differentials.** Select to apply smoothing to any differentials reported in tables or graphs.
Overlays	Select the files to overlay. 
Reference	Selects a sample file to compare analysis results with the current sample. 

Report Options Fields and Buttons Table (continued)

Field or Button	Description
Reports list box	Select the report names to include in the report.
Specification	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. 

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

5 Perform an Analysis

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

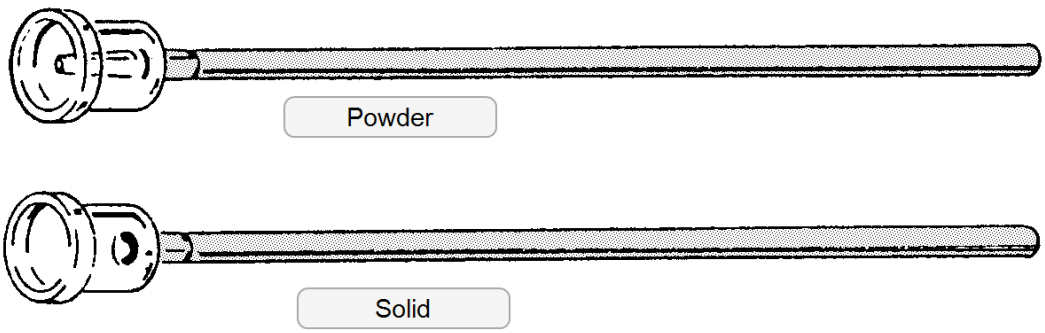
Prepare for Analysis

Create the sample file. A complete sample file must be created for each sample before beginning analysis on that sample.

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.

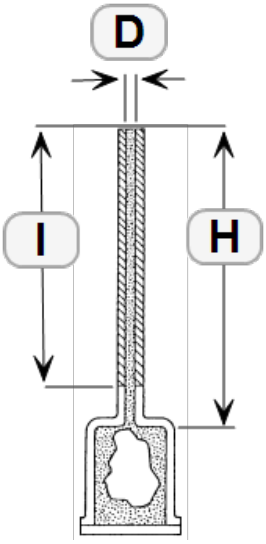
Penetrometer Selection Guide

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

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	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
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)(D2)(I)/4] × [0.001 cm^3/mm^3]
- Total Stem (Capillary) volume = [(3.14)(D2)(H) /4] × [0.001 cm^3/mm^3]
- Maximum Head Pressure (psia) = [H] × [0.01934 psia/mmHg]
- Maximum Head Pressure (MPa) = [H] × [0.000133 MPa/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:

Volume of sample = mass/density = (29g)/(8.9g/cc) = 3.26 cc

Approximate pore volume = fractional pore volume × sample volume =
0.20 (3.26 cc) = 0.652 cc

Approximate total volume = volume of pores + volume of sample
(3.26 + 0.652) cc = 3.91 cc

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 cc/1.131 cc) × 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%.

Volume of sample = mass/density = (15 g)/(3.5 g/cc) = 4.29 cc

Approximate pore volume of material = fractional porosity x sample volume =0.03 (4.29 cc)
= 0.13 cc

Approximate volume of interstice = (4.29 cc + 0.13 cc) (40/60) = 2.95 cc

Total volume of powdered sample = 4.29 cc + 0.13 cc + 2.95 cc = 7.37 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 × 15/7.37]. The penetration volume would then be about 0.26 cc [0.13 × 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 mm diameter, whereas a filling pressure of 1.5 psia will fill cavities as small as 120 mm 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 instrument maintenance, as well as improved data quality, dry the sample material in a shallow pan at 150 ºC 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. Cleaning the penetrometer.
2. Loading the sample.
3. Sealing the penetrometer.
4. Weighing the penetrometer assembly.

Clean the Penetrometer

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

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

CAUTION

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 - 31*
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 enure 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 I - 7*.
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.

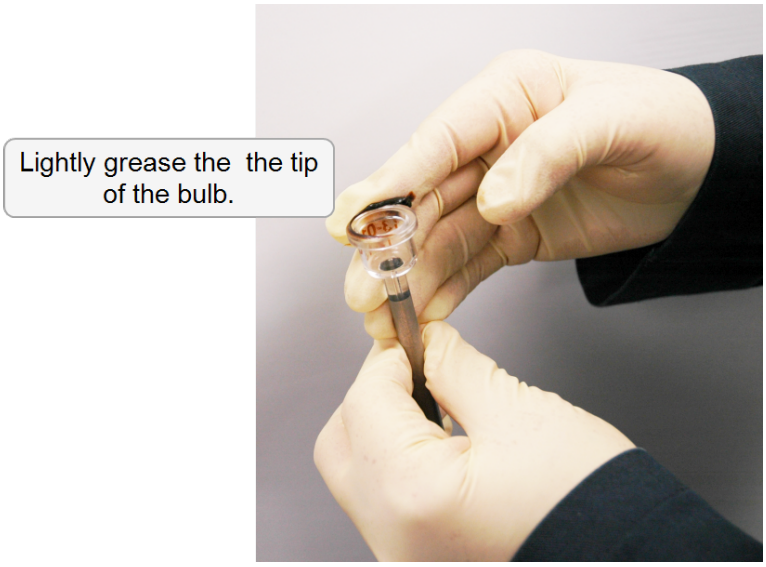
Seal the Penetrometer

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 the previous page*.

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.

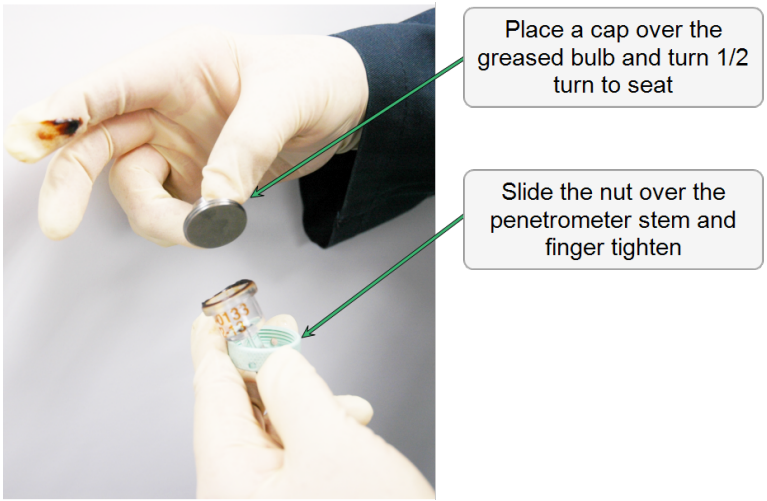


   a. Use a fingertip to apply a light coating of grease to the lip of the bulb.

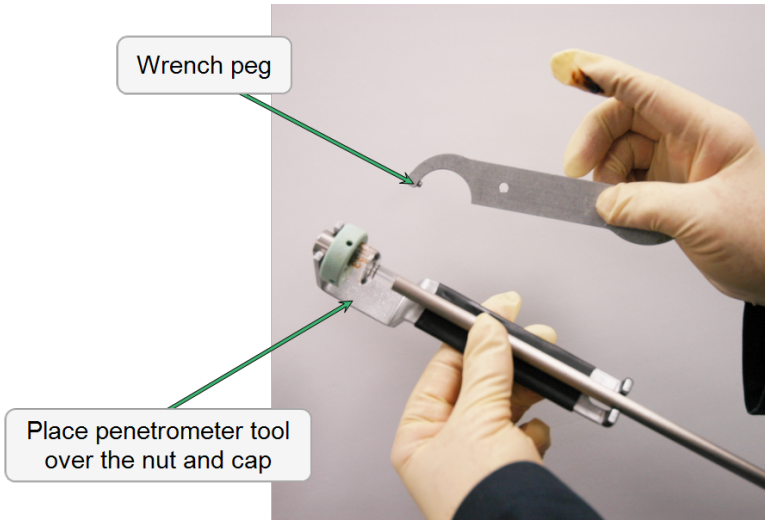
   b. Smooth the grease evenly around the lip of the bulb.

   c. Remove all excess grease from both the inside and outside of the bulb.

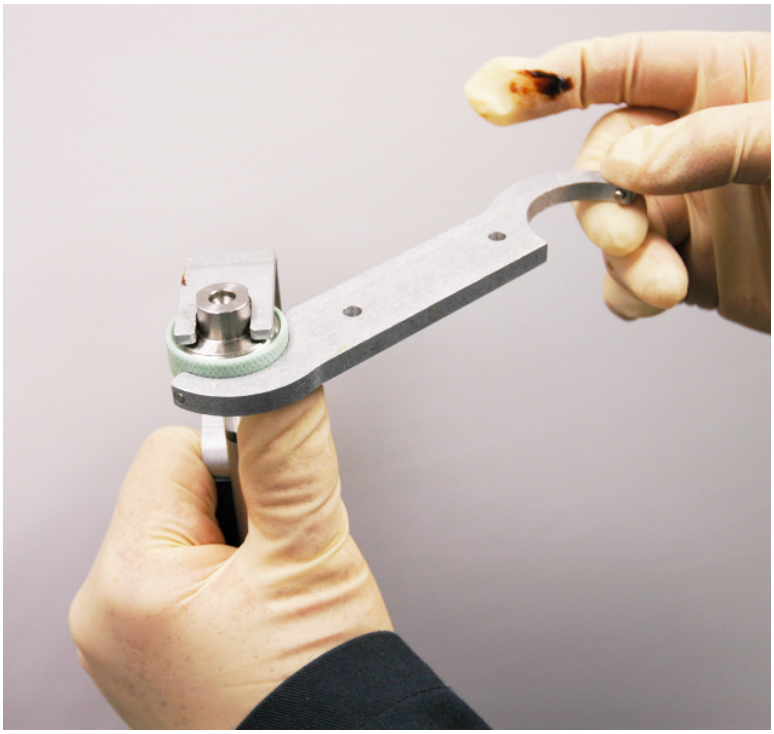
2. Hold the penetrometer upright. Place a cap over the greased bulb and turn the cap one half turn to seat.



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.

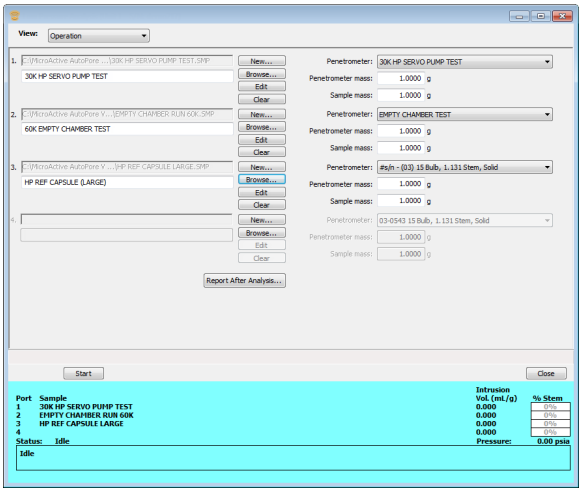
LOW PRESSURE ANALYSIS

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. For a selected port, click **Browse** and select a sample information file or click **New** to create a sample information file. If needed, change the *Penetrometer mass* and / or *Sample mass* field for the sample material being analyzed. To edit sample file parameters, click **Edit**. To create a new sample file, click *New*.
3. Click **Report after analysis** to generate reports automatically when the analysis is complete. On the *Report Settings* window, select the *Report after analysis* checkbox, then select the report destination. Select *Export after analysis* to export data as intrusion data. Select the report destination and click **OK** to return to the previous window.
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 - 13*.

- 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.

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.

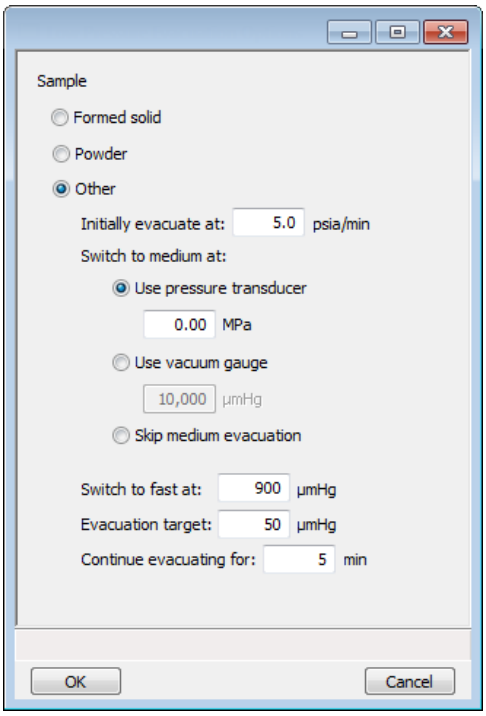
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

EVACUATE LOW PRESSURE

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.

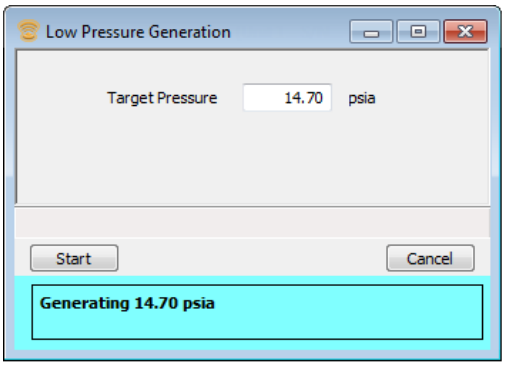


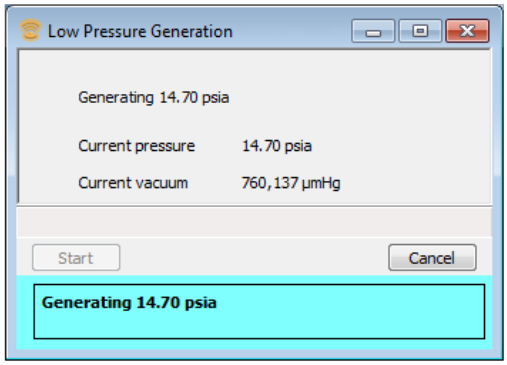
- **Initially evacuate at**. Enter the initial maximum evacuation rate.
- **Switch to medium at**. Enter the method and pressure the system must reach before medium evacuation begins.
  - Use pressure transducer
  - User vacuum gauge
  - Skip medium evacuation
- **Switch to fast at.** Enter the pressure the system must reach before fast evacuation begins.
- **Evacuation target.** Enter the evacuation pressure.
- **Continue evacuating for**. Enter the evacuation duration.

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.





1. Go to ***Unit [n] > Generate Low Pressure***.
2. Enter the target pressure.

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

3. Click **Start**. The current pressure displays.

MONITOR A LOW 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 low pressure analysis, the instrument schematic can be displayed to show the state of the low pressure system components. See *"Show Instrument Schematic" on page 2 - 22*.

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.
Status	Displays current low pressure status.
Suspend	Pauses the analysis in progress. **Resume** restarts the analysis.

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

HIGH PRESSURE ANALYSIS

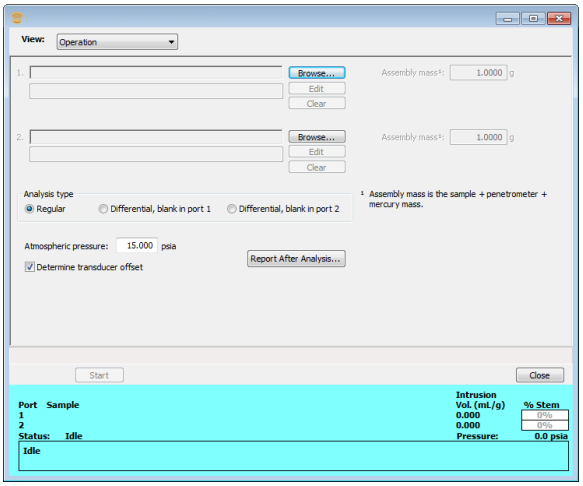
The instrument is designed to perform two high pressure analyses simultaneously. To run only one analysis, the other chambers must be closed tightly and have sufficient high pressure fluid to be drawn into the vent valve.

PERFORM A HIGH PRESSURE ANALYSIS

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



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. Click **Browse** to select the sample file with a status of *Low Pressure Complete* or *High Pressure Complete*.

   To change the assembly mass (sample + penetrometer + mercury mass), enter the mass (in grams) in the *Assembly mass* field.

If a selected sample file to be analyzed on a 9605 instrument contains pressures exceeding 33,000 psia, the following message displays:

Unit [*n*] Warning these analysis conditions contain pressures that are too large for the high pressure system of this instrument. Do you want to proceed without the high pressures?

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

2. In the *Analysis Type* group box, select the type of analysis to run.
3. To edit analysis conditions, click **Edit** for the applicable sample file to display the *Analysis Conditions* window.
4. 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 - 17*.

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	• **Regular.** Performs a differential analysis with the blank penetrometer in port 1. • **Differential, blank in port 1.** Performs a differential analysis with the blank penetrometer in port 1. • **Differential, blank in port 2.** Performs a differential analysis with the blank penetrometer in port 2. See *"High Pressure Differential Analysis" on page 5 - 17*
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***

High Pressure Analysis Fields and Buttons Table (continued)

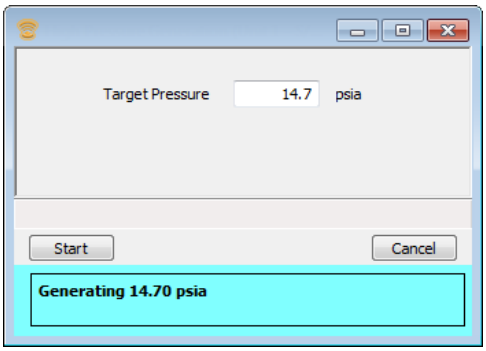
Field or Button	Description
	Pressure.

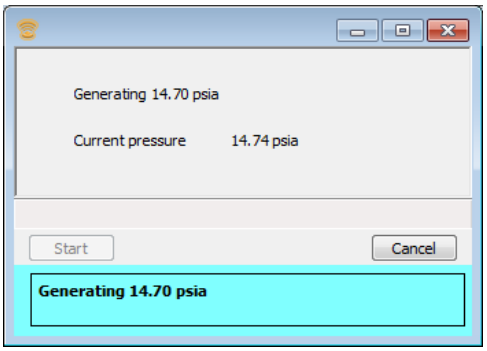
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

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.





1. Go to ***Unit [n] > Generate High Pressure***.
2. Enter the target pressure.
3. Click **Start**. The current pressure displays.

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 instrument 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.
Status	Displays the high pressure's current status.

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

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 *Formula* for the correction method. See *"Create Sample Files in Advanced Presentation Option" on page 3 - 1*.
2. Run the low pressure analysis on both files. See *"Perform a Low Pressure Analysis" on page 5 - 9*.

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 - 14*.

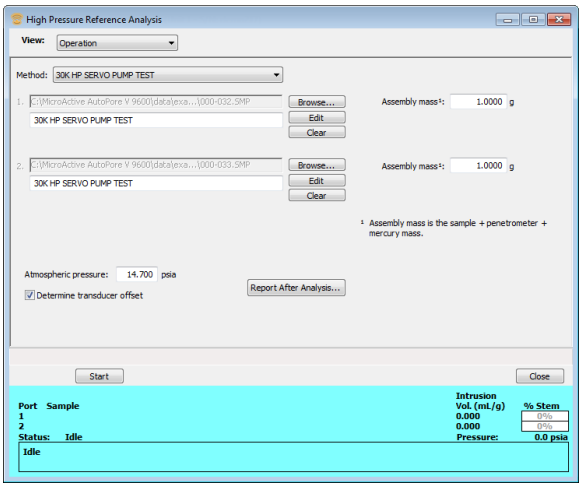
REFERENCE ANALYSIS

HIGH PRESSURE REFERENCE ANALYSIS

Unit [n] > High Pressure Reference Analysis

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

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



High Pressure Reference Analysis Fields and Buttons Table

Field or Button	Description
Assembly mass	This information is pulled from the selected sample file. Sample + penetrometer + mercury mass.

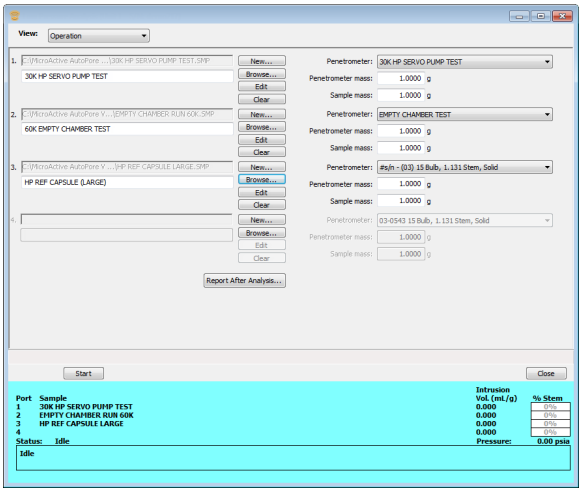
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

LOW PRESSURE REFERENCE ANALYSIS

Unit [n] > Low Pressure Reference Analysis

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

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



Low Pressure Reference 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 the *Common Fields and Buttons* **section of this operator manual.**

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 Sheets 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.

6 About Reports

Open and Close Reports

Reports > Open Report... > [.REP File]

Opens saved reports.

Reports > Close Reports

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

Start Reports

Reports > Start Report

1. Select one or more .SMP files with a *Complete* status from the library. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. Click **Report**.
2. Select the report destination in the *Report Settings* window, then click **OK**.

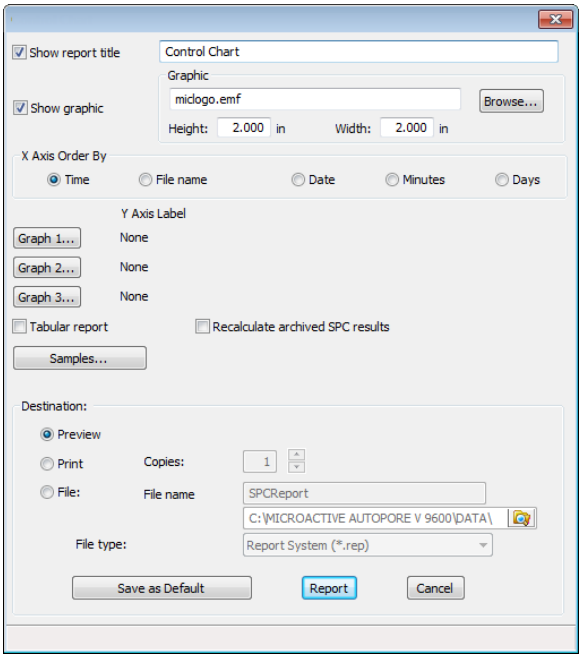
If only one report file was selected in Step 1, the *Selected Reports* window displays allowing the option to select additional reports. Select additional reports as needed, then click **OK**. If multiple files were selected, the reports are displayed in a tiled format.

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

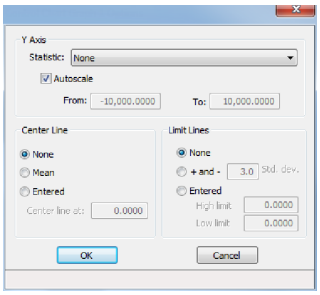
CONTROL CHART REPORT

Reports > Control Chart

Use to generate 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*]	Click to define the y-axis of each graph.  • **Statistic.** Displays the SPC variables selected on the ***Reports > SPC Report Options*** window. The selected variable will be plotted against time. This selection also becomes the y-axis label. • **Autoscale.** Allows the y-axis to be scaled automatically. To specify a range, deselect this option and enter a range in the *From* and *To* fields. • **Center Line.** Displays placement options for the center line in the graph. Choose *Entered* to specify placement of the line. • **Limit Lines group box.** Displays limiting lines options. Lines can be placed at some multiple of the standard deviation or at specified positions (*Entered*). When *Entered* is selected, enter the *High limit* and *Low limit* fields with appropriate values.
Recalculate archived SPC results	Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report.  If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application. When this option is selected, the following message displays: **Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC data speeds up future SPC reports.** **Do not show me this message again.**

Control Chart Fields and Buttons Table (continued)

Field or Button	Description
	If *Do not show me this message again* is selected, the message cannot be redisplayed without Micromeritics assistance. The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Report	Generates the report.
Samples	To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Available Files.** Contains files located in the directory specified in the *Look In* text box. • **Selected Files.** Files added from the *Available Files* list box. • **Add / Remove.** Select a file in the *Available Files* list box, then click **Add** to move the file to the *Selected Files* list box. Or select a file in the *Selected Files* list box, then click **Remove** to move the file back to the *Available Files* list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.

Control Chart Fields and Buttons Table (continued)

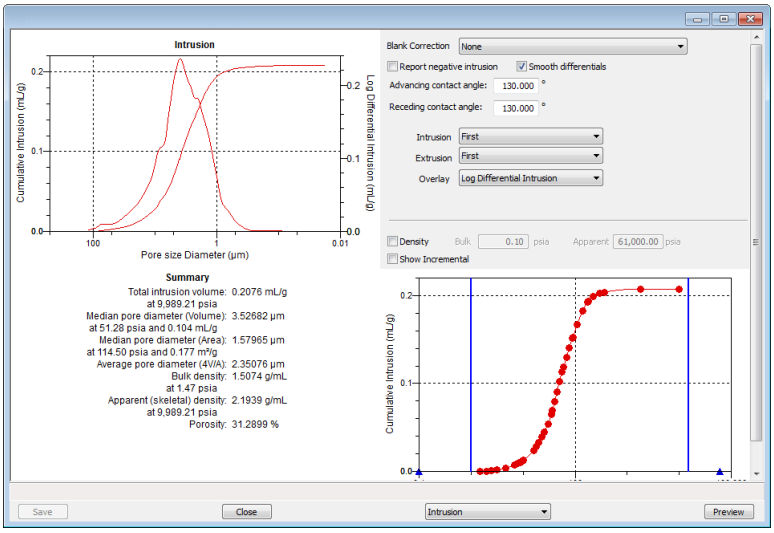
Field or Button	Description
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X Axis Order by	Select the order in which x-axis statistics are placed. Sort by: • **Time.** Time the files were analyzed. • **File name.** Alphanumeric order. • **Date.** Date the files were analyzed. • **Minutes.** Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. • **Days.** Number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.

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

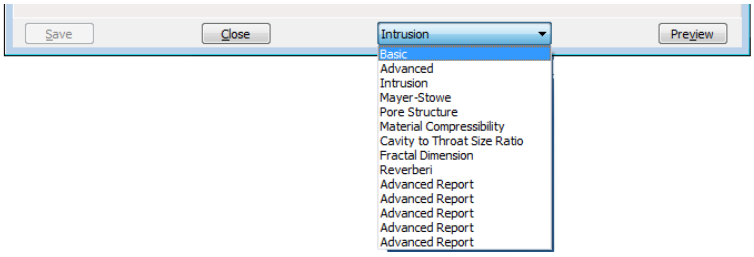
INTERACTIVE REPORTS

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

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



2. Modify the intrusion graph to show all or first intrusions and /or extrusions by selecting an option from the *Intrusion* or *Extrusion* drop-down lists.
3. Add an overlay to the intrusion graph by selecting an option from the *Overlay* drop-down list.
4. To view the reports selected for generation during or after the analysis, click **Preview**.
5. From the drop-down list at the bottom of the window:
   - change the presentation option 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.



6. 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.
7. After editing the report, click **Save** to save the changes in the sample information file.

MICROACTIVE REPORTS

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

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

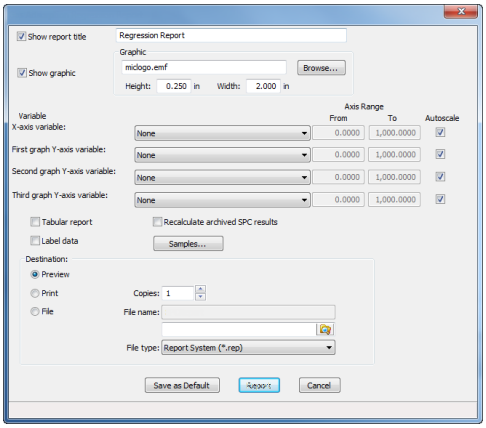
- Advanced
- Intrusion
- Mayer Stowe
- Pore Structure
- Material Compressibility
- Cavity to Throat Size Ratio
- Fractal Dimension
- Reverberi

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

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	When enabled, allows the x- and y-axes to be scaled automatically.
Axis Range	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if *Autoscale* is selected.
Label data	Use to label the points on the plot to correspond with the values in the sample files.
Recalculate archived SPC results	Use to have archived SPC values recalculated ensuring any changes made to the SPC Report Options are included in the new report. This option lengthens the time required to generate the report. If this recalculation option is enabled and sample files from an earlier application version are selected, it is recommended that copies of the archived sample files be used rather than the original. Selecting this option will make some archived sample files unreadable by the original application. When this option is selected, the following message displays: **Saving the recalculated SPC data may render some files unreadable by the original application. Saving the SPC**

Regression Report Fields and Buttons Table (continued)

Field or Button	Description
	data speeds up future SPC reports. **Do not show me this message again.** If *Do not show me this message again* is selected, the message cannot be redisplayed without Micromeritics assistance. The first time this option is used, the time it takes to generate the report is lengthened. The second time the report is generated, if using the same sample files used in the initial calculation, it is recommended that this option not be selected since the data was recalculated previously. If a sample file is added or removed from the report after the initial recalculation, this option should be selected again to ensure the data from the newly added or removed sample file is recalculated.
Samples	To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Available Files.** Contains files located in the directory specified in the *Look In* text box. • **Selected Files.** Files added from the *Available Files* list box. • **Add / Remove.** Select a file in the *Available Files* list box, then click **Add** to move the file to the *Selected Files* list box. Or select a file in the *Selected Files* list box, then click **Remove** to move the file back to the *Available Files* list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.

Regression Report Fields and Buttons Table (continued)

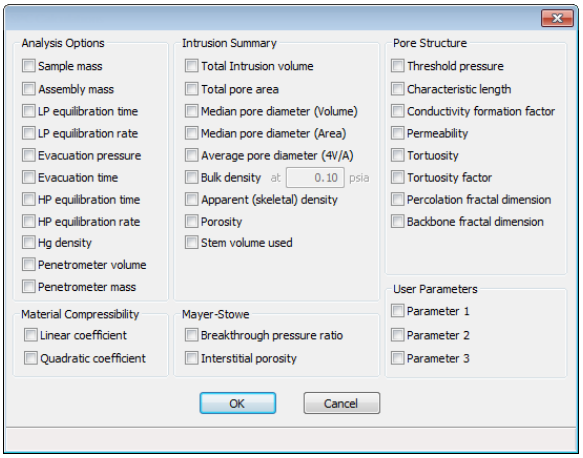
Field or Button	Description
Tabular report	Generates a tabular report of the included samples. A tabular report contains the numeric values contributed by each sample.
X- and Y-Axis Variable	Use to designate the x- and y-axes variables. The variables in the drop-down lists are those selected in the ***Reports > SPC Report Options*** window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.

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

SPC REPORT

Reports > SPC Report Options

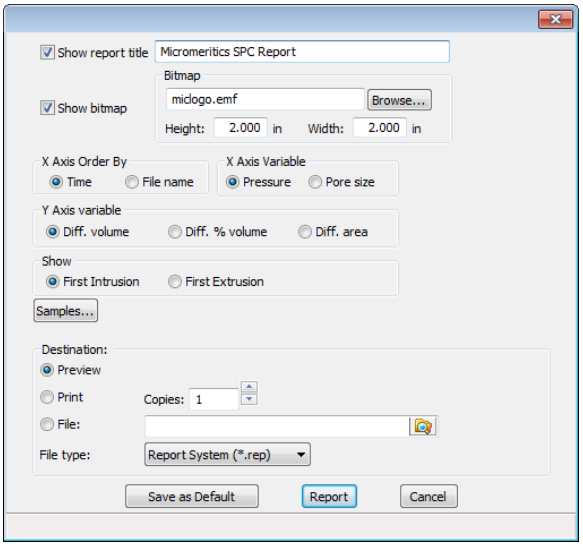
Use to generate reports with various *SPC* (Statistical Process Control) options. All selected variables must be computed for each sample file used in an SPC report; therefore, it is more efficient to select only the necessary variables.



PSD HISTORY REPORT

Reports > PSD History

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



1. From the main menu, select ***Reports > PSD History***.
2. On the *PSD History Options* window, select the following options as needed:

PSD History Report Fields and Buttons Table

Field or Button	Description
Report	Generates the report.
Samples	To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Available Files.** Contains files located in the directory specified in the *Look In* text box. • **Selected Files.** Files added from the *Available Files* list box. • **Add / Remove.** Select a file in the *Available Files* list box, then click **Add** to move the file to the *Selected Files* list box. Or select a file in the *Selected Files* list box, then click **Remove** to move the file back to the *Available Files* list box. Or double click the file name to move the file from one list box to the other.
Save as Default	Click to save selected report options as default report settings.
Show	Select one of the following options: • **First Intrusion.** Select to show the first intrusion. • **First Extrusion.** Select to show the first extrusion.
Show graphic	Use to show a graphic on the report header. Click **Browse** to locate the graphic. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Show report title	Select and enter a report title to appear on the report header.
X-axis Order By	Select one of the following: • **Time.** Time the files were analyzed. • **File name.** Alphanumeric order.

PSD History Report Fields and Buttons Table (continued)

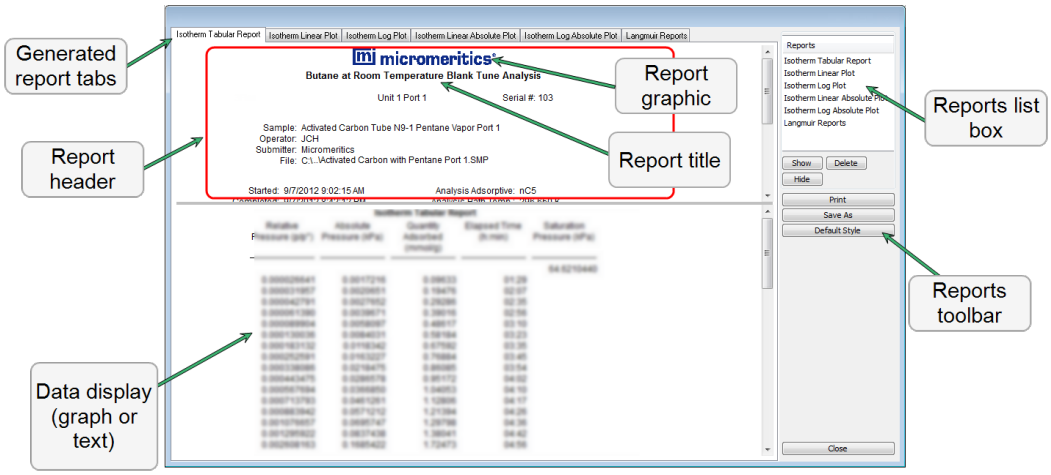
Field or Button	Description
X Axis variable	Select one of the following to display on the graph: • **Pressure.** • **Pore size.**
Y Axis variable	Select one of the following to display on the graph: • **Diff. volume.** • **Diff. % volume.** • **Diff. area.**

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

Report Features and Shortcuts

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

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



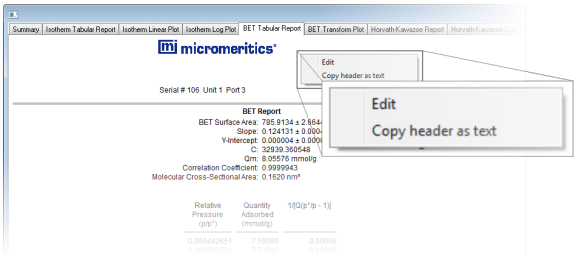
If configured, the report header can also contain a graphic and / or a title.

- Tabular and graphical reports contain sample and analyzer statistics such as analysis date / time, analysis conditions, etc.

- The headers contain notes of sample file changes occurring after analysis.
- Summary report headers contain the same information as tabular and graphical reports with the exception of notes.

REPORT HEADER SHORTCUTS

Display header shortcuts by right clicking in the report header.

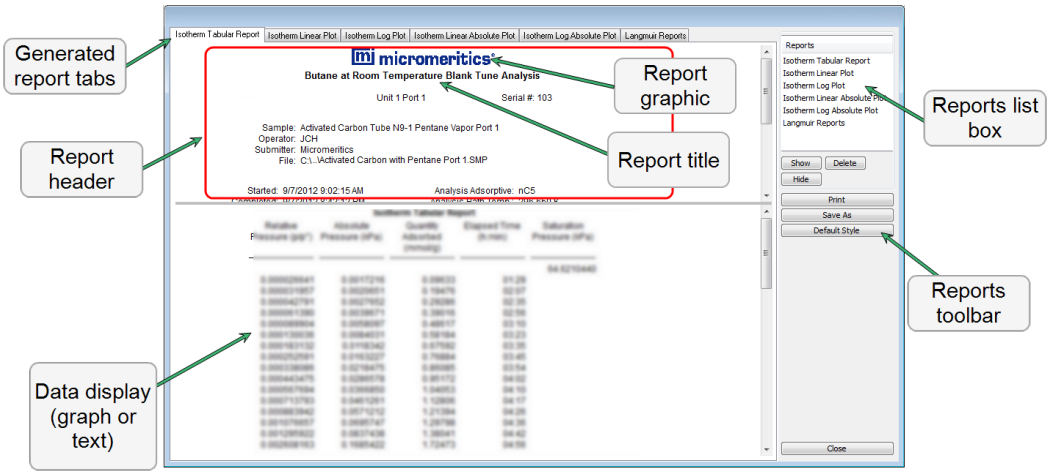


Report header Shortcut Field and Button Table

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

REPORT TOOLBAR

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



Report Toolbar Fields and Buttons Table

Field or Button	Description
Default Style	Click to specify default report parameters for fonts and curve properties. • **Font Type.** Use to edit the font type and attributes for the selected item. Select an item in the list, click **Edit**, and select from various font options. • **Thickness**. Enter a thickness number for the curve. • **Histogram Fill Style.** Select a histogram fill option. • **Graph border line thickness.** Enter a thickness number for the graph border.
Delete	Deletes the selected report in the *Reports* list box. Deleted reports will have to be regenerated if deleted in error.
Hide	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the *Reports* list box.
Print	Displays the *Print* window for report output. • **Name** drop-down list and **Properties**. Select the printer from the drop-down list and click **Properties** to change printer setup, etc. • **Copies.** Select the number of copies and collate option. • **Current.** Selects the active report (or selected tab).

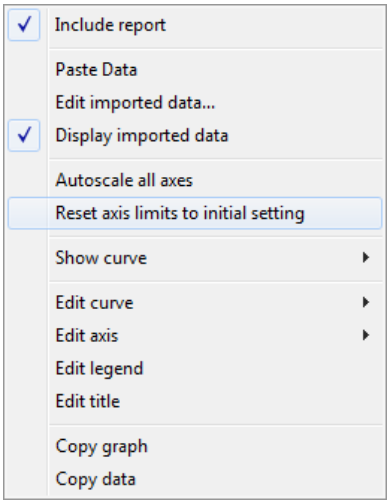
Report Toolbar Fields and Buttons Table (continued)

Field or Button	Description
	• **All.** Selects all reports in the *Reports* list box. • **Shown.** Selects only the reports not hidden. • **Clear.** Clears all selections.
Reports list box	Contains a list of all generated reports. The same reports display as tabs at the top of the report header unless the report has been hidden using the **Hide** button.
Show	Displays the selected or hidden report in the *Reports* list box.

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

GRAPH FEATURES AND SHORTCUTS

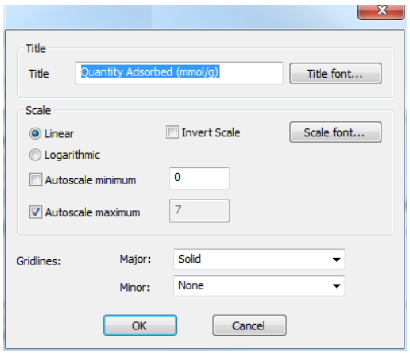
Display graph report shortcuts by right clicking in the body of the graph report.



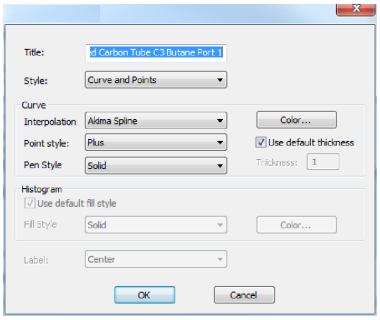
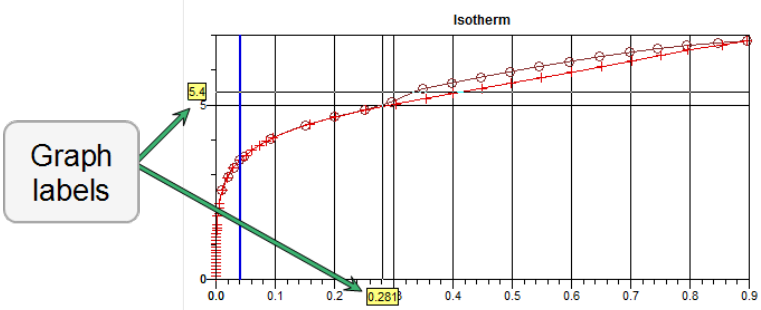
Graph Shortcuts Options and Description Table

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

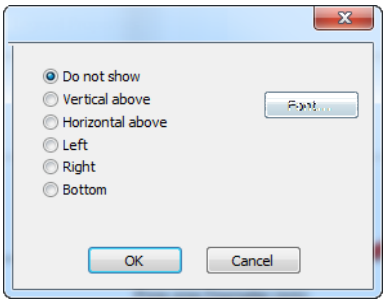
Graph Shortcuts Options and Description Table (continued)

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

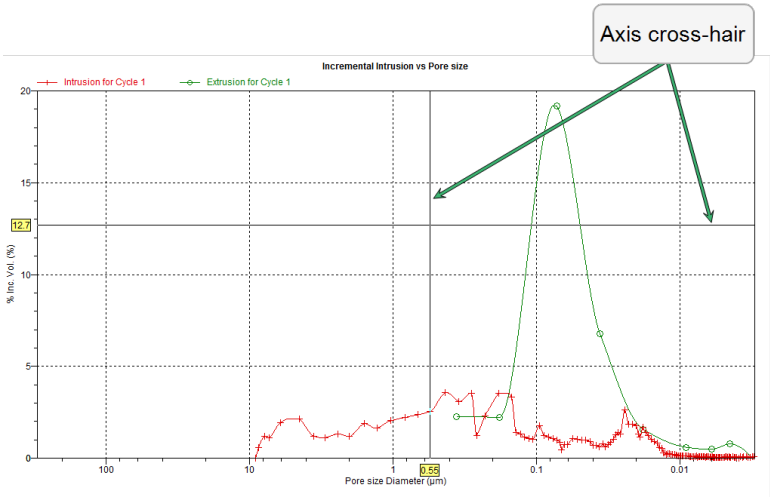
Graph Shortcuts Options and Description Table (continued)

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

Graph Shortcuts Options and Description Table (continued)

Field or Button	Description
	Select new font attributes for the report data. To return to the default fonts, enable *Use default font*. 
Edit title	Use to change the graph title and font. Click **Font** to font attributes. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.
Include report	When selected, places a checkmark to the left of the report in the *Select Reports* list box on the *Report Options* tab.
Paste Data	Used with pore distribution data reports only. Use to paste ASCII text data from the clipboard onto the the active graph.
Reset axis limits to initial setting	Removes the cross-hair and returns the graph back to the initial setting.
Show curve	Displays a list of all curves. Select the curve(s) to display.

Axis Cross-hair



The cross-hair feature displays axis coordinates.

1. Left click on the graph to view the cross-hair coordinates.
2. To remove the cross-hair, right click in the graph area and select either *Autoscale all axes* or *Reset axis limits to initial setting*.

Graph Grid Lines

Options > Graph Grid Lines

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

Graph Grid Lines Fields and Buttons Table

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

Zoom Feature

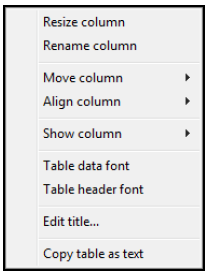
Use the zoom feature to examine graph details.

1. Open the graph.
2. Hold down the left mouse button, then drag the mouse pointer across the graphical area to be enlarged. A box will display in the area to be enlarged.

3. Release the mouse button. The enlarged area fills the graph area. To return to normal view, right click in the graph area, then select either *Autoscale all axes* or *Reset axis limits to initial setting* on the shortcut menu.

TABULAR REPORT FEATURES AND SHORTCUTS

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



Tabular Reports Shortcut Options and Descriptions Table

Field or Button	Description
Align column	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. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.
Move column	Right click the column to be moved. Select *Move column* on the shortcut menu and select *Left* or *Right* for the move.
Rename column	Right click the column to be renamed. Select *Rename column* on the short-cut menu and enter the new column name.
Resize column	Right click the column to be resized. Select *Resize column* on the shortcut menu and enter the new column width in inches.
Show column	Displays a list of all columns. Click a column to add a checkmark to show the column or remove the checkmark to hide the column.
Table data font	Right click in the report data. Select *Table data font* on the shortcut menu. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.
Table header font	Right click in the report data. Select *Table header font* on the shortcut menu. Deselect the *Use default font* to enable font options. Select new font attributes for the report data. To return to the default fonts, enable *Use default font*.

GRAPH OVERLAYS

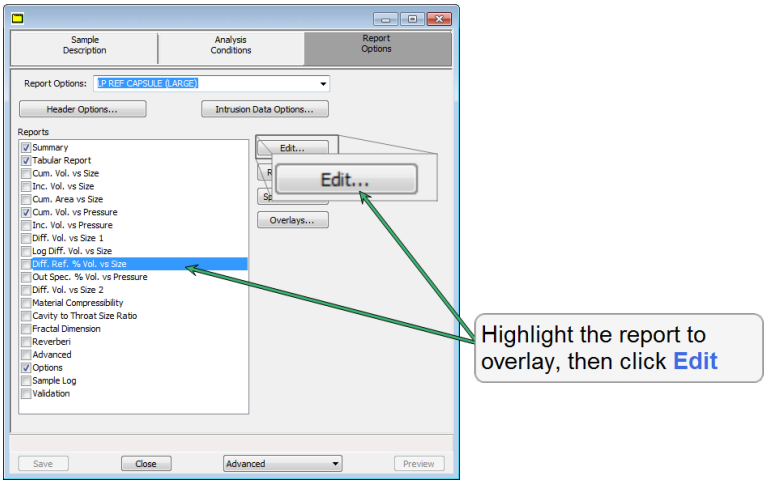
Use the graph overlay functions to compare multiple graph options. Graphical lines are differentiated by the use of varying colored symbols outlined on a legend. Overlays may be generated in two ways:

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

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

OVERLAY MULTIPLE GRAPH OPTIONS

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

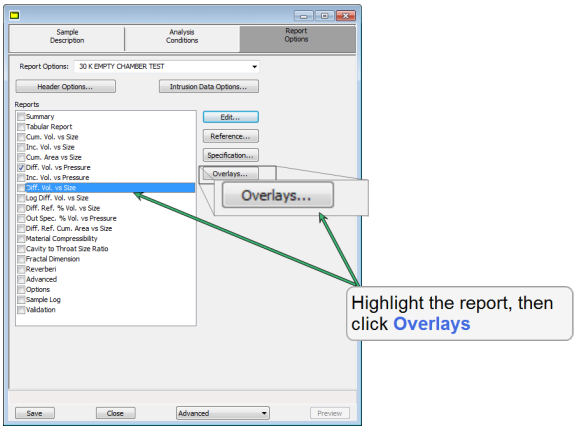


6. On the *Plot Options* window, select *Options* to include in the overlay. If the x- and / or y-axes are to be autoscaled, select *Autoscale*; otherwise, enter the *From* and *To* points for the axes.
7. In the *Y-Axis* group box, select *Variable* and/or *Overlay* options.
8. Click **OK** to return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

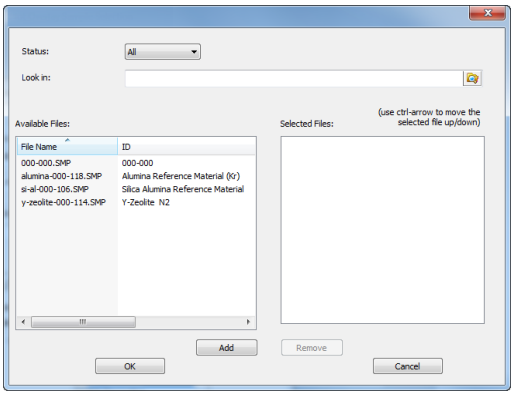
OVERLAY MULTIPLE SAMPLE FILES

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



- Double click a file name in the *Available Files* box to move the file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available Files* box, double click the file name in the *Selected Files* box, or
- Select a file name in the *Available Files* box. Click **Add** to move the selected file to the *Selected Files* box. To move a file from the *Selected Files* box back to the *Available*

Files box, select a file name in the *Selected Files* box, then click **Remove**. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files.

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

REPORT EXAMPLES

REVERBERI REPORT PLOT

Micromeritics Instrument Corporation

oActive AutoPore V 9600

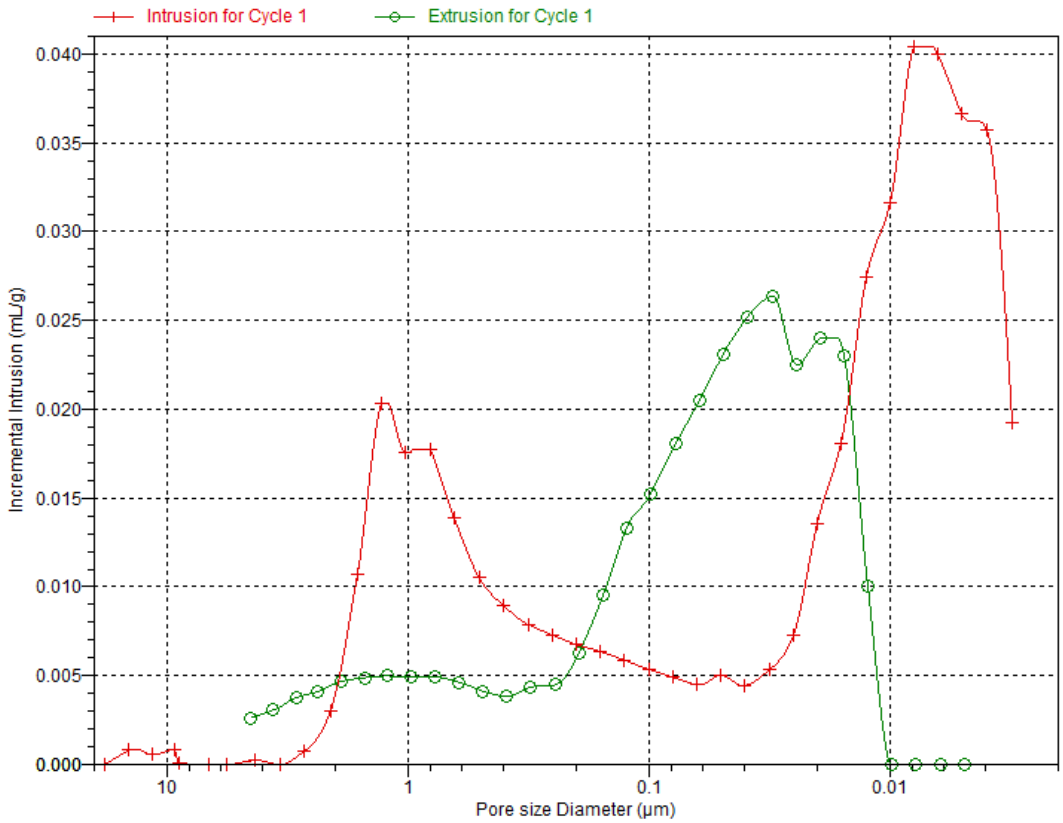
MicroActive AutoPore V 9600
Serial # 102 Port 3/1

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

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

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

Incremental Intrusion vs Pore size



SILICA ALUMINA REFERENCE MATERIAL REPORT

Micromeritics Instrument Corporation

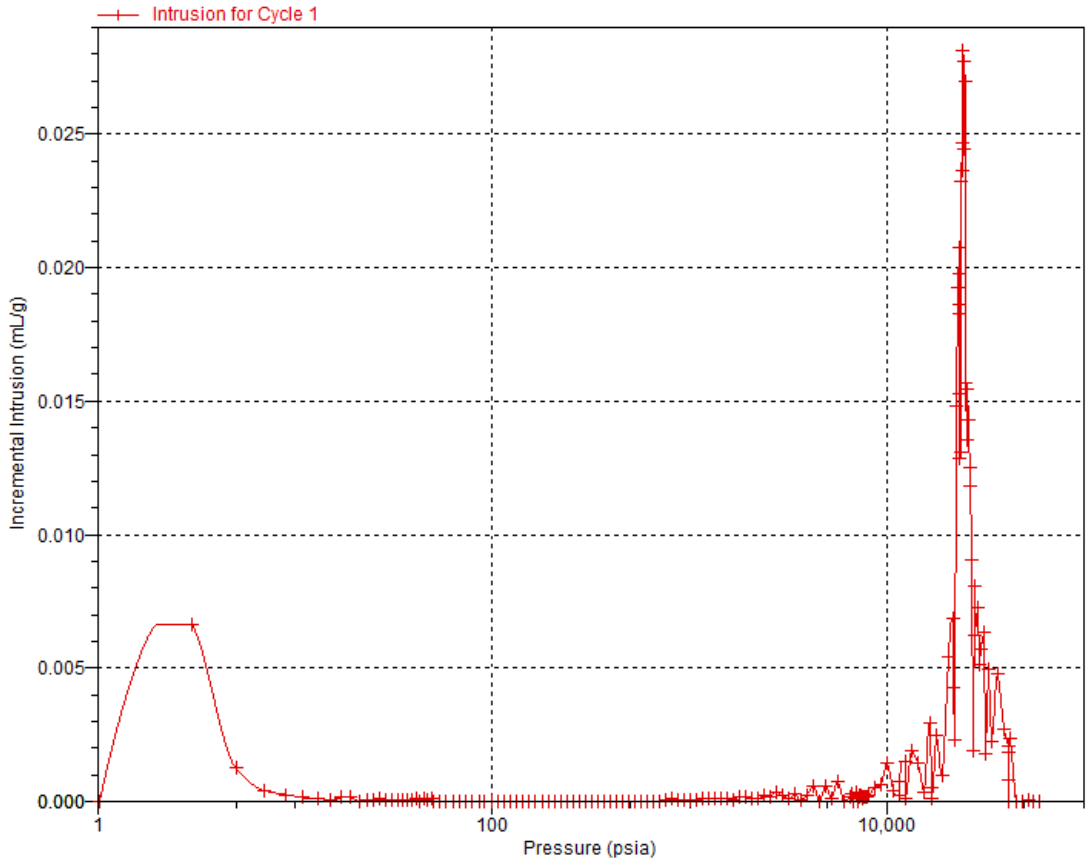
MicroActive AutoPore V 9600

MicroActive AutoPore V 9600
Serial # 102 Port 1/1

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

LP Analysis Time:	4/8/2014 8:52:51 PM	Sample Mass:	1.0652 g
HP Analysis Time:	4/9/2014 8:33:55 AM	Stem Volume Used:	51 %
Report Time:	4/17/2014 7:24:39 AM	Show Neg. Int:	No
Report Range:	0.10 to 61,000.00 psia	Correction Type:	None
Adv. Contact Angle:	130.000 °	Mercury Temperature:	21.64 °C
Rec. Contact Angle:	130.000 °		

Incremental Intrusion vs Pressure



GARNET TABULAR REPORT

Micromeritics Instrument Corporation

MicroActive AutoPore V 9600

MicroActive AutoPore V 9600
Serial # 102 Port 1/1

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

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

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

Tabular Report

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

CONTROLLED PORE GLASS PLOT

CONTROLLED PORE GLASS

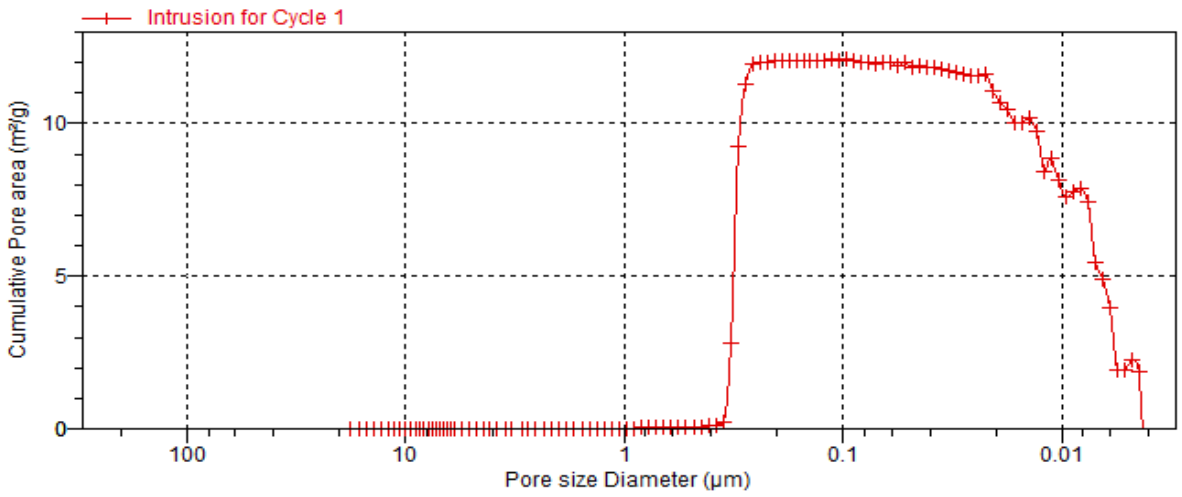
MicroActive AutoPore V 9600

MicroActive AutoPore V 9600
Serial # 102 Port 1/1

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

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

Cumulative Pore Area vs Pore size



7 Report Options

File > Open > [.RPO File]

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

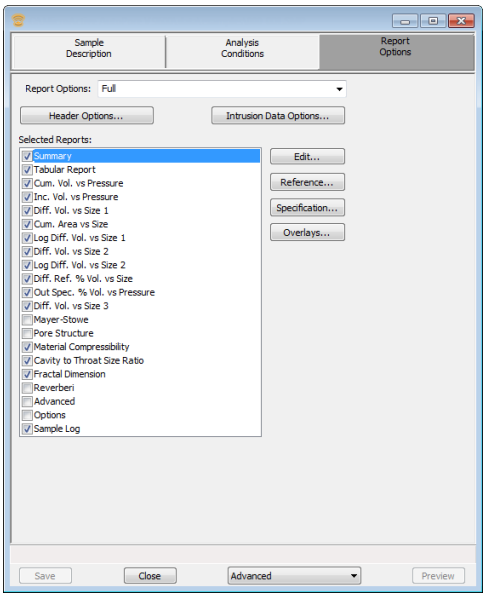
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

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

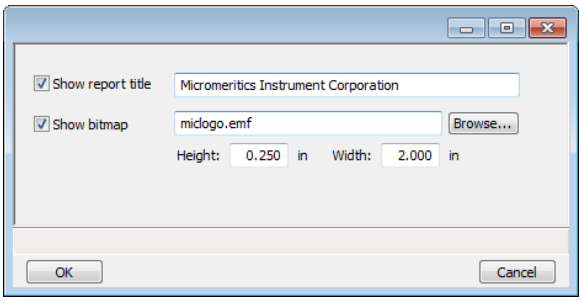
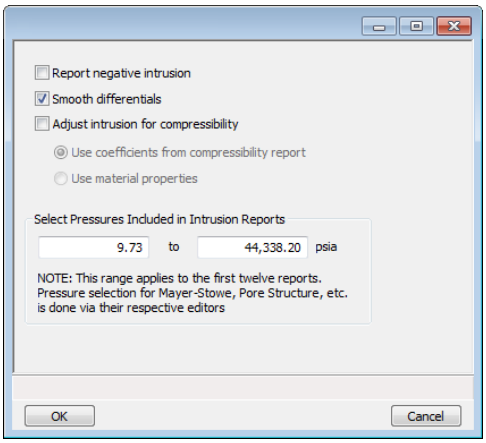
Reports can be generated for data:

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

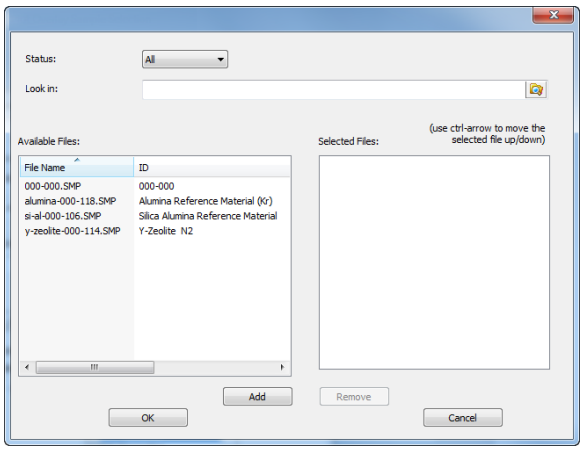
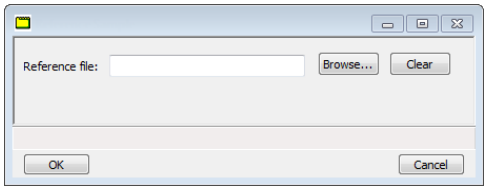
Customized report options files can be created then loaded into a sample file, allowing quick generation of reports.



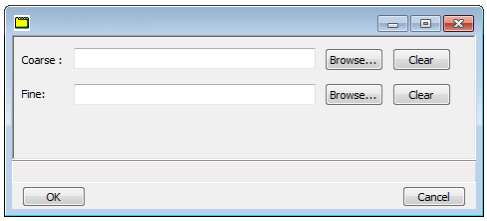
Report Options Fields and Buttons Table

Field or Button	Description
Edit	Edit graph options. • **Plot Points.** Select to plot points on the graph.. • **Plot Curve.** Select to plot curves on the graph.. • **Show Histogram.** Select to show the graph as a histogram. When selected, the *Plot Points* and *Plot Curve* selections are disabled.
Header Options	 **Show report title**. Enter a report title to appear on the report header. **Show bitmap.** Displays the selected graphic on the report header. Click **Browse** to locate the graphic in either .BMP or .EMF format. • **Height / Width.** Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.
Intrusion Data Options	 **Adjust intrusion for compressibility:** • **Use coefficients from compressibility report.** Select to have the application use the coefficients from the *Material Compressibility* report rather than from the *Material Properties*.

Report Options Fields and Buttons Table (continued)

Field or Button	Description
	• **Use materials properties.** Select to have the application use the parameters from *Material Properties* rather than from the *Material Compressibility* report. **Report negative intrusion.** Select to report small incorrect polarities (negative intrusions or positive extrusions) which may indicate the presence of noise, improper blank correction, or instrument malfunction. **Select Pressures Included in Intrusion Reports.** Enter range of pressure. This range applies to the first twelve reports on the options list. **Smooth differentials.** Select to apply smoothing to any differentials reported in tables or graphs.
Overlays	Select the files to overlay. 
Reference	Selects a sample file to compare analysis results with the current sample. 

Report Options Fields and Buttons Table (continued)

Field or Button	Description
Reports list box	Select the report names to include in the report.
Specification	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. 

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

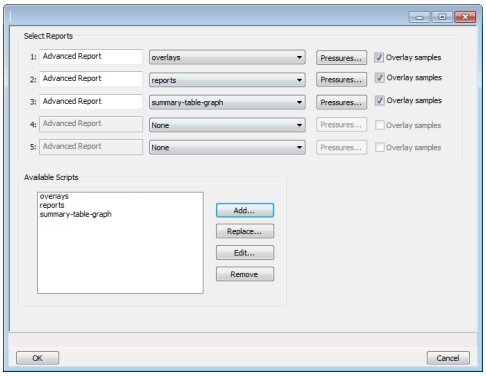
ADVANCED REPORT OPTIONS

See *"Python Module - Advanced Reports" on page H - 1*.

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**.

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

1. Create the Python script and save it in the *Scripts* directory.
2. Open a Sample File with a *Completed* status.
3. Select *Advanced* at the bottom of the window to return to the tabbed view.
4. On the *Report Options* tab, select *Advanced* in the *Selected Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add** in the *Available Scripts* group box to locate and select the Python script. Repeat for each script to be added.



6. In the *Selected Reports* group box, click the drop-down arrows to select up to five Python scripts previously added in the *Available Scripts* box.
7. Click **Pressures** to add pressure points to the report.
8. Click **OK** to return to the *Report Options* tab.
9. On the *Report Options* tab window, click **Preview**. The Python Reports will be included on the tabs across the top portion of the *Reports* window.
10. Select the *Overlay samples* checkbox to enable the overlay sample feature.

Advanced Report Options Fields and Buttons Table

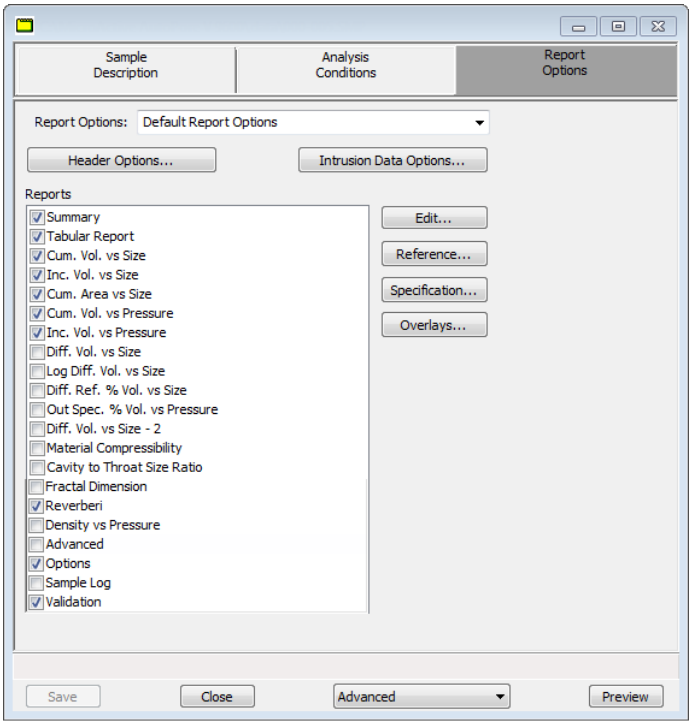
Field or Button	Description
Add	Click to add additional Python reports.
Available Scripts	Lists the available reports and provides the option to add, replace, edit or remove reports.
Overlay samples	Use to overlay samples as defined by the function.
Advanced Report 1 through 5	Use the drop-down lists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

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

GRAPH REPORT OPTIONS

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**.

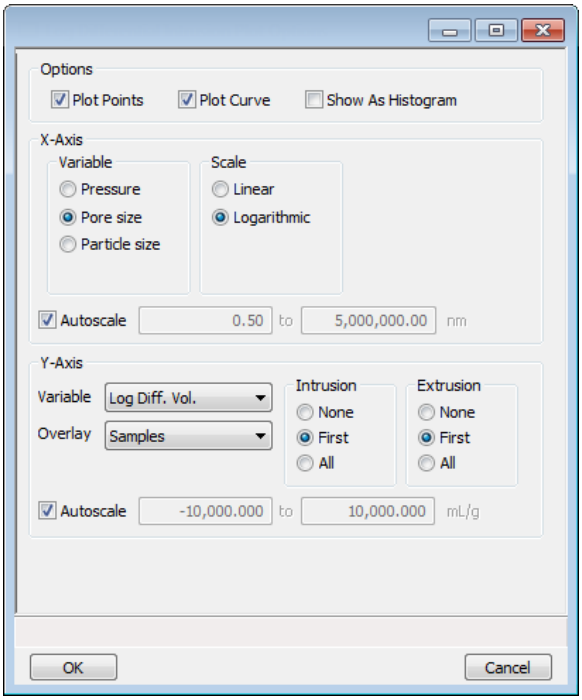
- Cumulative Area vs Size Plot Options
- Cumulative Volume vs Pressure Plot Options
- Cumulative Volume vs Size Plot Options
- Difference from Reference % Volume Plot Options
- Differential Intrusion Plot Options
- Differential Volume vs Size Plot Options
- Incremental Volume vs Pressure Plot Options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



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

EDIT GRAPH REPORT OPTIONS

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



Graph Report Options Fields and Buttons Table

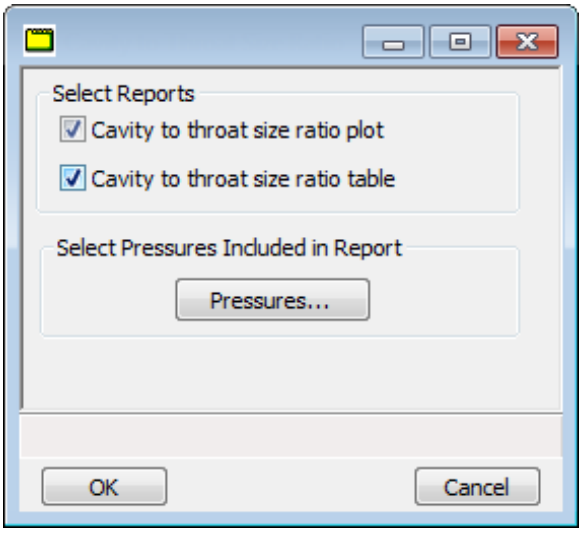
Field or Button	Description
Autoscale	When enabled on the report parameters windows, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y-ranges will be set so that all the data is shown. If *Autoscale* is not selected, the entered range is used.
Options	Select one or more graph display options. **Plot Points.** Select to plot the points on the graph. **Plot Curve.** Interpolated from data points. **Show As Histogram.** When selected, *Plot Points* and *Plot Curve* options are disabled.
Overlays button	Click to select sample files that contain data to be overlayed onto the selected plot.
Reference button	Click to select a sample file to compare analysis results of the current sample.

Graph Report Options Fields and Buttons Table (continued)

Field or Button	Description
Specification button	Click to specify sample files to use for the boundaries of the coarse and fine specifications.
X-Axis group box	Select options for the x-axis. **Variable.** **Scale.**
Y-Axis	Select options for the y-axis. **Variable**. Select the y-axis variable from the drop-down list. **Overlay.** [*Optional*]. Select an option to overlay. **Intrusion / Extrusion.** Select the data points to plot. • **None.** No intrusion (or extrusion) data points • **First.** Points from the first intrusion (or extrusion) cycle • **All.** Include all intrusion (or extrusion) data points
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**	

CAVITY TO THROAT SIZE RATIO REPORT OPTIONS

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**.



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

Cavity to Throat Size Ratio Fields and Buttons Table

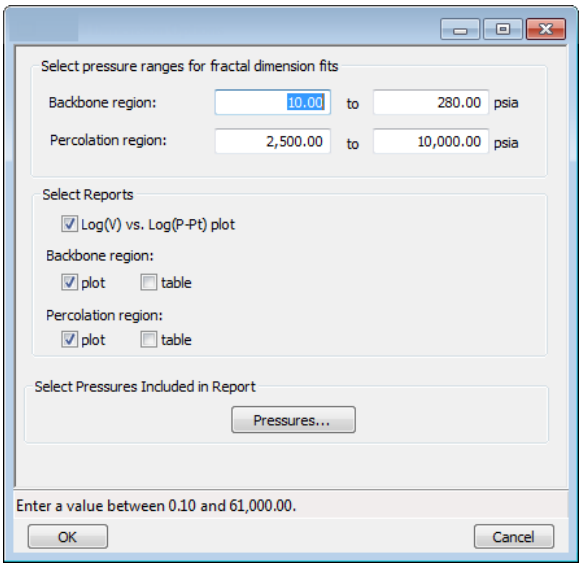
Field or Button	Description
Cavity to throat size ratio plot	Select to show the ratio plot.
Cavity to throat size ratio table	Select to show the ratio table
Pressures	Select to enter the minimum and maximum calculation pressure range.

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

FRACTAL DIMENSION REPORT OPTIONS

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

The 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

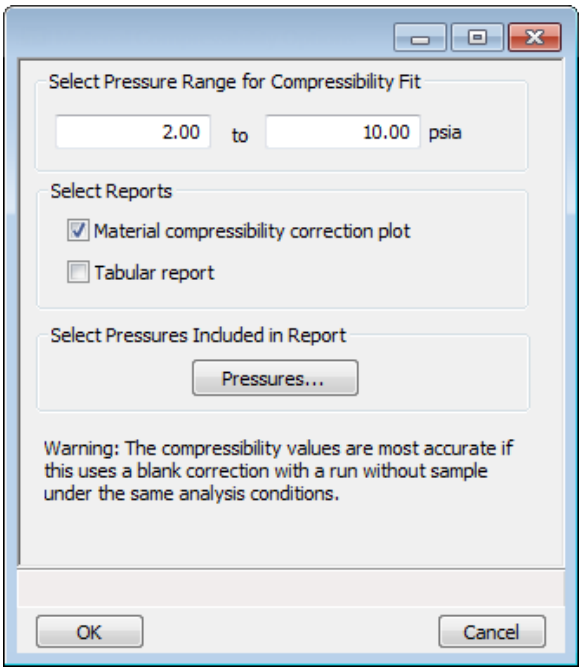
Field or Button	Description
Backbone region	**Range.** Enter the pressure at which the calculations are to be performed.
Percolation region	**Range.** Enter the pressure at which the calculations are to be performed.
Select Reports	• **Show Log(V) vs Log (P-Pt) graph.** Select to generate an additional graph to help select linear range for calculations. • **Backbone region.** • **Percolation region**.
Pressures	Select to enter minimum and maximum pressures

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

MATERIAL COMPRESSIBILITY REPORT OPTIONS

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

The 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	Enter the Calculation pressure range.
Range	**P1.** Enter the beginning pressure. **P2.** Enter the ending pressure.
Show Graph	Displays the results in graph format. The graph plots pressure on a log scale on the x-axis and the volume compressed readings as points on the y-axis, with the theoretical curve based on the calculated values overlaid.

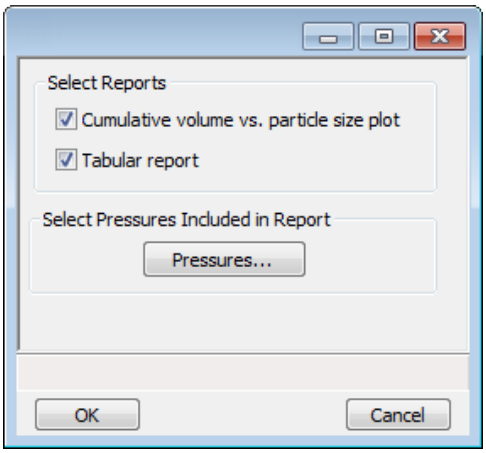
Material Compressibility Fields and Buttons Table (continued)

Field or Button	Description
Show Table	Displays the results in table format. The table displays pressure, volume compressed, predicted volume compressed, and error.

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

MAYER-STOWE REPORT OPTIONS

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**.

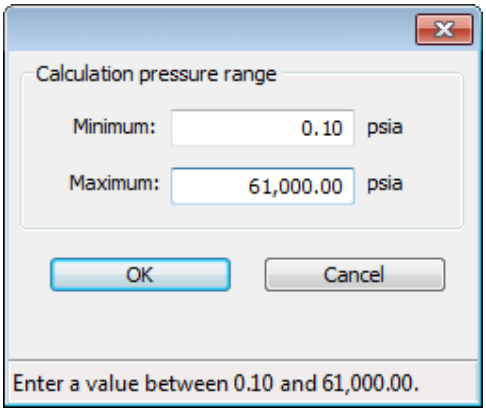


Select to show the report options for Mayer-Stowe.

Mayer-Stowe Options Fields and Buttons Table

Field or Button	Description
Cumulative volume vs particle size plot	Select to show the ratio plot.
Tabular report	Select to show the ratio table

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



Click the **Pressures** button to select.

Mayer-Stowe Pressure Fields and Buttons Table

Field or Button	Description
Pressures	Select to enter the minimum and maximum calculation pressure range.

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

Options Report

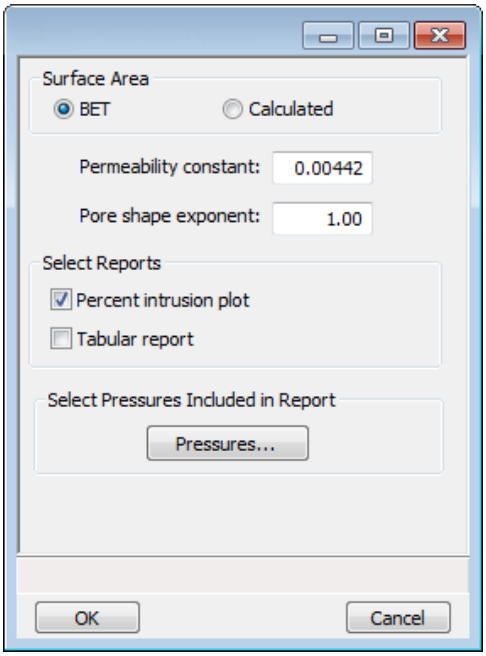
The *Options* report for mercury porosimetry analyses lists the conditions used to perform the analysis—such as:

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

Options reports cannot be edited.

PORE STRUCTURE OPTIONS

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**.



Select to show the Port Structure options.

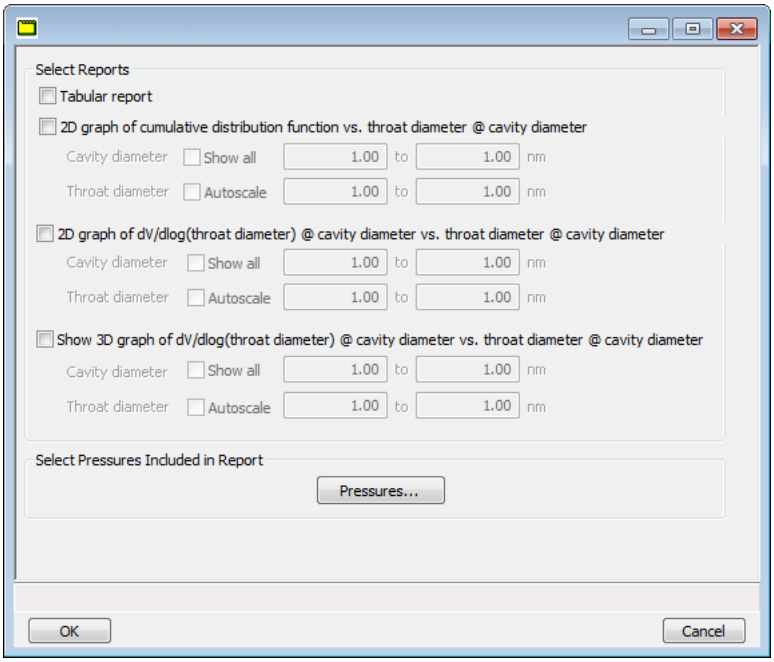
Pore Structure Options Fields and Buttons Table

Field or Button	Description
Surface Area	Selected the type of Surface Area to reference. Calculated should be the default selection.
Permeability constant	Viewed on Report Summary
Pore shape exponent	Viewed on Report Summary
Select Reports	Select the report type.
Pressures	Select to enter minimum and maximum pressures

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

REVERBERI REPORT OPTIONS

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**.



Reverberi Fields and Buttons Table

Field or Button	Description
Cavity diameter	Select *Show all* to display all diameters or enter a specific range to display.
Show 2D graph of cumulative distribution ...	Select to display a 2D graph with this description.
Show 2D graph of dV/dlog (throat diameter) ...	Select to display a 2D graph with this description.
Show 3D graph of dV/dlog (throat diameter) ...	Select to display a 3D graph with this description.

Reverberi Fields and Buttons Table (continued)

Field or Button	Description
Show table	Select to display a table in the report.
Throat diameter	Select *Autoscale* to autoscale the throat diameter or enter a specific range to display.

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

SAMPLE LOG REPORT

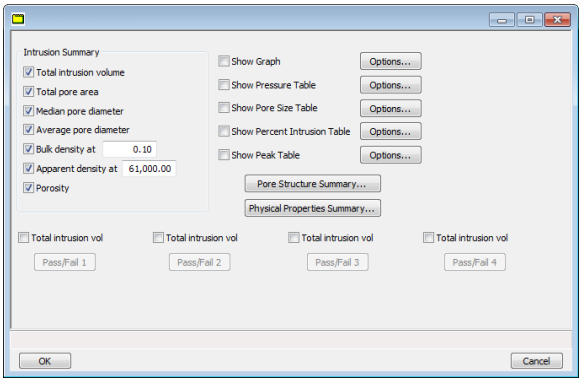
This report provides information on:

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

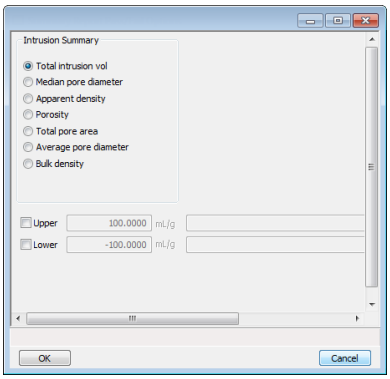
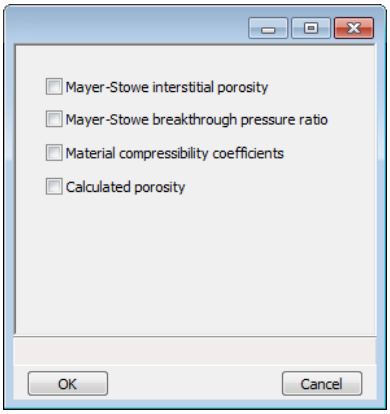
SUMMARY REPORT

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

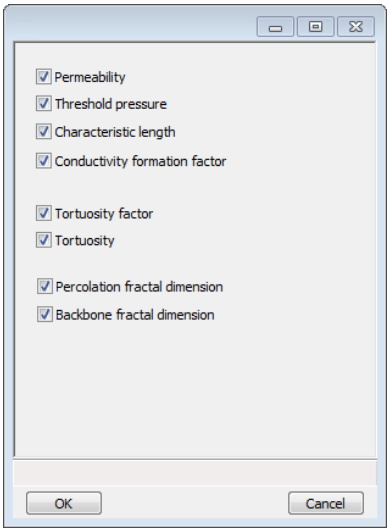
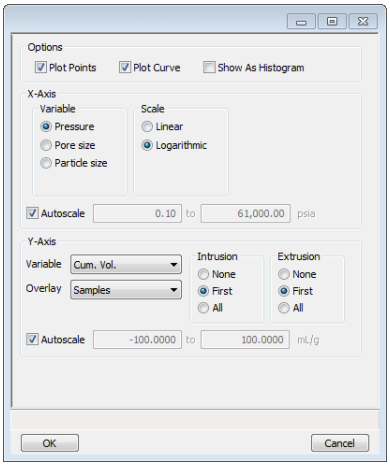
The *Summary Report* provides a condensed listing of selected data results.



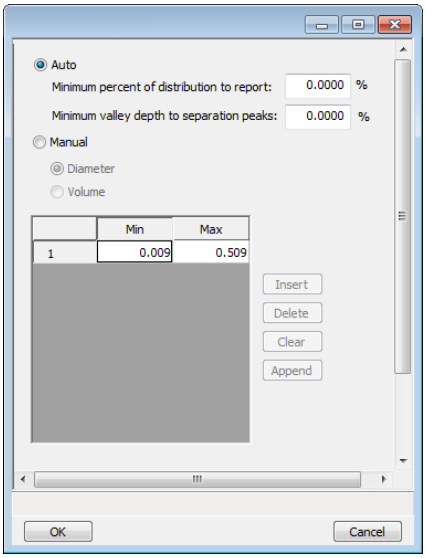
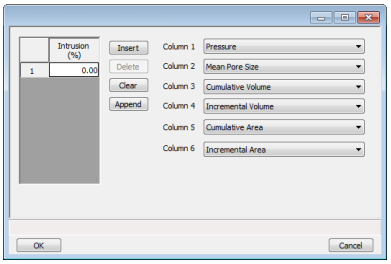
Summary Report Fields and Buttons Table

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

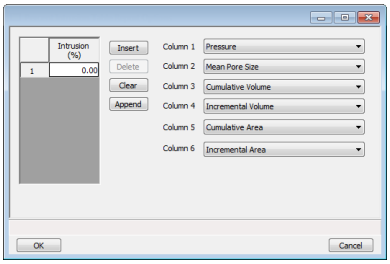
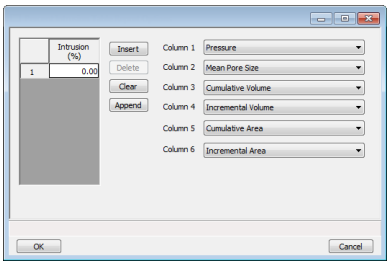
Summary Report Fields and Buttons Table (continued)

Field or Button	Description
	
Show Graph	Select to display the report in graph format. Click **Options** to the right of *Show Graph* to select how the graph should display.  See *"Report Options" on page 4 - 12* for a description of fields and buttons on this window.
Show Peak Table	Select to display the peak table in the report. Click **Options** to the right of *Show Peak Table* to select how the graph should display.

Summary Report Fields and Buttons Table (continued)

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

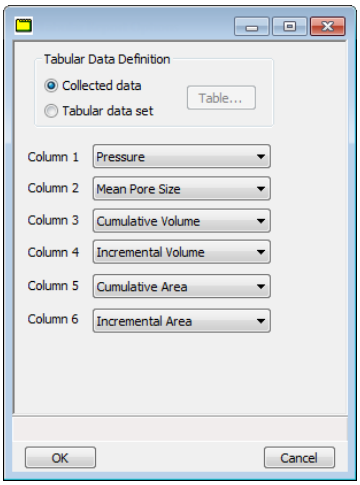
Summary Report Fields and Buttons Table (continued)

Field or Button	Description
	
Show Pressure Table	Select to display pressure points in the report. Click **Options** to the right of *Show Pressure Table* to select points to display. Use the drop-down fields to specify the data to appear in the specified columns for report generation. 
	For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

TABULAR REPORT OPTIONS

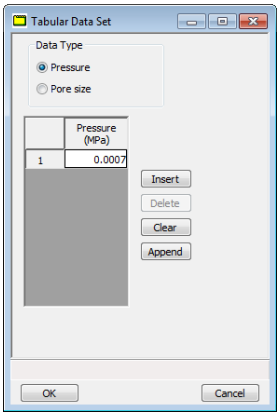
To edit reports, open the *Sample Information* file. Select the *Report Options* tab, then highlight the report name in the *Selected Reports* list box. Click **Edit**.

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



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

Tabular Report Options Fields and Buttons Table

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

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

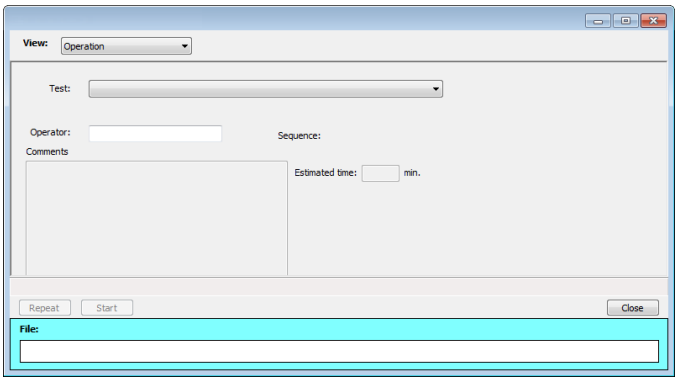
Blank Page

8 DIAGNOSTICS

START DIAGNOSTIC TEST

Unit [n] > Diagnostics > Start Diagnostic Test

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



Start Diagnostic Test Fields and Buttons Table

Field or Button	Description
Comments	Displays comments from the selected diagnostic test.
Estimated time (min.)	Approximate time for test completion.
File	Shows a status bar of steps complete once the test begins.
Next	Starts the next test.
Operator	Enter information to identify the person running the service test.
Repeat	Repeats the selected diagnostic test.
Report after test	Automatically generates reports to the selected destination when the test is complete.

Start Diagnostic Test Fields and Buttons Table (continued)

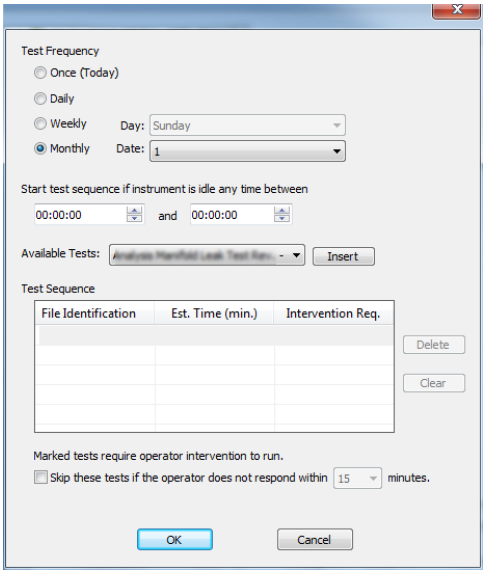
Field or Button	Description
Sequence	Sequence number assigned to the test.
Test	Select the diagnostic test to be performed. • Low Pressure Vacuum Gauge Test • Low Pressure Outgas Test • Low Pressure Transducer Test • Low Pressure Leak Test • High Pressure Transducer Test • High Pressure Speed / Cycle Test • Low Pressure Servo Test • High Pressure Servo Test • Low Pressure Full Leak Test

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

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	Select one or more tests to run unattended. Select the test, then click **Insert** for the test to display in the *Test Sequence* box.
Insert	Inserts the selected test in the *Available Tests* drop-down list.
Skip these tests if the operator does not respond within [*n*] minutes	Check this option if any test requiring operator intervention should be omitted if the operator does not respond within the specified time.
Start test sequence if instrument is idle any time between 00:00:00 and 00:00:00 .	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.

Schedule Diagnostics Test Frequency Fields and Buttons Table (continued)

Field or Button	Description
Test Frequency	Select how often the test is to run unattended.
Test Sequence	Provides the test file identification and estimated run time. A checkmark in the *Intervention Required* column indicates that operator intervention is required. To remove a test from the sequence, select the test, then click **Delete**. Alternatively, click **Clear** to clear the entire table of all entries. To add a test to the test sequence, highlight a row in the *Test Sequence* box, select a test from the *Available Tests* list, then click **Insert**. The new test will be inserted above the highlighted row.

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

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.

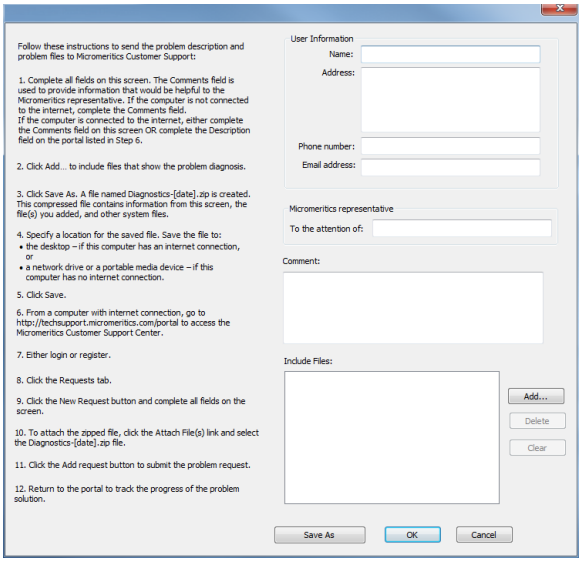
1. To open a diagnostic test report, select a service test report, then click **Open**. Alternatively, double click the report file name.
2. On the *Selected Reports* window, select the reports to display, then click **OK**.
3. The selected reports display on separate tabs at the top of the report window.

SAVE FILES FOR PROBLEM DIAGNOSIS

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

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

- *[instrument model]*.ini
- info[sn].dat
- cal [sn].dat
- [sn].dat
- UserInformation.txt
- Any files selected by the user



To send the problem description and problem files to Micromeritics Customer Support.

1. Complete all fields. The *Comments* field is used to provide information that would be helpful to the Micromeritics representative.
   - If the computer is not connected to the internet, complete the *Comments* field.
   - If the computer is connected to the internet, either complete the *Comments* field on this window OR complete the *Description* field on the portal listed in Step 6.
2. Click **Add** to include files that show the problem diagnosis.
3. Click **Save As**. A file named *Diagnostics-[date].zip* is created. This compressed file contains

information from this window, any added file(s), and other system files.

4. Specify a location for the saved file. Save the file to:
   - the desktop - if this computer has an internet connection, or
   - a network drive or a portable media device - if the computer is not connected to the internet.
5. Click **Save**.
6. From a computer with internet connection, go to http://techsupport.micromeritics.com/portal to access the Micromeritics Customer Support portal.
7. Either log in or register.
8. Click the *Requests* tab.
9. Click **New Request**, then complete all fields on the window.
10. To attach the zipped file, click the *Attach File(s)* link, then select the *Diagnostics-[date].zip* file.
11. Click **Add request** to submit the problem request.
12. 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	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.
Include Files	• **Add.** Click to select additional files to send with this problem diagnosis. To select more than one file, hold down the **Ctrl** key on the keyboard while selecting the files, or hold down the **Shift** key to select a range of files. • **Delete.** Select the file in the *Include Files* box, then click **Delete** to remove the file from the list. • **Clear**. Click to clear all files from the *Include Files* box.
Save As	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.

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

Field or Button	Description
Micromeritics representative	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	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).

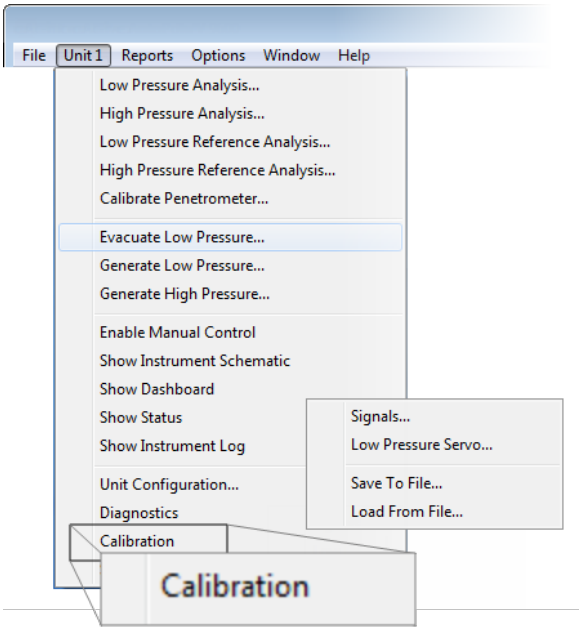
For fields and buttons not listed in this table, see the *Common Fields and Buttons* **section of this operator manual.**

Blank Page

9 CALIBRATION

Unit [n] > Calibration

Use to perform system calibrations.



Calibrations can be saved to a file and reloaded later.

LOAD CALIBRATION FROM FILE

Unit [n] > Calibration > Load from File

Use to load a previously saved calibration file.

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

Changing the calibration may affect the analyzer's performance.

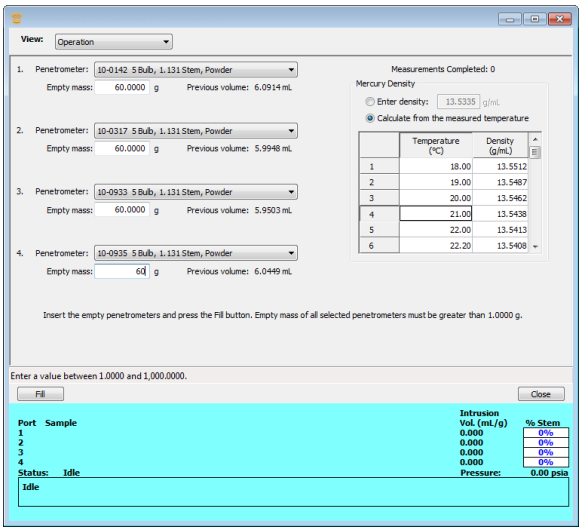
CALIBRATE THE PENETROMETER

Unit [n] > Calibrate Penetrometer

Up to 4 penetrometers can be calibrated simultaneously.

To have the application determine sample density or if blank correction by formula will be used, 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.

The *Penetrometer Volume Calibration* form can be used as a worksheet and to maintain a record, however the system will automatically calculate the Mean Volume and report it. This form can be used to record the process of calculating a penetrometer's volume three times and averaging the calculations. When the *Penetrometer Properties* file is created, use the average volume. See *"Penetrometer Volume Calibration Worksheet" on page I - 3*.



1. Go to ***Unit [n] > Calibrate Penetrometer***.
2. For each penetrometer to be calibrated, click the drop-down arrow to the right of the *Penetrometer* field and select a *Penetrometer Properties* file. If this is a new penetrometer, a new *Penetrometer Properties* file must be created for each new penetrometer, then select the *Penetrometer Properties* file from the drop-down list. A new *Penetrometer Properties* file can be added by clicking **Browse** from the drop-down list and entering the new file name. See *"Penetrometer Properties" on page 4 - 10*.
3. Verify that the *Empty mass* field contains the correct information for each file selected. If necessary, modify the field. The **Fill** button is disabled unless at least one port has a penetrometer selected, and until each port with a penetrometer has an *Empty Mass* value greater than (not equal to) 1.0000.

4. In the *Mercury Density* group box, select either *Enter density* to manually enter the mercury density in the text box or select *Calculate from the measured temperature* to use the temperature and density entered in the table.

   To modify the table parameters, click in the appropriate cell and enter the new parameter.

5. Prepare and load the penetrometers as for a low pressure analysis (see *"Perform a Low Pressure Analysis" on page 5 - 9*). Click **Fill** and wait for the operation to complete, then weigh the penetrometers and enter the *Full Mass* for each. The *Measurements Completed* field will update to include the measurement just finished. The *Full Mass* fields will be initialized to 1.0000. The **Repeat** and **Accept** buttons will be disabled until all selected ports have a *Full Mass* greater than their *Empty Mass*. When a valid *Full Mass* is entered, the penetrometer volume for this measurement is calculated and the *Mean Volume* and standard deviation for all measurements are updated.

6. Repeat these steps as many times as needed (three times are recommended). The system will automatically calculate the *Mean Volume* and report it. Click **Accept** when finished. The *Mean Volumes* will be stored in the selected *Penetrometer Properties* files.

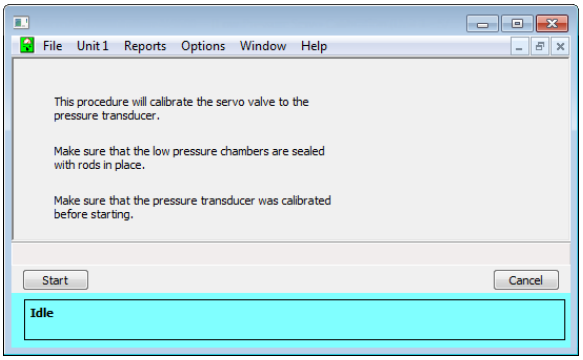
CALIBRATE THE LOW PRESSURE SERVO

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.

1. Ensure the low pressure chambers are sealed with rods in place.
2. Go to ***Unit [n] > Calibration > Low pressure Servo***.



3. Click **Start**. Calibration begins.

SAVE CALIBRATION TO FILE

Unit [n] > Calibration > Save to File

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

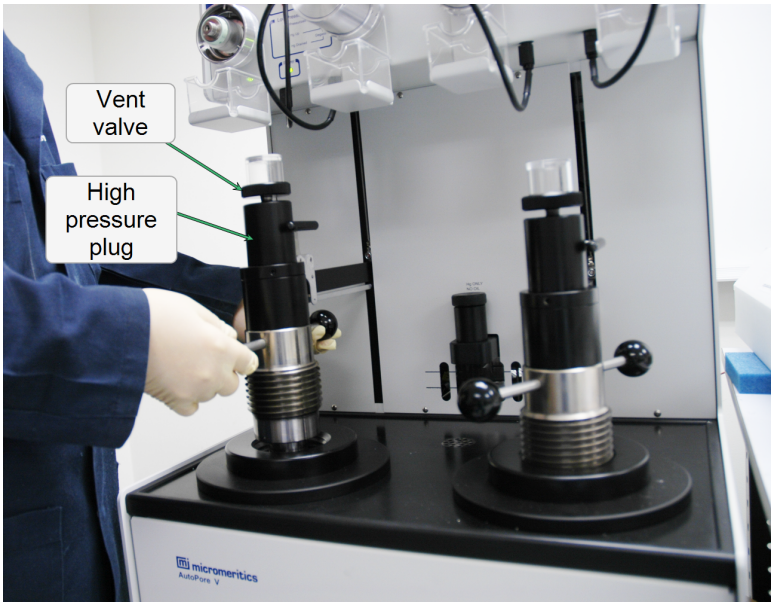
The default file naming convention for calibration files can be used or the file name can be changed. The default file name of 0217-2013-04-25.CAL is interpreted as:

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

10 Hardware Components and Accessory Installation

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.

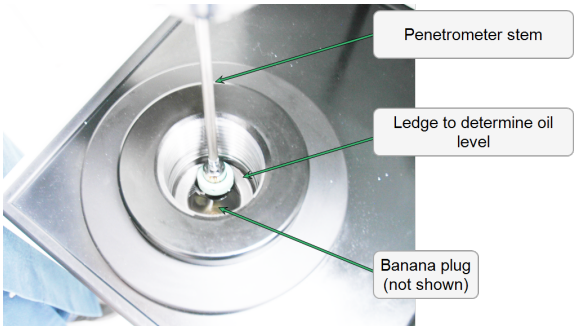


1. Turn the vent valve slowly counter-clockwise to release excess pressure.
2. Unscrew the chamber plug assembly by turning the arms counter-clockwise. Lift the chamber plug assembly as far as it will go. The chamber assembly contains a latching device which automatically locks into place when the assembly is in the topmost position.
3. 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 - 17* and *"Maintain High Pressure Fluid Level" on page 11 - 16*.
4. Gently lower the penetrometer assembly, bulb down, into the chamber until it rests firmly on the banana plug.

5. Pull the latch release down to unlock the chamber plug assembly.Gently lower the assembly while ensuring that the penetrometer is inserted into tthe assembly hole. Lower the assembly to within two or three inches above the pressure chamber.

6. Check the high pressure fluid level in the chamber to ensure that the high pressure fluid is level with the ledge. If the level is below the visible ledge, add high pressure fluid to bring the level to the ledge. If the level is above the ledge, remove fluid to bring the level to the ledge. See

"Maintain High Pressure Fluid Level" on page 11 - 16.

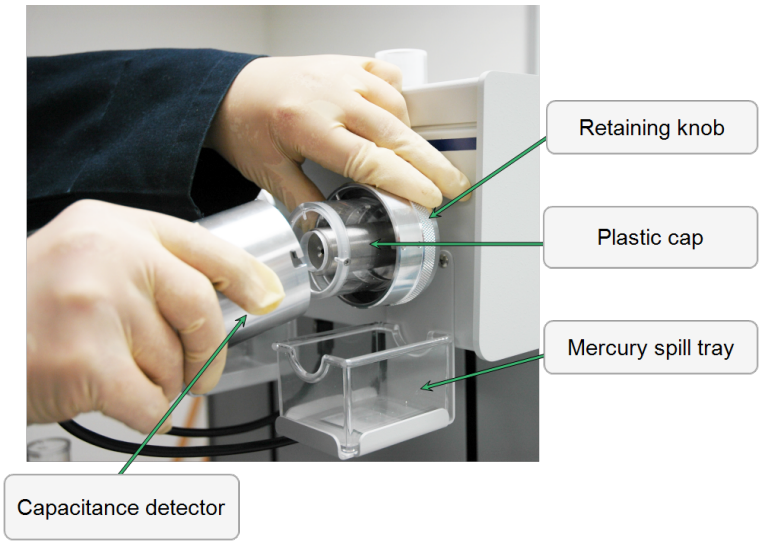


INSTALL A PENETROMETER IN A LOW PRESSURE PORT

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.

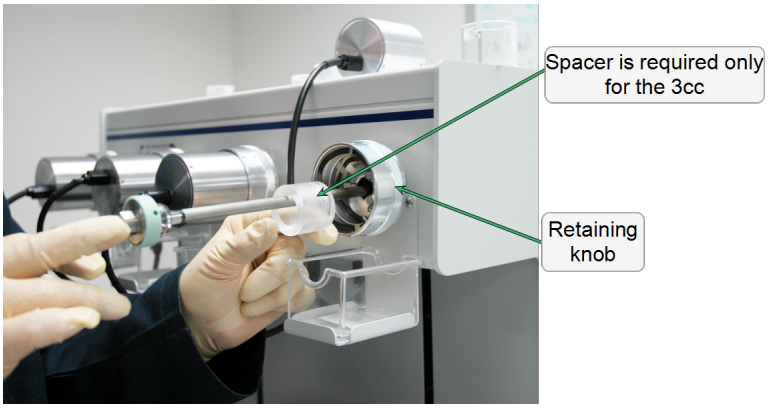
1. Remove the capacitance detector from the low pressure port by turning it counter-clockwise and pulling forward. Place it on top of the instrument or stand it on the surface next to the high pressure chamber.



2. Turn the plastic cap counter-clockwise to remove.
3. Turn the retaining knob counter-clockwise until it turns with little resistance.

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

4. If the low pressure port contains a blank rod, remove it. If it contains a penetrometer, see *"Remove Penetrometer from a Low Pressure Port" on page 10-6*.
5. 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.

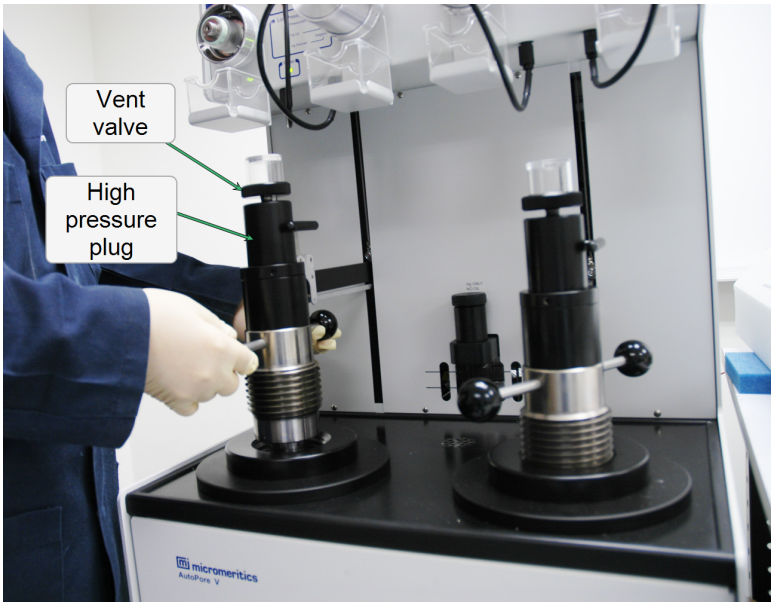


6. Tighten the retaining knob by turning it clockwise until the penetrometer is firmly seated. Do not tighten with excessive force.
7. Place the plastic cover over the penetrometer assembly and turn clockwise to tighten.
8. Install the capacitance detector over the penetrometer and turn clockwise to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



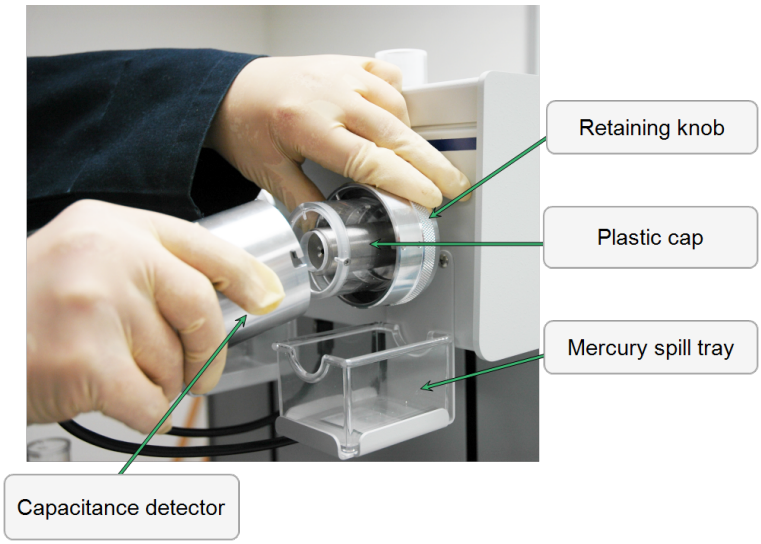
2. Unscrew the chamber plug by turning the arms counter-clockwise. Lift the plug assembly as far as it will go. Fluid begins to drain from the vent valve. Pause a few seconds to allow fluid to drain back into the chamber.
3. Remove the penetrometer assembly. Hold it over the chamber for a few moments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

REMOVE 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 instrument 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 - 9*.

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



3. Turn the plastic cap counter-clockwise and remove.
4. 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.

5. 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.

6. 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. The rack for full penetrometers (part number 962-25827-00) can be used for this purpose.

Blank Page

11 Maintenance and Troubleshooting

The instrument has been designed to provide efficient and continuous service; however, certain maintenance procedures should be followed to obtain the best results over the longest period of time.

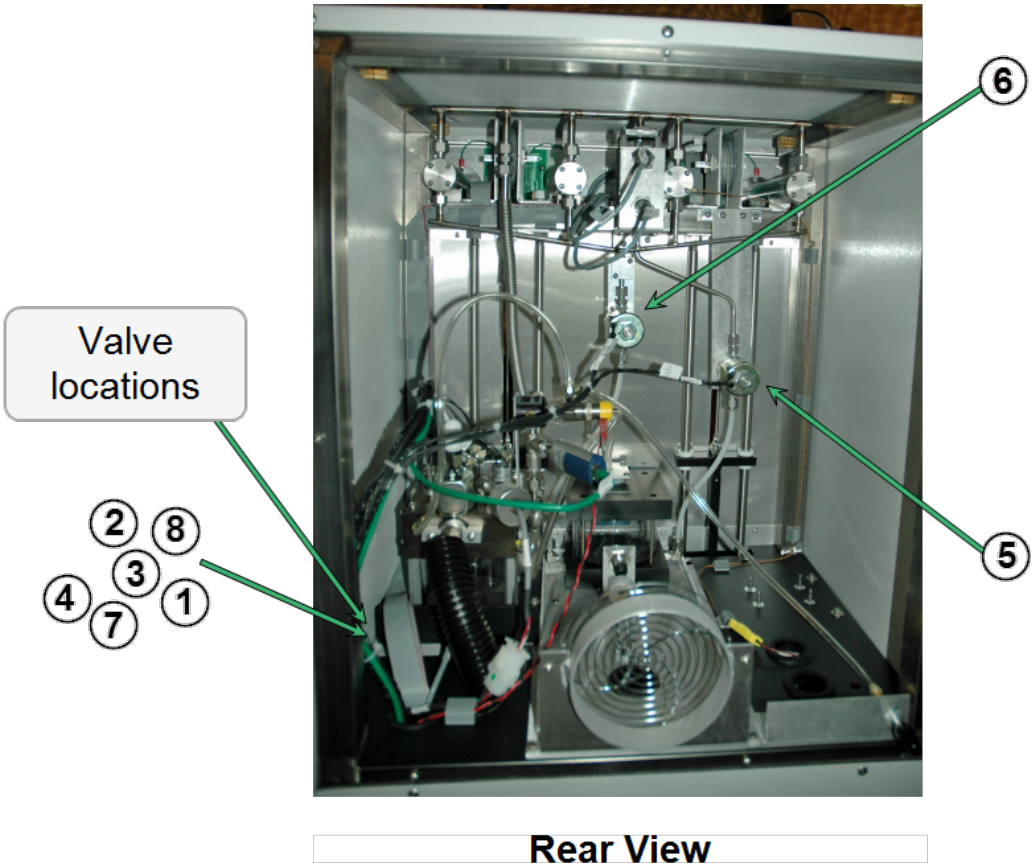
What happened...	Why...	What to do....
Status display is too large for monitor screen.	Monitor resolution is set below 800 × 600.	Refer to the computer operating system manual to reset the monitor resolution at or above 800 × 600.
Difficulty attaining adequate vacuum conditions during low pressure analyses.	Vacuum pump oil is low.	Add vacuum pump oil.
	Vacuum hose not properly connected.	Ensure the connection to the vacuum pump is good and that external clamps are tight.
	Sample contains excess moisture adsorbed from atmosphere.	Prepare samples (prior to loading penetrometer) by heat and / or vacuum to remove moisture. CAUTION: Never heat a sample that has contacted mercury.
	Proper sealing not achieved on penetrometers	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.
	Scratches or other imperfections in either the lip of the penetrometer bulb or the penetrometer cap.	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. Exam-

What happened...	Why...	What to do....
		ination of the surface under low magnification (approximately 20X) helps determine when the surface is free from flaws. 5. Clean the ground surface before regreasing.
	Leaking valves.	Check the valves for leaks. See *"Check Valves for Leaks" on page 11 - 27*.
	Moisture has accumulated in the system.	Remove the accumulated moisture. See *"Remove Moisture from the Instrument" on page 11 - 29*.
The low pressure analysis is complete and the system has returned to near atmosphere, but the mercury drained indicator is not illuminated.	Indicator is not working properly.	Double-check 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. Consult your Micromeritics service representative. If the status display window shows that the mercury has overfilled (the alarm state), see *"Handling Mercury Overfill" on page 11 - 19*. If the status display shows that the mercury degasser is either filled or partially filled, see *"Drain the Low Pressure System" on page 11 - 9*.
	Mercury is not drained. (Status display shows either filled or partially filled.)	See *"Drain the Low Pressure System" on page 11 - 9*.
Mercury warning buzzer sounds in pulses. (Mercury overfill)	Mercury detected by overflow sensor in low pressure port system.	See *"Handling Mercury Overfill" on page 11 - 19*.
Mercury warning buzzer sounds with continuous tone.	Mercury has been improperly drawn into the mercury trap.	Drain excess mercury into the reservoir. see*"Handling Mercury Overfill" on page 11 - 19*. Remove the plug extending down from the mercury trap. The plug is reached through the front door. Facing the instrument, the plug is in the upper, back, right-hand corner. See *"Drain Spilled Mercury Dish" on page 11 - 10*. Position a container beneath the trap before removing the plug.
The high pressure system failed to attain the specified pressure	Low fluid level in the hydraulic pump.	Add fluid to the hydraulic pump. See *"Maintain Hydraulic Pump Fluid Level" on page 11 - 22*.

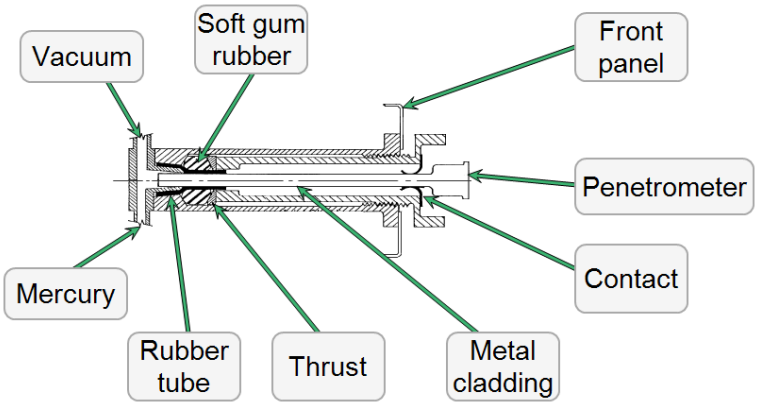
What happened...	Why...	What to do....
High pressure system failed to retain a reasonably constant pressure.	Leakage around the high pressure chamber cap.	Replace the cup seals and backup rings on the high pressure chamber cap. See *"Replace Chamber Cup Seals" on page 11 - 19*.
Cannot attain low pressure data points above atmospheric pressure	Gas supply pressure too low.	Verify gas supply regulator is set at 40 to 45 psig (276 to 310 kPa) and that there is sufficient gas in the supply bottle.

SYSTEM COMPONENTS

LOW PRESSURE SYSTEM COMPONENTS



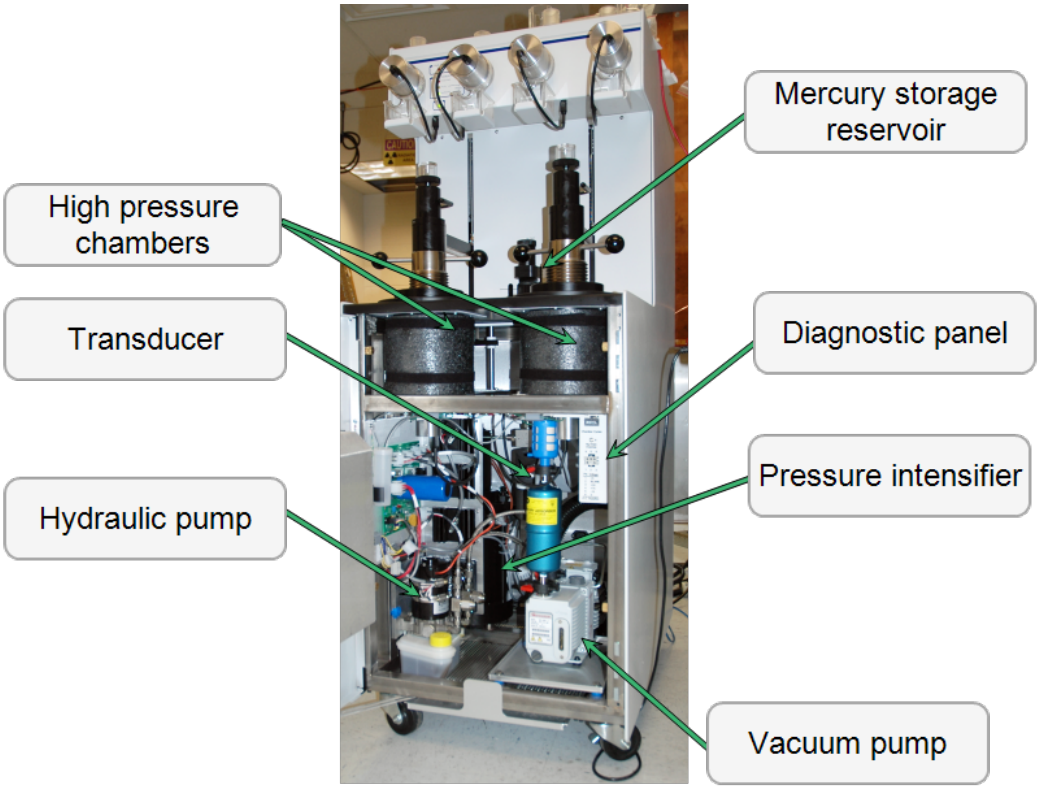
Low Pressure System Valves

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	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. 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).
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 Reser-voir valve	See Vacuum valve [3].
Low pressure ports		A sample encased in a penetrometer is first evacuated, filled with mercury, then pressurized to between 15 and 50 psia. 

Low Pressure System Valves (continued)

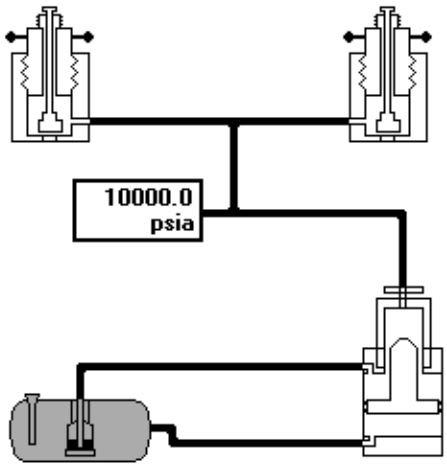
Item #	Component	Description
		Penetrometer stems inserted into a station are sealed for vacuum and against mercury leakage by compression of a soft, gum rubber cylinder near the tip. The rubber is compressed by turning the large knob or pressure collar that protrudes from the front. The inner rim of this knob serves also as the mounting mechanism for a capacitance transducer after the knob has been tightened.
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		Removes trapped gases as mercury passes to low pressure stations then into the penetrometer. **HG Up indicator.** Illuminates when mercury level fills the penetrometers. The sensor completes an electric circuit and flow is stopped by closure of the mercury fill solenoid valve. **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. **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 - 19*.
Mercury trap		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 instrument. 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.

High Pressure System Components



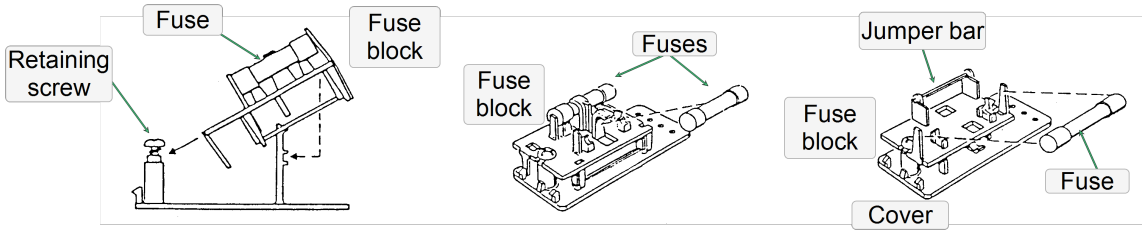
Front View

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. 
Hydraulic pump	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. 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.
Pressure intensifier	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.
Transducer	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.

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.

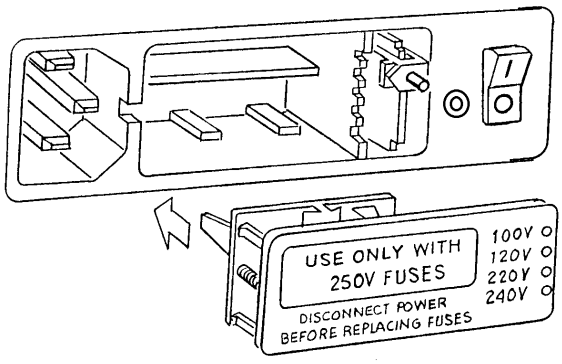
1. Reposition the fuse block, if necessary:
   a. Remove the fuse block retaining screw.
   b. Lift the fuse block from the cover.
   c. Rotate the fuse block.
   d. Mount the fuse block to the cover.
   e. Replace the retaining screw.
2. Insert appropriate fuse for the input power source. Refer to the 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

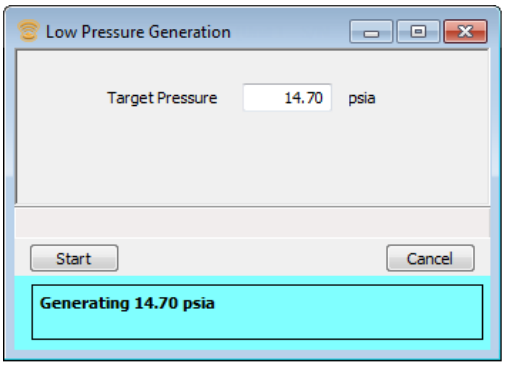
selected.

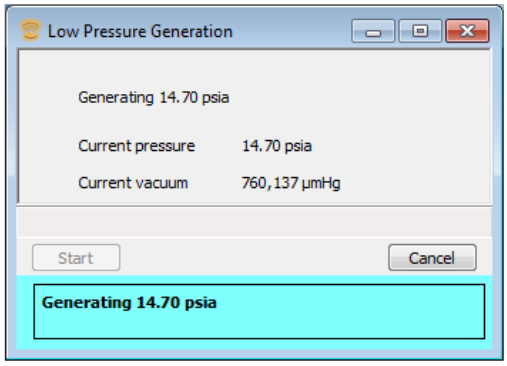


4. Connect the power cord to the analyzer and plug into an appropriate power source.

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.

   The *Hg reservoir evacuated* indicator should be displayed on the instrument 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 - 15*). If the *Hg reservoir evacuated* indicator still is not displayed, contact a Micromeritics Service Representative.

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 - 19*.

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.

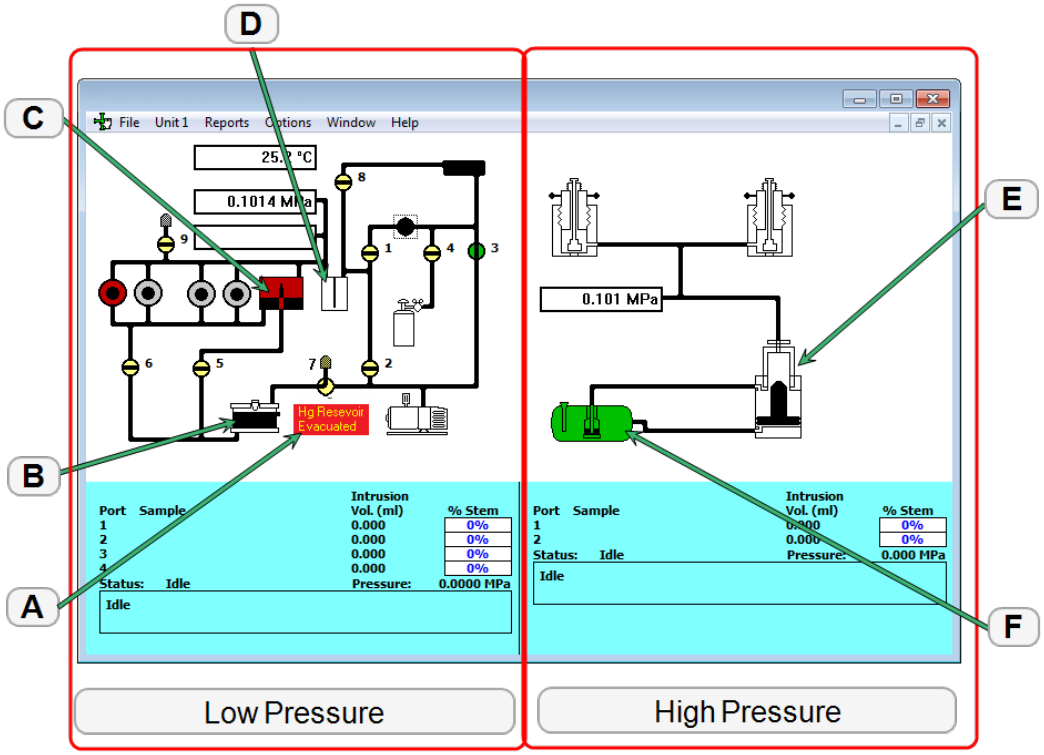
Approximately 3 mm (1/8 in.) of oil should remain in the container to prevent the escape of mercury vapors.

ENABLE MANUAL CONTROL

Unit [n] > Enable Manual Control

Use to enable the manual control of certain system valves and elevator components. 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

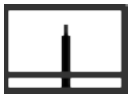
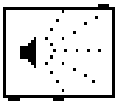
Schematic Components Table (continued)

Schematic Components	Description
8	Evacuation reservoir
A	Hg Reservoir Indicator
B	Mercury reservoir
C	Mercury degasser
D	Mercury trap
E	Intensifier
F	Hydraulic pump
Pressure	• 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.

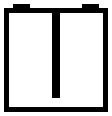
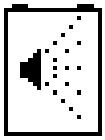
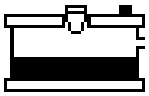
Analyzer Schematic Icon Table

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

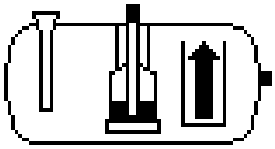
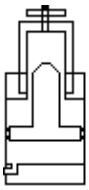
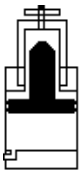
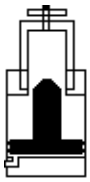
Low Pressure Schematic Icons

Mercury Degasser. Displays the mercury level.			
			
Drained	Partially Filled	Filled	Overfilled. This alarm displays with a red background. This is an alarm state.

Low Pressure Schematic Icons (continued)

Mercury Trap. Displays the state of the mercury trap.	
 Empty	 Contains more than 6 mm of mercury. This is an alarm state.
Mercury Reservoir. Displays the level of mercury in the reservoir.	
 If the mercury level is low, see *"Maintain Mercury Level" on page 11 - 15*.	 Level is OK.
Hg Reservoir Vacuum Switch	
	When illuminated, indicates that mercury reservoir has been evacuated.

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

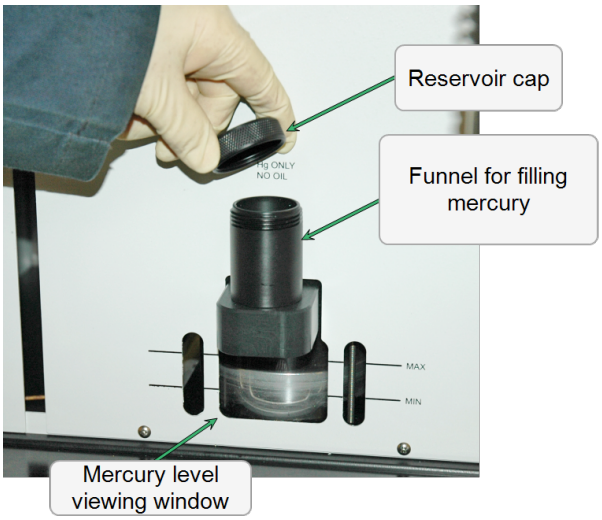
Perform the following preventive maintenance procedures to keep the analyzer operating at peak performance. Micromeritics also recommends that preventive maintenance procedures and calibration be performed by a Micromeritics Service Representative every 12 months.

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.
Low pressure ports	Grease every 600 samples or 3 months.
Mercury dish	Check daily for spills. Service as needed.
Mercury level	Service daily.
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.



The instrument 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 instrument 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.

CAUTION

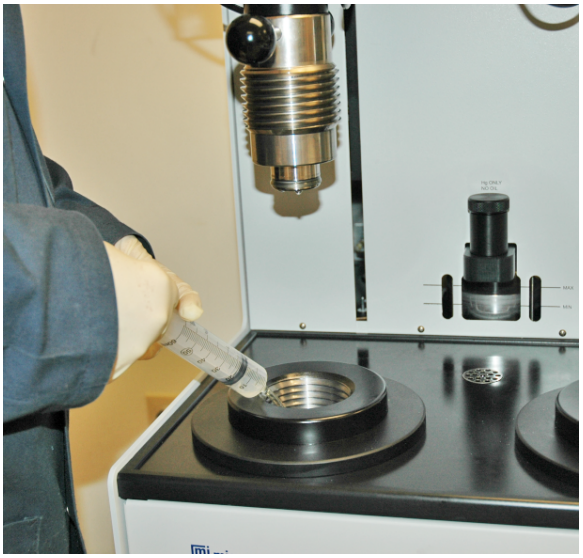
Overfilling may interfere with the vacuum system. Reservoir cap should be finger tight only.

CAUTION

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.

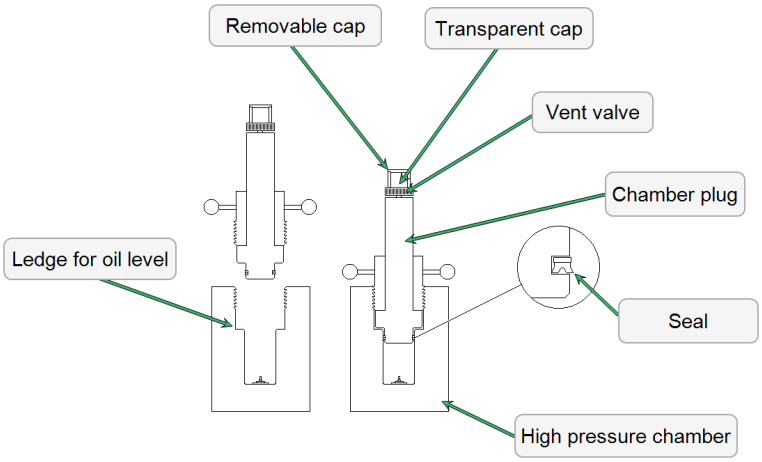


SEAL THE HIGH PRESSURE CHAMBER

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.

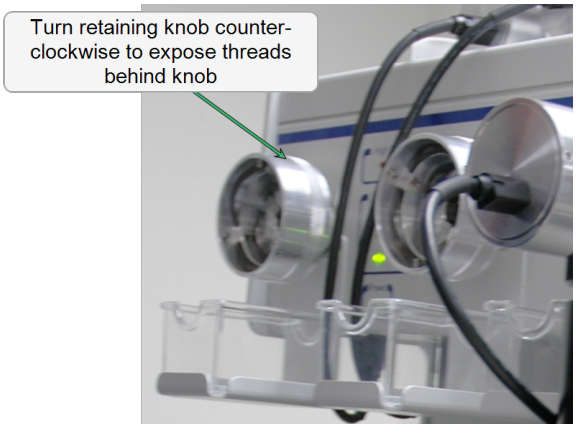
- Large air bubbles may be caught in the chamber. Slowly loosen and tighten the plug approximately 1/2 turn several times. This should cause any air bubbles to rise through the transparent cup to the surface. Any remaining tiny bubbles will not affect the analysis.
- If no fluid is visible in the cup 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. Unscrew the retaining knobs until the threads disengage.



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

HANDLING MERCURY OVERFILL

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

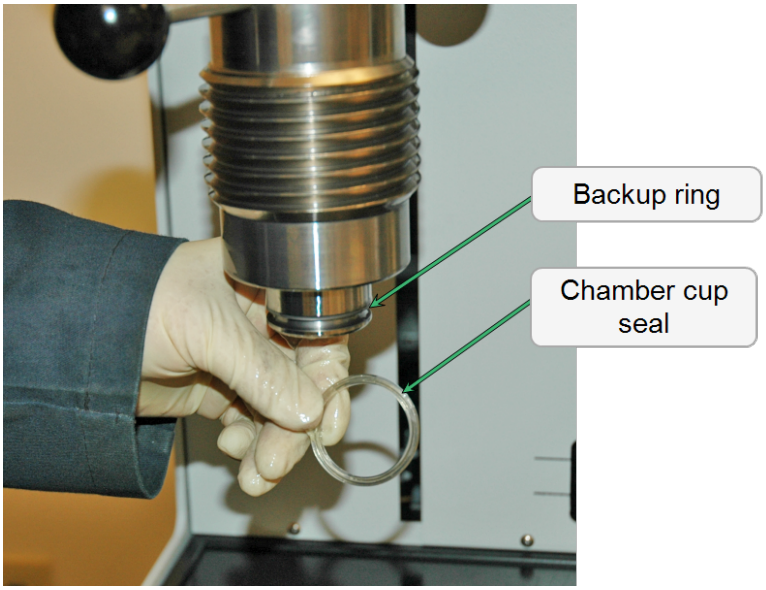
   The *Hg reservoir evacuated* indicator should be displayed on the instrument schematic. If it is not shown, do not proceed to the next step. Instead, verify that the mercury reservoir fill cap is properly sealed on top of the mercury filling funnel (see ***"Maintain Mercury Level" on page 11 - 15***). 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 instrument 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 instrument may be powered off.

REPLACE CHAMBER CUP SEALS

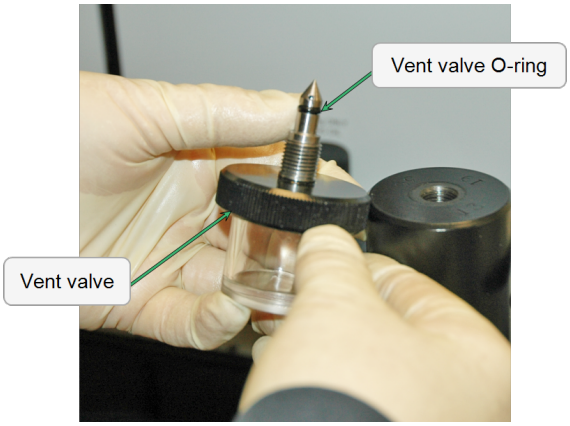
The chamber plug is sealed into the high pressure chamber by means of a cup seal and a backup ring. There is also an O-ring in the vent valve. See ***" Replace the Vent Valve O-ring" on the next page***.These rings should be checked monthly and replaced when the backup ring extrudes, small slivers come 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

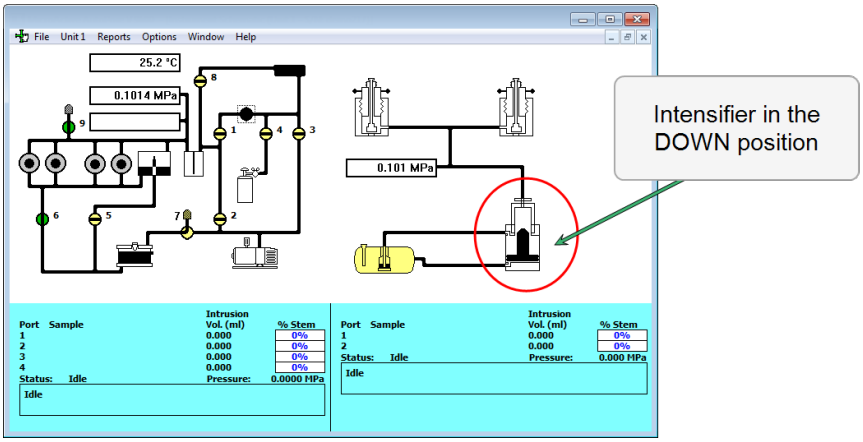
The high pressure fluid should be changed after every 600 samples or 3 months to ensure accurate results. It should also be changed if mercury is spilled into a high pressure chamber. Small drops of mercury in the bottom of the chamber can cause erroneous results.

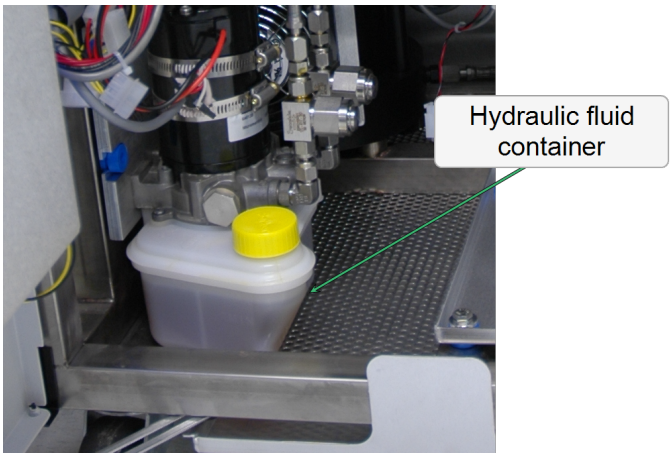
1. Remove the high pressure fluid from the high pressure chambers using the syringe provided.
2. Clean the high pressure chambers using a clean cloth dampened with IPA.
3. When the chambers are completely dry, add new high pressure fluid.

MAINTAIN HYDRAULIC PUMP FLUID LEVEL

The hydraulic pump fluid container is located in the lower left corner inside the instrument'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.





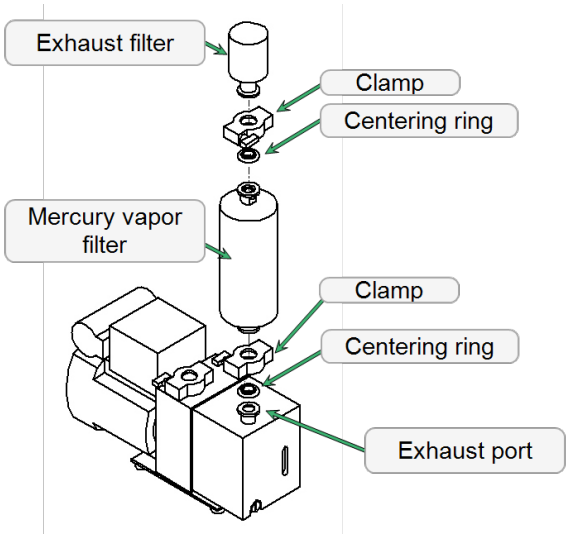
CAUTION Filling the fluid container when the intensifier is in the up position can cause the fluid to overflow the when intensifier is lowered.

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

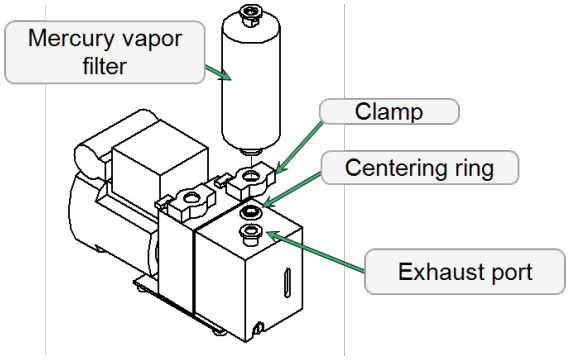
REPLACE VACUUM PUMP EXHAUST FILTER

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 dust 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 instrument 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 instrument.
7. Remove the vacuum pump from inside the instrument.
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.

CAUTION

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 instrument.
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 " on the next page***.

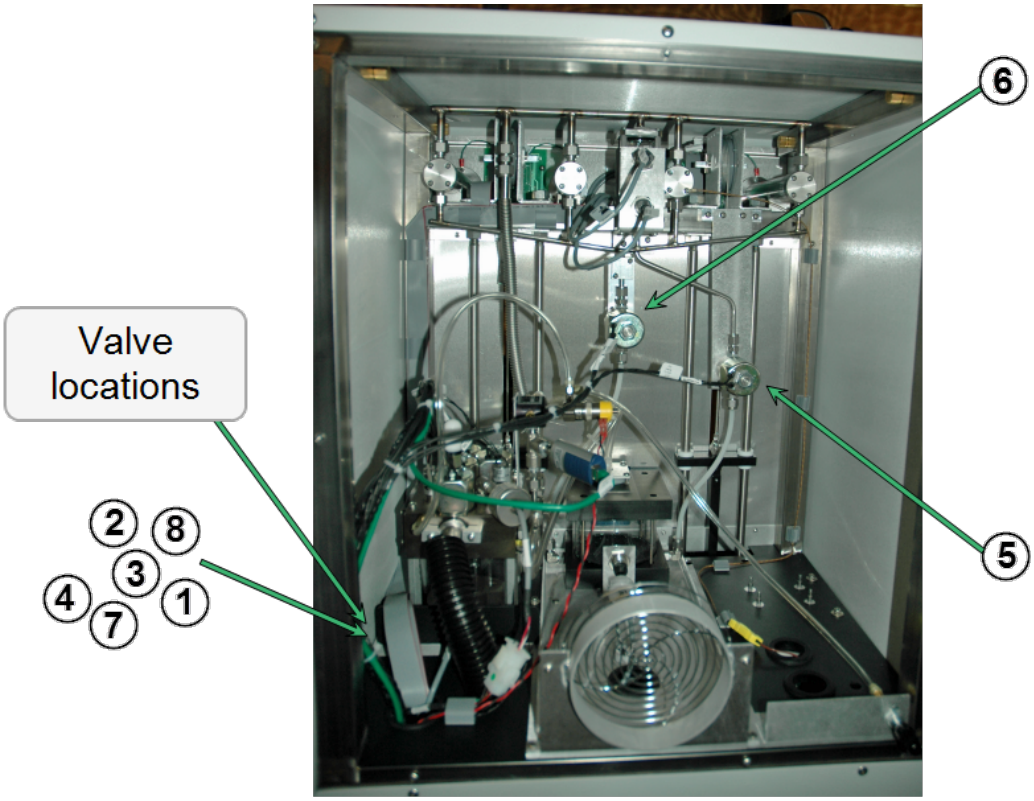
CLEAN VALVES

CAUTION

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 instrument 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.



Rear View

3. 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

4. Place a container below the valves to capture any retained mercury.
5. Remove the plunger housing from each using the special spanner wrench from the accessory kit.
6. 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.
- Use only a mild detergent in water to clean safety shields. The use of isopropyl alcohol can damage the shield surface.

REMOVE MOISTURE FROM THE INSTRUMENT

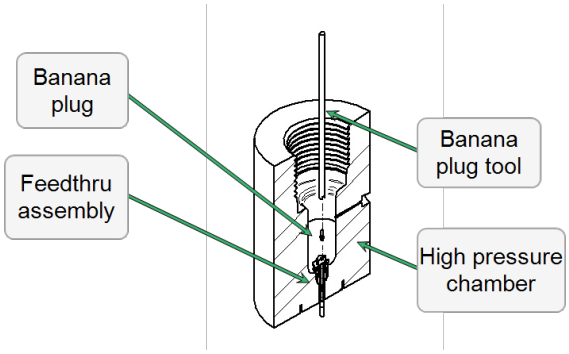
A difficult-to-detect vacuum problem arises if moisture is allowed to collect in the system. A probable accumulation point is the restricting frits that control flow rates. The best way to remove moisture is to evacuate the instrument at full rate for several days. If the indicated vacuum continues to 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 pre-cleaning samples in a drying oven.

REPLACE THE BANANA PLUG

HIGH PRESSURE CHAMBER

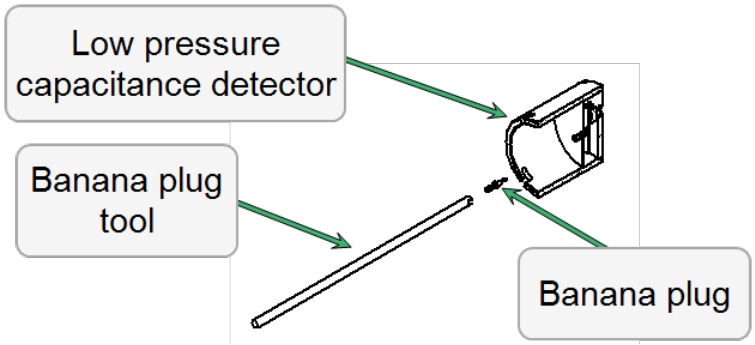
1. Remove the fluid from the high pressure chamber using the syringe provided.
2. Remove any mercury droplets found in the high pressure chamber. Clean the high pressure chamber using a clean dry cloth.
3. Locate the feedthru assembly in the bottom of the high pressure chamber. Remove the banana plug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

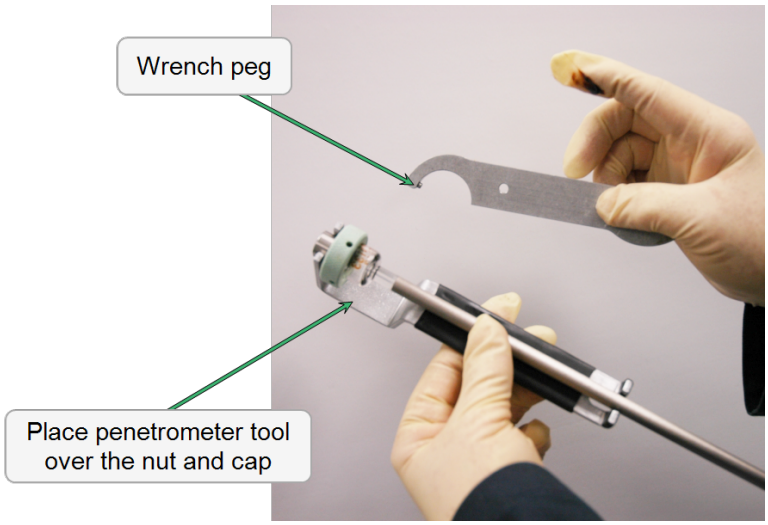
LOW PRESSURE CAPACITANCE DETECTOR

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.

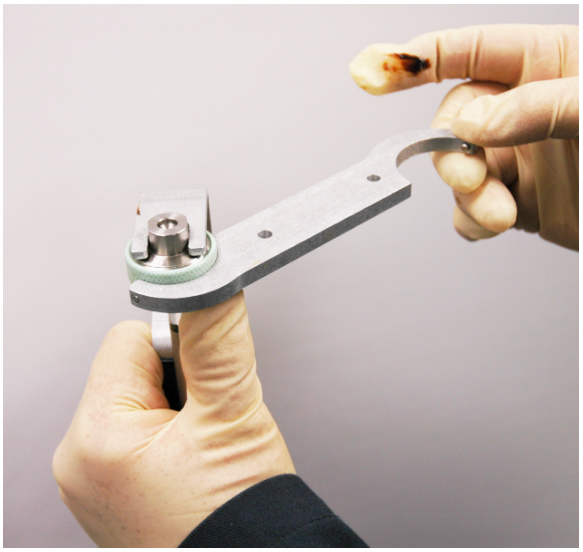
PENETROMETER NUT



REMOVE THE PENETROMETER NUT

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

1. Place the penetrometer tool over the nut.
2. 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.
3. Position the wrench so the peg on the wrench fits into the notch on the nut.



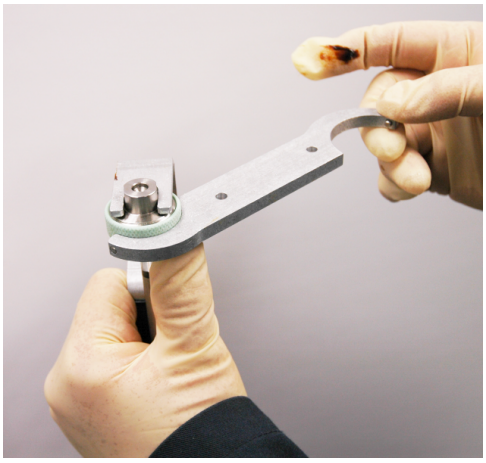
4. While holding the penetrometer tool, turn the wrench clockwise to loosen and remove the nut.

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.

RECOVER FROM POWER FAILURE

If the application is exited while analyses are in progress, the analyses continue and data are collected. Reports already queued will print; however, reports for new data collected after exiting the program will not print.

If power is interrupted to the computer only, analyses continue, and data are collected, but not permanently saved on the computer disk. When power is restored to the computer, any data collected after power was interrupted are permanently saved to the computer, and the instrument can be operated as usual. Changes made to open and unsaved files are lost when power is interrupted.

If power is interrupted to the analyzer, data being collected or transmitted are corrupted or lost.

Blank Page

12 Parts and Accessories

Order system components and accessories using one of the following methods:

- Call our Customer Service Department at 1-770-662-3636
- Email orders to Orders@Micromeritics.com
- Contact your local sales representative

Part Number	Item and Description
Analyzer Optional Equipment	
090-00000-00	Mercury QuikVac 090 - provides safe and convenient method for spilled mercury droplet removal using integrated vacuum pump and reservoir in a portable device
942-33021-00	Exhaust fan kit, for ducting air from around the working surface to an exhaust system
AutoPore V Systems	
960-50000-00	AutoPore V 9605 Automatic Mercury Porosimeter 33,000 psia maximum (no vacuum pump)
960-50000-11	AutoPore V 9620 Automatic Mercury Porosimeter 60,000 psia maximum with 110/120V oil-sealed vacuum pump system
960-50000-22	AutoPore V 9605 Automatic Mercury Porosimeter 33,000 psia maximum with 220/240V oil-sealed vacuum pump system
962-00000-00	AutoPore V 9620 Automatic Mercury Porosimeter 60,000 psia maximum (no vacuum pump)
962-00000-11	AutoPore V 9620 Automatic Mercury Porosimeter 60,000 psia maximum with 110/120V oil-sealed vacuum pump system
962-00000-22	AutoPore V 9620 Automatic Mercury Porosimeter 60,000 psia maximum with 220/240V oil-sealed vacuum pump system
Gas Bottle Accessories	
004-25549-00	Reducer, 1/8 in. tube × 1/4 in. tube
004-33601-00	Expansion kit; adds an additional outlet to the gas regulator
004-33602-00	Pressure relief kit; prevents excessive gas pressure in the event of regulator failure (not to be used with toxic gases)
004-62200-58	Gas regulator, CGA 580 fitting, 100 psig (He, N2, Kr, Ar)
230-02001-00	Gas inlet line, 1/8 in. × 6 ft., copper
Mercury Safety	
004-32031-00	Dish, polystyrene, 2.5 × 3.3 × 2
962-25859-00	Mercury vapor filter assembly
962-29600-00	Mercury vapor detector pump
962-29601-00	Mercury vapor detection tube, 10 Pack

Part Number	Item and Description
Operating Supplies	
004-16004-01	Mercury, 5 lbs
004-16007-00	Apiezon-H vacuum grease, 25 g; operating range is 15 to 250 °C
004-16011-00	Oil, synthetic, for hydraulic pump
008-16045-00	Dow Corning silicone grease, high vacuum, 150g tube
920-16001-00	High-pressure fluid, 1 liter, a proprietary blend of oils
930-54601-00	Cleaning brush, 0.4cc stem, used to aid in the cleaning of penetrometers
930-54602-00	Cleaning brush, 1.1cc stem
930-54603-00	Cleaning brush, 1.8cc stem
942-25823-00	Syringe, 50cc, used to withdraw high-pressure fluid when cleaning chambers
962-33601-00	Operating supplies (6 month supply), spanner wrench for solenoid valves, modified banana plugs, mercury vapor detection tubes (10 pack), mercury vapor filter, high pressure fluid
962-33602-00	Extended operating supplies (12 month supply), spanner wrench for solenoid valves, modified banana plugs, mercury vapor detection tubes (10 pack), mercury vapor filter, apiezon vac grease, high pressure fluid
Operator Manuals and Software	
962-33004-00	Software package, AutoPore V (includes software and operator manual)
962-42800-00	AutoPore V operator manual; includes binder, dividers, and contents
Penetrometer Accessories	
942-25863-00	Spacer for 15cc penetrometers
950-09802-00	Penetrometer holder
950-09803-00	Penetrometer wrench
950-25820-00	Penetrometer rack (for storage of penetrometers when not in use)
950-25822-00	Support for weighing penetrometers
950-25831-00	Closure set for 3 and 5 cm^3 penetrometers; includes nut and seal
950-25831-01	Nut for 3cc and 5cc penetrometers
950-25831-02	Seal (metal cap) replacement for 3cc and 5cc sample volume penetrometers
950-25831-03	Spacer for 3cc and 5cc penetrometers
950-25832-00	Closure set for 15 cm^3 penetrometers; includes nut and seal
950-25832-01	Nut for 15cc penetrometer
950-25832-02	Seal (metal cap) replacement for 15cc sample volume penetrometers
950-33605-00	Closure kit for 3 and 5 cm^3 penetrometers; includes six closure sets, tools, and instructions
950-33606-00	Closure kit for 15 cm^3 penetrometers; includes six closure sets, tools, and

Part Number	Item and Description
	instructions
962-25807-00	Penetrometer enclosure assembly (for viewing penetrometers during mercury fill process)
962-25827-00	Rack for full penetrometers
Penetrometer Assemblies for Powder Samples	
920-61702-01	Glassware only for 950-61702-00
920-61704-01	Glassware only for 950-61704-00
920-61706-01	Glassware only for 950-61706-00
920-61708-01	Glassware only for 950-61708-00
920-61710-01	Glassware only for 950-61710-00
920-61712-01	Glassware only for 950-61712-00
920-61714-01	Glassware only for 950-61714-00
920-61716-01	Glassware only for 950-61716-00
950-61702-00	Penetrometer, powder; 15cc sample volume, 0.37cc intrusion volume
950-61704-00	Penetrometer, powder; 15cc sample volume, 1.06cc intrusion volume
950-61706-00	Penetrometer, powder; 15cc sample volume, 1.72cc intrusion
950-61708-00	Penetrometer, powder; 5cc sample volume, 0.37cc intrusion volume
950-61710-00	Penetrometer, powder; 5cc sample volume, 1.06cc intrusion volume
950-61712-00	Penetrometer, powder; 5cc sample volume, 1.72cc intrusion volume
950-61714-00	Penetrometer, powder; 3cc sample volume, 0.39cc intrusion volume
950-61716-00	Penetrometer, powder; 3cc sample volume, 1.12cc intrusion volume
Penetrometer Assemblies for Solid Samples	
920-61701-01	Glassware only for 950-61701-00
920-61703-01	Glassware only for 950-61703-00
920-61705-01	Glassware only for 950-61705-00
920-61707-01	Glassware only for 950-61707-00
920-61709-01	Glassware only for 950-61709-00
920-61711-01	Glassware only for 950-61711-00
920-61713-01	Glassware only for 950-61713-00
920-61715-01	Glassware only for 950-61715-00
920-61724-01	Glassware only for 950-61724-00
920-61725-01	Glassware only for 950-61725-00
950-61701-00	Penetrometer, solids; 15cc sample volume, 0.37cc intrusion volume
950-61703-00	Penetrometer, solids; 15cc sample volume, 1.06cc intrusion volume
950-61705-00	Penetrometer, solids; 15cc sample volume, 1.72cc intrusion volume

Part Number	Item and Description
950-61707-00	Penetrometer, solids; 5cc sample volume, 0.37cc intrusion volume
950-61709-00	Penetrometer, solids; 5cc sample volume, 1.06cc intrusion volume
950-61711-00	Penetrometer, solids; 5cc sample volume, 1.72cc intrusion volume
950-61713-00	Penetrometer, solids; 3cc sample volume, 0.39cc intrusion volume
950-61715-00	Penetrometer, solids; 3cc sample volume, 1.12cc intrusion volume
950-61724-00	Penetrometer, solids; 15cc sample volume, 3.01cc intrusion volume
950-61725-00	Penetrometer, solids; 15cc Sample Volume, 3.86cc Intrusion
Penetrometer Simulators	
930-17801-00	Penetrometer simulator, 82 pF, used to verify capacitance (intrusion) calibration
930-17801-01	Penetrometer simulator, 150 pF, used to verify capacitance (intrusion) calibration
930-17801-05	Penetrometer simulator, 15 pF, used to verify capacitance (intrusion) calibration
Reference Material	
004-16822-00	Silica Alumina SA,PV
Reference Volume	
004-25646-00	O-ring, 1mm × 8mm, Buna-N, 70 Durometer (o-ring used in capsule assemblies)
940-14800-00	Retaining clip
940-25600-00	Membrane, 8 microns
940-25600-01	Membrane, 3 microns
940-34800-00	Capsule assembly Hg volume - small
940-34800-01	Capsule assembly Hg volume - large
960-34801-00	Mercury volume reference kit - used to verify that the accuracy of mercury intrusion volume measurements, made by the AutoPore and it's associated penetrometer, are within specifications
Replacement Parts	
003-21273-00	Banana plug, unmodified (for use with the capacitance detector assemblies)
003-51123-00	Fuse, 3AG SLO-BLO,6-1/4A, 250V
003-51134-01	Fuse, 5x20mm, SLO-BLO,5A, CE
003-63801-01	Cable, Ethernet straight-thru; for connecting instrument to control module (computer)
004-25034-00	O-ring, -008 70 Durometer Buna-N (replacement O-ring for pressure relief valve)
004-25297-00	Backup ring for chamber plug seal

Part Number	Item and Description
004-25652-00	O-ring, -217 75 Durometer Viton (O-ring used in mercury reservoir cap)
920-33616-00	High pressure vessel maintenance kit; (contains 2 (ea.) -008 O-rings for pressure relief valve, back-up rings, u-shaped cup seal, and modified banana plugs, along with 6 (ea.) penetrometer contacts, and 1 (ea.) cup seal instruction sheet
922-25890-00	Seal for high pressure chamber plug
922-33607-00	Cup seal kit; contains 1 (ea.) u-shaped cup seal, back-up ring, and instruction sheet
930-25849-00	Banana plug, modified for use with feedthru in high pressure chamber
942-25600-00	Cover for spilled mercury dish
962-33002-00	Mercury reservoir cap and O-ring
Tools	
004-54039-00	Spanner wrench for solenoid valves
922-09808-00	Tool, banana plug
Vacuum Pumps and Accessories	
004-16003-01	Oil, vacuum pump, 1 Liter
004-25509-00	Clamp, NW 10/16
004-25630-00	Centering ring, NW 16
004-27040-00	Filter, for vacuum pump exhaust
062-62801-11	Vacuum pump with built-in anti-suckback valve, 100/120 VAC, includes hose kit
062-62801-23	Vacuum pump with built-in anti-suckback valve, 220/240 VAC, includes hose kit

Blank Page

A Blank and Sample Compression Corrections for Mercury Porosimetry

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

Baseline Errors

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

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

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

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

APPROACHES FOR ERROR COMPENSATION

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

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

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

The third technique provides the highest degree of compensation possible and can be attained when the exact penetrometer to be used later is loaded with a nonporous sample of the same 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.

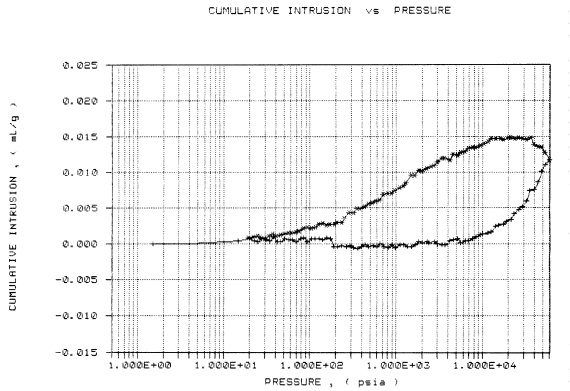


Figure 1

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.

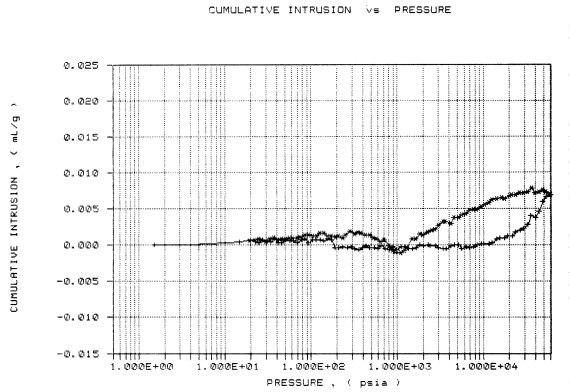


Figure 2

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.

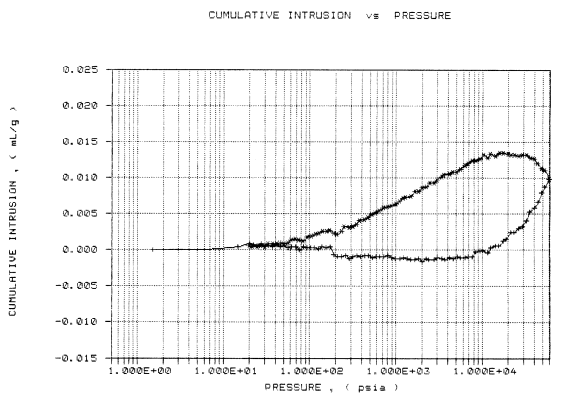


Figure 3

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.

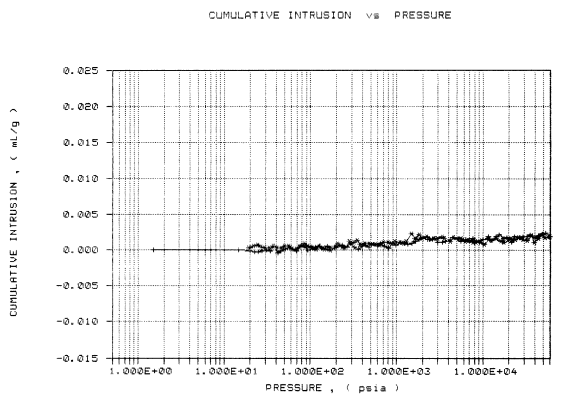


Figure 4

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.

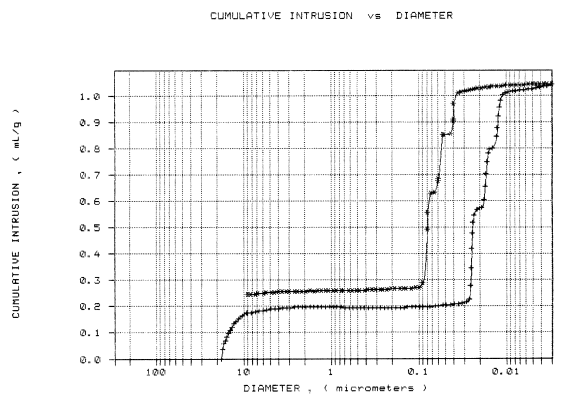


Figure 5

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.

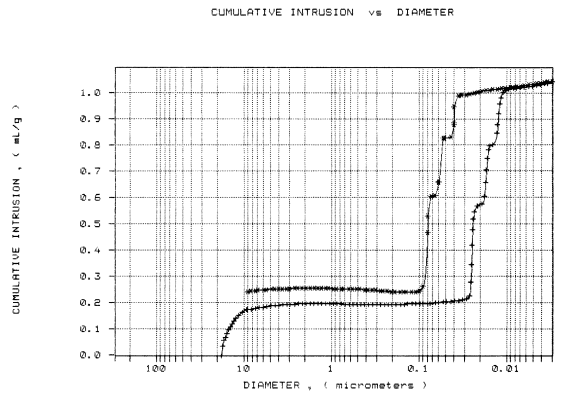


Figure 6

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.

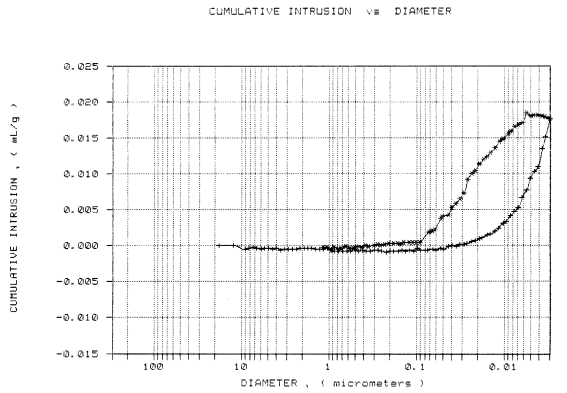


Figure 7

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.

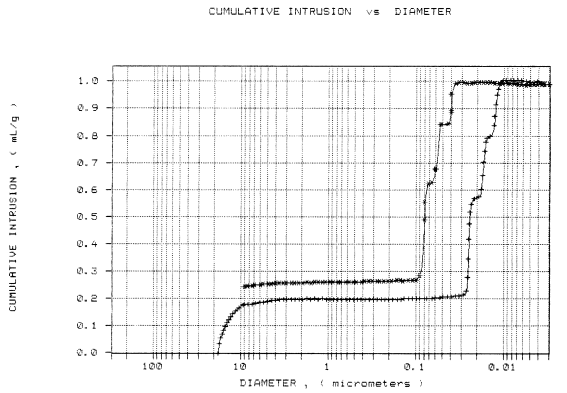


Figure 8

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 cancelled because the compression coefficient of mercury is close to that of the controlled pore glass used as sample. Many solid materials have compression coefficient fairly close to that of mercury, making this a very effective means of blank correction in many cases.

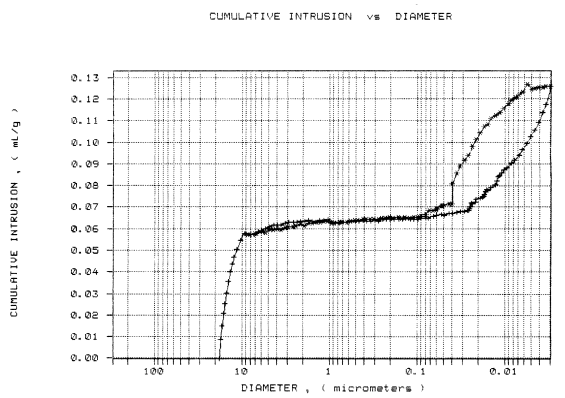


Figure 9

Uncorrected data from an essentially nonporous sample of the same type of glass shown in Figure 5. The 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.

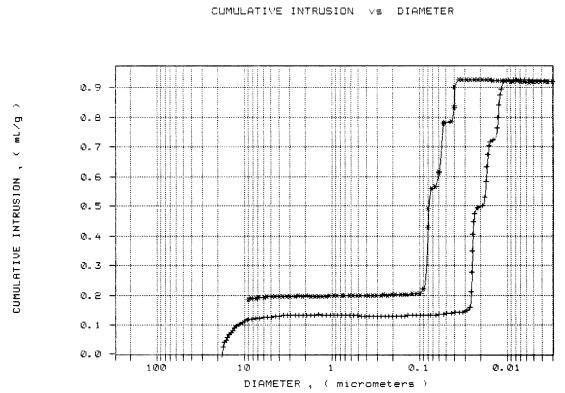


Figure 10

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 ºC 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 DATA REDUCTION

Data for presentation in tabular and plot form is calculated in the following manner:

P_i	=	head-corrected pressure as stored
V_{ri}	=	intrusion volume as stored
θ	=	user-entered contact angle
γ	=	user-entered surface tension
W_s	=	user-entered sample mass
W_p	=	user-entered mass for penetrometer
W_{psm}	=	user-entered mass for penetrometer + sample + mercury
V_p	=	user-entered volume for penetrometer
V_c	=	user-entered volume for capillary (stem)
V_{bup}	=	bulk volume at the filling pressure
V_{bup}	=	bulk volume at the user-entered pressure
ρ_{Hg}	=	user-entered density for mercury

$$\text{WASHCON=Washcon constant} = \frac{10^4 \mu m / cm}{\left(68947.6 \ \ dynes / cm^2\right)\left(psia\right)} = 0.145038$$

For all calculations requiring interpolation between collected data points, an Akima[1] method semi-spline is used.

Diameter for the ith point is:

$$D_i = \frac{WASHCON \ \ \gamma(-4\cos\theta)}{P_i}$$

Radius for the i^{th}point is:

$$R_i = \frac{D_i}{2}$$

Cumulative specific intrusion volume for the i^{th}point is:

$$I_i = \frac{V_i}{W_s}$$

1) "A New Method of Interpolation and Smooth Curve Fitting Based on Local Procedures," Journal of the Association of Computing Machinery, 17(4) 1970, 589-602.

Mean diameter for the ithpoint is:

$$Dm_i = \frac{D_i + D_{i-1}}{2}$$

Incremental specific intrusion volume for the thpoint is:

$$Ii_i = I_i - I_{i-1}$$

Incremental specific pore area for the thpoint is:

$$Ai_i = \frac{4 \times Ii_i}{Dm_i}$$

Cumulative specific pore area for the thpoint is:

$$A_i = Ai_i + Ai_i + \ldots + Ai_i$$

If more than 8 data points are available, differential and log differential specific intrusion volume are calculated as follows.

Differential and log differential data are the 1st derivative of the cumulative specific intrusion volume (all) data as a function of calculated log diameter, normalized by the diameter or log diameter interval. This derivation is comprised of four transformations.

1. Interpolation of cumulative specific intrusion volume vs. log diameter is made to get cumulative specific intrusion volume corresponding to evenly spaced log diameters.
2. The uniform cumulative specific intrusion volume data are then subjected to a 1st derivative calculation, using a 9-point smoothing method. This gives the desired differential data in terms of uniform intervals of collected data.
3. Log differential data are normalized by dividing by the log diameter interval between points. Since the points are evenly log spaced, this interval is the same for all points. Differential data are normalized by dividing by the diameter interval between points. Since the points are evenly log spaced, this interval is larger for larger diameters.
4. Interpolation of the differential or log differential data vs. log diameter is made to get data corresponding to collected data points.

If 8 or fewer data points are available, differential and log differential specific intrusion volume are calculated as:

Differential specific intrusion volume by diameter for the ith point is:

$$Id_i = \frac{-Ii_i}{D_i - D_{i-1}}$$

Log differential specific intrusion volume by diameter is:

$$I1d_i = \frac{-Ii_i}{\log D_i - \log D_{i-1}}$$

Differential specific intrusion volume by radius for the ithpoint is:

$$Ir_i = \frac{-Ii_i}{R_i - R_{i-1}}$$

Log differential specific intrusion volume by radius is:

$$I1r_i = \frac{-Ii_i}{R_i - R_{i-1}}$$

Total intrusion volume is:

$$V_{tot} = V_j$$

where the jthpoint is the first such that:

$$P_{j+1} \leq P_j - 10 \quad \text{and} \quad P_{j+1} \leq P_j \times 0.995$$

Total specific intrusion volume is:

$$I_{tot} = \frac{V_{tot}}{W_s}$$

Percent of total specific intrusion volume for the ith point is:

$$Ip_i = \frac{100 \times I_i}{I_{tot}}$$

Total specific pore area is:

$$A_{tot} = A_j$$

for point j as defined above.

Median diameter by volume is:

$$D_{mv} = D_k$$

where

$$l_k = \frac{I_{tot}}{2}$$

and P_k is interpolated from I_k and the collected data, and D_k is calculated from P_k

Median diameter by area is:

$$D_{ma} = D_k$$

where

$$I_k = \frac{I_{tot}}{2}$$

and P_k is interpolated from A_k and the collected data, and D_k is calculated from P_k.

Average diameter is:

$$D_{av} = \frac{4 \times I_{tot}}{A_{tot}}$$

BLANK CORRECTION BY FORMULA

For equilibration time 6 seconds: $X = \log\left(\frac{T}{6}\right)$

For equilibration time < 6 seconds: $X = 0.0$

$$A_i = \left[1.23 \times 10^{-7} + 2.67 \times 10^{-7} X\right] - V_p\left[1.78 \times 10^{-7} + 1.0 \times 10^{-8} X\right]$$
$$+ V_m\left[1.64 \times 10^{-7} + 2.4 \times 10^{-8}\right]$$

For intrusion,

$$B = A_1 P_i + A_2 P_i^2$$

For extrusion points ≥ 1000 psia,

$$B = A_1 P_i + A_2 P_i^2 + 8.85 \times 10^{-3}\left(1\frac{P_i}{60000}\right)$$

For extrusion points < 1000 psia,

$$B = A_1 P_i + A_2 P_i^2 + 8.7 \times 10^{-6} P_i$$

Blank-corrected intrusion volume for the i_{th} point is:

$$V_i = Vr_i - B$$

where

T	=	equilibration time in seconds
V_m	=	volume of mercury in penetrometer; where, volume of mercury = $\frac{Wpsm - Ws - Wp}{Ym}$
P_i	=	pressure for this data point
V_i	=	corrected intrusion volume

Blank correction by file is described in *"Create Blank Correction File" on page 3 - 8*.

Bulk volume is: $$V_b = V_p - V_m$$

Bulk density is: $$Y_b = \frac{W_s}{V_{bfp} - V_{bup}}$$

Skeletal volume is: $$V_s = V_b - V_{tot}$$

Skeletal density is: $$Y_s = \frac{W_s}{V_s}$$

Porosity % is: $$P_{pc} = \frac{100 \times V_{tot}}{V_b}$$

Percent capillary used is: $$V_{pc} = \frac{100 \times V_{tot}}{V_c}$$

COMPUTATION ALGORITHM FOR VOLUMETRIC PRESSURE COEFFICIENTS OF COMPRESSIBILITY

The data acquired during the AutoPore run is examined to determine that at least seven intrusion data points having progressively ascending pressures have been designated for use in the computation. Note that the intrusion values which will initially be referred to are not *specific* values. They are "total" values never having been divided by the sample material mass. Later, it will be necessary to shift to specific values.

The specified blank data are examined to determine that at least seven blank intrusion data points having progressively increasing pressures are available to use with the specified pressure computation range. Of the seven, the pressure values of the two blank data end points must fall within 5 % (either above or below) of the two end points of the sample material run data.

Interpolation by spline curve polynomial or other suitable technique is to be applied to the blank data to allow computation of blank intrusion volumes at pressures which exactly match those in the experimentally acquired data i.e., take a pressure from the acquired data, enter the interpolation routine and find and save the blank intrusion volume which would correspond to that exact pressure. Repeat this for each pressure value in the acquired data set.

Pointwise at each experimental pressure value, subtract the blank intrusion values as interpolated above from the experimentally acquired data intrusion values to give a "blank-corrected acquired data intrusion values set" or more simply "blank corrected data" for short.

Assume that at each experimental pressure, *Pn*, the corresponding blank corrected intrusion, *V(Pn)*, is computed using the second order polynomial expression

$$V\left(P_n\right) = V_0 + B^{*}P_n + C^{*}P_n^{\,2}$$

where

V_0	=	the exact volume of the sample material computed as the ratio of the sample mass and the sample density supplied by the user or, alternatively, supplied as the pre-measured sample volume by the user;
B	=	the linear pressure coefficient of volumetric compressibility must be a negative real number to avoid violation of fundamental physical laws; and
C	=	the quadratic pressure coefficient of volumetric compressibility.

Construct the summation of differences as follows and solve for the values of *B* and *C* which produce the least squared error:

$$\sum_{n=1}^{N_{\max}} \left[V\left(P_n\right) - \left(V_0 + B * P_n + C * P_n^{\,2}\right) \right]^2 = \text{minimum}$$

Where N_{max} is the index of the uppermost blank corrected data point. Now it is necessary to stop using total values and change to the use of specific values; convert the total values *B* and *C* to specific values, *b* and *c*, by dividing them by the sample material volume.

The first and second order pressure coefficients of volumetric compressibility of mercury must be added to the computed first and second order coefficients yielded here. They are expressed in the same units. The resulting values are *b'* and *c'*. It is necessary to do this addition because the blank corrected experimental data actually is a measure of the sample material's differential compressibility compared to that of mercury. One may imagine that a repeat of the blank run could be considered as a test of a some unit volume of mercury itself immersed in the surrounding mercury. The result should be the same as the blank run since in reality nothing has (at least on purpose) been changed. The blank corrected data consists of all zero volume changes with pressure and the *b* and *c* computed from it will likewise both be zero.

Also one should consider the situation which we have experienced wherein a less compressible material such as stainless steel is tested. Since both mercury and glass compress more than does the steel, the mercury column actually must rise in the bore of the penetrometer as the pressure is increased. This is interpreted as a negative intrusion volume change with pressure and leads to the computation of values for *b* (positive) and *c* which are physically impossible. Only when they are interpreted as values relative to mercury can they be valid and, by addition of mercury's coefficients respectively, they can be expressed as absolute values.

The values of *b'* and *c'* produced by this calculation will likely be in units of *absolute milliliters per milliliter * psia* and *absolute milliliters per milliliter * psia squared* if internal AutoPore computations are, as expected, performed in these units. Reporting these in alternate units of measure will be required. The most useful alternate units will be *milliliters per milliliter* megaPascal* and *milliliters per milliliter* kpsia* and analogous second order units. Strictly speaking, convention requires that the duplicated fundamental units of measure in the numerator and denominator be eliminated. This results in expressing the first order coefficient as *meters squared per Newton.* This choice also is provided in spite of its less intuitive impression.

FRACTAL DIMENSIONS

Pore space in sedimentary rocks exhibit fractal characteristics. The fractal dimension of these materials has been shown to be an important petrophysical parameter partly because capillary pressure and other transport coefficients scale as power laws of fluid saturation. The scaling exponents often relate to the fractal dimension of the medium. Angulo[1)] , et al, show that fractal dimensions of a quantity related to pore space bulk can be determined by mercury intrusion porosimetry.

According to percolation theory (see reference to Katz and Thompson in Permeability section of this appendix), at some threshold pressure PT, the invading fluid first spans the entire sample, that is, the fluid percolates for the first time. This then produces a geometrical configuration of fluid known as the percolation backbone and pressures from the point of percolation to completion of the backbone are in the *backbone formation* region. At greater pressures, filling of pore cavities behind smaller pore throats continues but without the sudden influx of fluid as observed at the threshold pressure. The backbone is a fractal with fractal dimension DH, but at higher pressures, the geometry of the fluid cluster changes rapidly to another fractal with fractal dimension DV (>DH) of the supporting media.

MIP DATA REDUCTION

In order to calculate a fractal dimension, the threshold pressure, Pthresh, must be known. The threshold pressure is the pressure at which the intrusion volume vs.pressure curve is steepest. This is either a calculated value (if chosen) or the value entered on the Material Properties dialog if the *Use entered threshold pressure* option is selected.. It is the same value used in permeability calculations.

If necessary, the value is calculated as follows. First set up an Akima spline for specific intrusion volume (Ii) vs. pressure (Pi) for all points on the first intrusion cycle. This is used to calculate the slope, (dI/dP)i, at each pressure. Use these values to set up another Akima spline for slopes vs. pressures. Finally, use the second Akima spline to find the value of pressure that gives the maximum slope. This is the threshold pressure, Pthresh. In addition, the user must specify the backbone formation and percolation pressure ranges over which calculations are to be performed.

The equation that defines fractal dimension is:

$$I = \alpha\left(P - p^{thresh}\right)^{(3-D)}$$

I	=	specific intrusion volume
P	=	pressure
P_{thresh}	=	threshold pressure

1) R.F. Angulo, V. Alvarado, and H. Gonzalez, “Fractal Dimensions from Mercury Instrusion Capillary Tests,” II LAPEC, Caracas, March 1992.

D	=	the fractal dimension
α	=	proportionality constant

This equation is transformed to the following to make it linear in the unknown parameters.

$$\log(I) = (d - D)\log(P - P_{thresh}) + \log\alpha$$

D and α are calculated by least squares fit to this equation, using all collected points (I_i, P_i) where P_i is in the user-selected range and above the threshold pressure.

MATERIAL PERMEABILITY

BACKGROUND

Permeability is a basic permeable medium property that, unlike porosity, cannot be defined apart from fluid flow.

Permeability is the proportionality " constant" between the fluid flow rate and an applied pressure or potential gradient.

Hydrologists, petrologists, and other branches of geology need to measure the intrinsic properties of rock and soils to both store and transmit fluid. These are porosity, permeability, the hydraulic conductivity of Darcy's law, and specific storage.

BASIS OF DATA REDUCTION METHOD TO BE USED

1. A.J. Katz and A.H.Thompson, Quantitative prediction of permeability in porous rock: Physical Review, Series B, Vol. 34, pp. 8179-8191 (1986).
2. A.J. Katz and A.H. Thompson, "Prediction of Rock Electrical Conductivity From Mercury Injection Measurements," *Journal of Geophysical Research, Vol. 92*, No. B1, pp. 599-607, (1987).
3. E.J. Garboczi, "Mercury Porosimetry and Effective Networks for Permeability Calculations in Porous Materials," NIST.
4. Kelli Murbach, "Permeability in Cement Impedance Spectroscopy," Case Western Reserve University.
5. P.J. Tumidajski and B. Lin, "On the Validity of the Katz-Thompson Equation for Permeabilities in Concrete", pp. 643-647.
6. A.H. Thompson, A.J. Katz, and C.E. Krohn, "The microgeometry and transport properties of sedimentary rock," *Advances in Physics*, Vol. 36, No. 5, pp. 625-694 (1987).
7. A.H.Thompson, S.W. Sinton, S.L. Huff, A.J. Katz, R.A. Raschke, and G.A.Gist, "Deuterium magnetic resonance and permeability in porous media," *Journal of Applied Physics, Vol. 65*, pp. 3259-3263 (1989).

THEORY

In their 1986 paper, Katz and Thompson introduced a model for absolute permeability, the key relationship being

$$k = c\iota_c^2 \sigma / \sigma_o$$

where k is absolute permeability in terms of the rock conductivity σ and a characteristic length l_c. The constant c is of the order of (1/226 = 0.00442), and σ_o is the conductivity of the brine in the pore space. The characteristic length is determined experimentally from the threshold pressure in a mercury injection experiment. The equation follows from the percolation arguments of Ambegaokar, Halperin, and Langer (1971) and pertaining specifically to electron transport in amorphous semiconductors, but which are generally applicable to systems characterized by a broad distribution of conductances.

THE KATZ-THOMPSON METHOD OF DATA REDUCTION USING MERCURY POROSIMETRY

In order to calculate the permeability, the characteristic length, L_{char}, must be determined. This is determined from the threshold pressure, P_{thresh}, using the Washburn equation. The threshold pressure is the pressure at which the intrusion volume vs. pressure curve is steepest. This is either a calculated value (if chosen) or the value entered on the Material Properties window (see *"Fractal Dimensions" on page C - 8*). The specific volume intruded at pores larger than L_{char}, I_{thresh} , is also used. This is calculated by interpolating the specific intrusion volume vs. pore diameter curve at L_{char}.

If a conductivity formation factor (s/so), is entered, the permeability is calculated as:

Perm	=	$CL^2_{char}\sigma/\sigma_o$
C	=	user-entered permeability constant
σ/σ_o	=	user-entered conductivity formation factor

If the conductivity formation factor was not entered, calculations proceed using the length at which the conductance is maximum, L_{max}. The conductance is maximum when $(I-I_{thresh})D^3$ is maximum, where I is specific intrusion volume and D is diameter. To find the diameter at which this is the case, an Akima spline is set up for ($I_i > I_{thresh}$). The spline is then used to find the value of the diameter, L_{max} , at which this curve is maximized (not necessarily a node point). From this, the fractional volume of connected pore space involving pore widths of size L_{max} and larger, S_{Lmax}, can be calculated by interpolating the specific intrusion volume vs. pore size curve to L_{max} and dividing by the total specific intrusion volume I_{tot}.

With this in hand, the permeability and conductivity formation factor can be calculated as:

$$Perm = \frac{1}{89} L_{\max} \quad \frac{2L_{\max}}{L_{char}} \cdot I_{tot} \cdot Y_b \cdot S_{L\max}$$

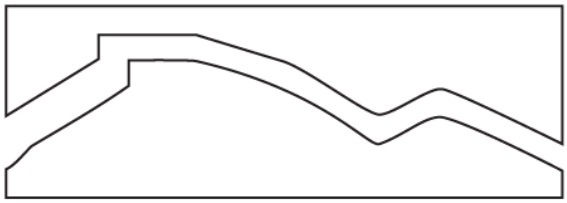
where

C	=	user-entered permeability constant
Y_b		bulk density, either calculated (if chosen) or user-entered on the *Material Properties* window

TORTUOSITY

The terms tortuosity and tortuosity factor are often used interchangeably. Tortuosity is the ratio of actual distance traveled between two points to the minimum distance between two points.

$$\xi = tortuosity = \frac{\text{Actual distance traveled}}{\text{shortest distance}} = \frac{\ell_e}{\ell} \tag{1}$$



Required parameters (units specified as mass, volume, length, and area):

ρ	=	density (mass/volume) – from pycnometry
V_{tot}	=	total pore volume (volume/mass)
K	=	permeability (area)

CALCULATING TORTUOSITY

The tortuosity can be calculated from the following expression:

$$\xi = \sqrt{\frac{\rho}{24K(1+\rho V_{tot})} \int_{\eta=r_{c,\min}}^{\eta=r_{c,\max}} \eta^2 f_v(\eta)\, d\eta} \tag{2}$$

where

$$-\int_v (r_c) = \frac{dV(r_c)}{dr_c}, \text{ from MIP} \tag{3}$$

In order to calculate the tortuosity, the weighted average pore size, D_{avg}, must be found. This is accomplished as:

$$D_{avg}^2 = Y_s = \left[\frac{1}{2} 1_1 O_i^2 + \Sigma I_i D_i^2 + \frac{1}{2} I_n O_n^2\right]$$

Y_s	=	skeletal or true density, either calculated (if chosen) or user-entered on the *Material Properties* window
D_i	=	pore diameter for the ith point
I_i	=	specific intrusion volume for the ith point

Given this value, the tortuosity is calculated as:

$$\xi = \sqrt{\frac{D_{avg}^2}{4 \cdot 24 Perm(1 - YI_{tot})}}$$

where

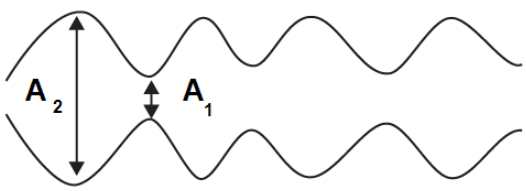
Perm	=	C L^2 char s/so
I_{tot}	=	total specific intrusion volume

CALCULATING TORTUOSITY FACTOR

Tortuosity factor is commonly used in the area of heterogeneous catalysis and is the ratio of tortuosity to constriction.

$$\tau = \frac{D_{eff}}{D\theta_c} \tag{4}$$

$$\tau = \frac{\xi}{\sigma} \tag{5}$$



$\beta = \frac{A_2}{B_2}$, area ratio

$\sigma = f(\beta)$, constriction factor

Carniglia has derived a simple expression for calculating the Tortuosity Factor of porous media. While this expression was derived using Fick's first law of diffusion and is convenient to calculate, the use of this correlation is severely limited by the data required to calculate the tortuosity factor.

V_{tot}	=	total pore volume
ρ_b	=	bulk density
S	=	total BET surface area
ΔV_i	=	change in pore volume within a pore size interval
d_i	=	average diameter within a pore size interval

For non-intersecting cylindrical pores the following simple correlation may be used:

$$\tau = 2.23 - 1.13 V_{tot} \rho_b$$

where

$$0.05 \le V_{tot} \rho_b \le 0.95$$

This correlation is limited to values of τ ranging from 1 to 2.2.

A generalized correlation has also been developed, however the generalized method requires diffusivity data for the system and conditions of interest (temperature and pressure). It is worth noting that if this diffusivity data is available, tortuosity factor can be calculated directly from equation 4.

$$\tau = \left(2.23 - 1.3 V_{tot} \rho_b\right)\left(0.92\left(\frac{4}{S}\Sigma\frac{V_i}{d_i}\right)^{1+\epsilon}\right)$$

where

ε	=	pore shape exponent, Carniglia has assigned a value of 1 for cylinders.

The tortuosity factor is calculated as:

$$TF = 0.92\left[\left(\Sigma\frac{\iota_i}{D_i}\right)\frac{4}{S}\right]$$

ΔI_i	=	difference in specific intrusion volume for two adjacent points $I_i - I_{i-1}$
$\overline{D}$	=	average pore size for the interval between adjacent points $0.5\ (D_i + D_{i-1})$
S	=	user-entered BET surface area
I_i	=	specific intrusion volume for the ith point
D_i	=	pore diameter for the ith point
I_{tot}	=	total specific intrusion volume
Y_b	=	envelope density, either calculated (if chosen) or user-entered on the *Material Properties* window

D PORE SURFACE AREA COMPUTATION

It is sometimes asserted that pore wall surface area computed on the basis of the work required to immerse a surface in mercury is superior to assuming the pores are cylindrical and calculating area from geometric relationships. What those who make the assertion fail to recognize is that mathematically and in practice, the two computations are identical as shown below.

WORK

The reversible work dW required to immerse an area dA of a non-wetting object in mercury[1] is

$$dW = \gamma \cos \Theta dA \tag{1}$$

where γ is the surface tension of mercury and θ its contact angle with the object. In the case of mercury and pores, this work is supplied when the external pressure P forces a volume of mercury dV into pores. Equation 1, therefore, becomes

$$\gamma \cos \Theta dA = -PdV \tag{2}$$

Assuming that γ and θ do not vary with pressure, equation 2 can be written

$$A = -\frac{\int PdV}{\gamma \cos \theta} \tag{3}$$

which, expressed for evaluation from pressure-volume mercury penetration data, becomes

$$\Sigma \quad A = -\frac{\Sigma P \quad V}{\gamma \cos \theta} \tag{4}$$

[1] Rootare, H.M. and Prenzlow, C.F., "Surface Areas from Mercury Porosimeter Measurements," J. Phys. Chem., 71, 2733-6 (1967).

CYLINDRICAL GEOMETRY

The basic relationship describing the penetration of mercury into a cylindrical pore of diameter D derived from equating the applied pressure to the resisting surface tension[1] is

$$PD = -4\gamma\cos\Theta \tag{5}$$

The relationship among wall area, diameter, and volume for a cylinder is

$$A = \frac{4V}{D} \tag{6}$$

Combining equations 5 and 6, yields

$$A = \frac{PV}{\gamma\cos\Theta} \tag{7}$$

which, as before, when written for evaluation from pressure-volume mercury penetration data, becomes

$$\Sigma \quad A = -\frac{\Sigma P \quad V}{\gamma\cos\theta} \tag{8}$$

[1] Washburn, E.W., "Note on a Method of Determining the Distribution of Pore Sizes in a Porous Material," Proc. Nat. Acad. Sci., 7, 115-6 (1921).

E PROPER HANDLING OF MERCURY

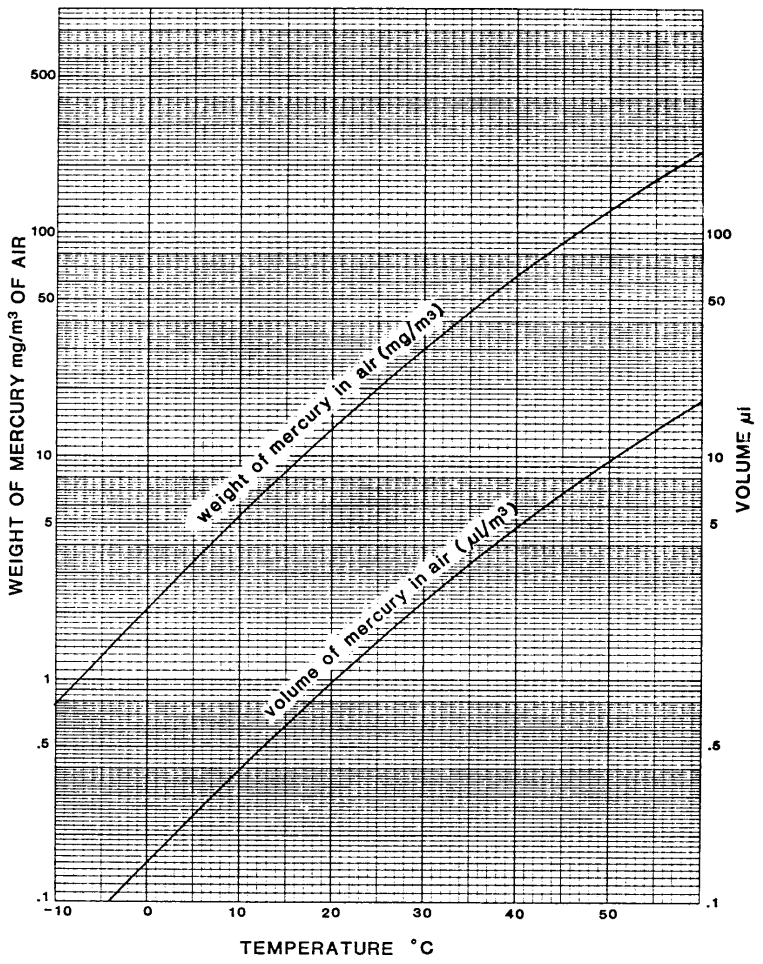
Because of its low melting point (-38.87 ºC), mercury (Hg) is slightly volatile at ordinary room temperatures and its vapor may pose a health hazard if allowed to accumulate in the work space. Although mercury can enter the body through the skin, lungs or digestive system, breathing air laden with high 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 ºC: $P = 1.20 \times 10^{-3}$ mmHg. As temperature increases from 20 to 40 ºC, 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 ºC 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 decontaminants[1)] 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. Handwashing 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.

Blank Page

F THEORY

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

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

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

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

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

Mercury extruding from pores upon reduction of pressure is in general accord with the above equation, but indicated pore diameters are always offset toward larger diameters. This results from equivalent volumes of mercury extruding at pressures lower than those at which the pores were intruded. It is also commonly observed that actual pores always trap mercury. The first phenomena is usually attributed to receding contact angles being less than advancing ones. The second is likely due to pore irregularities giving rise to enlarged chambers and “inkwell” structures. These phenomena give rise to hysteresis phenomena, i.e., distinct intrusion and extrusion P-V curves. See ***"Pore Surface Area Computation" on page D - 1*** for a discussion of surface area calculations.

Blank Page

G USE OF THE MAXIMUM INTRUSION VOLUME OPTION

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

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

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

Blank Page

H PYTHON MODULE - ADVANCED REPORTS

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

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

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

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

RUN A SCRIPT

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

EDIT A SCRIPT

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

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

REMOVE A SCRIPT

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

SUMMARY REPORT

This script produces a summary report with two summaries:

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

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


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

The result is:

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

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

GRAPHIC REPORT

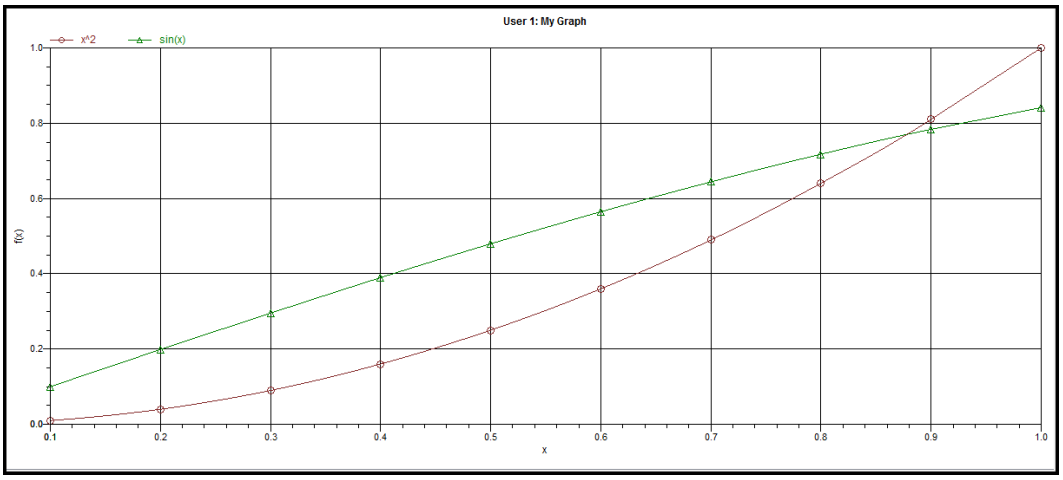
This script is an example of the mic module producing a graph with two curves:

```
import mic
import numpy as np

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

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

The results are:



ACQUIRE BASIC INFORMATION FOR MERCURY

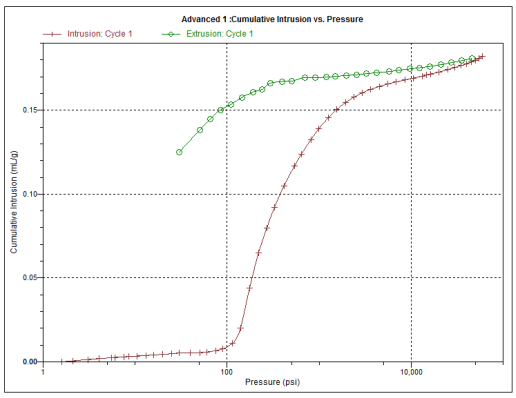
This script produces a graph of the intrusion and extrusion data, and a graph of the corresponding distribution of pores. It applies the mic module python calls mic.intrusion and mic.extrusion.

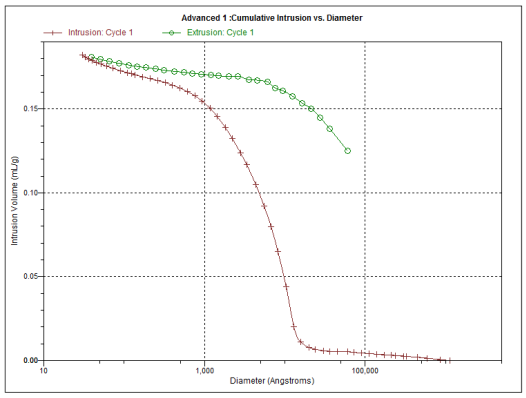
```
import mic
```

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

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

The results are:





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

- Sample Information
- Material Mercury Properties
- Intrusion Summary Results

```
import mic

mic.summary( "Summaries" )

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

```
      "%8.3f" % mic.sample_information("assembly mass"),
      "%8.3f" % mic.sample_information("penetrometer mass") ] )

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

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

```
        "%8.3f" % mic.report("hgsum", "quadratic compressibility coefficient")])
```

The results are:

Sample Information:

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

Material & Mercury Properties

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

Intrusion Summary Results

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

MIC MODULE PYTHON CALLS

TABLES

Create a New Tabular Report

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

Keyword arguments:

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

Add a Table

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

```
mic.table.addtable( name )

Keyword arguments:

   name --- the table name
```

Add a Column

This script adds a column to the last created table:

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

Keyword arguments:

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

SUMMARY REPORTS

Create a New Summary Report

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

Keyword arguments:

   title --- the summary title
```

Add a Summary Section

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

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

Keyword arguments:

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

Graphic Reports

Create a New Graphical Report

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

Keyword arguments:

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

Add a Curve

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

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

Keyword arguments:

   name      --- the curve name
   x         --- list of x values; must be a list of floats
                 (or convertible) and the same length as y
   y         --- list of y values; must be a list of floats
```

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

Add a Curve Using the Second Y-Axis

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

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

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

GET INTRUSION-EXTRUSION DATA

```
mic.intrusion( xaxis = 'pressure', cycle = 1 ) :

Keyword arguments:

   xaxis --- Specifies what the dependant variable will be
             'pressure' in units of psi (default)
```

```
              diameter' in units of Angstrom

   cycle --- Specifies the cycle number
             (default = 1)

Usage:

   p, v = mic.intrusion()
   p, v = mic.intrusion('pressure', 2)
   d, v = mic.intrusion('diameter')

   p    --- array of pressures (psi)
   d    --- array of diameters (Angstrom)
   v    --- array of intrusion volumes (ml/g)

mic.extrusion( xaxis = 'pressure', cycle = 1 ) :

Keyword arguments:

   xaxis --- Specifies what the dependant variable will be
             pressure' in units of psi (default)
             'diameter' in units of Angstrom

   cycle --- Specifies the cycle number
             (default = 1)

Usage:

   p, v = mic.intrusion()
   p, v = mic.intrusion('pressure', 2)

   d, v = mic.intrusion('diameter')
   p    --- array of pressures (psi)
   d    --- array of diameters (Angstrom)
   v    --- array of intrusion volumes (ml/g)
```

GET PROPERTIES OF SAMPLE MATERIAL

```
mic.material_properties( property='' , sample_number = 0 ) :

Keyword arguments:

   property      --- the property value to return.
                     If '' or None, then return all the properties
                     strings available for the sampe material.
                     Default value is None

   sample_number --- Identifier for the sample material to retrieve
```

```
                      0           : current sample file (default)
                      1 through 8 : corresponding overlay sample file

Usage:

   property_list  = mic.material_properties()
   material_name = mic.material_properties( 'material name')


In the above first usage case, the list of all available material
property keywords is returned.
```

GET MERCURY PROPERTIES

```
mic.mercury_properties( property='' , sample_number = 0 ) :

Keyword arguments:

   property  ---     the property value to return.
                     If '' or None, then return all the properties
                     strings available for the sample material.
                     Default value is None.


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

Usage:

   property_list   = mic.mercury_properties()
   mercury_density = mic.mercury_properties( 'density')

In the above first usage case, the list of all available mercury
property keywords is returned.
```

Blank Page

I WORKSHEETS

Worksheets in this section may be copied as needed.

ANALYSIS CONDITIONS WORKSHEET

File Name: _____

Analysis conditions identifier	
Low pressure equilibration	☐ Time ☐ Rate
Max. intrusion volume	
Evacuation	Initially evacuate at _____ Switch to medium at _____ Switch to fast at _____ Evacuation target _____ Continue evacuating for _____
High pressure equilibration	☐ Time ☐ Rate ☐ Pressure-controlled scan ☐ 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 ☐ **Blank** ☐ **Formula** ☐ **None**	If *Blank*, give file name:

PENETROMETER VOLUME CALIBRATION WORKSHEET

Calculate the penetrometer 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 Penetrometer 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 Penetrometer 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 Penetrometer 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 Volume of Penetrometer _____ ml

* See *Density of Mercury* table for values.

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

PRESSURE TABLE AND TABULAR DATA WORKSHEET

- For pressure table, enter:

  Filling pressure: _____
  Last low pressure point index: _____

- For tabular data, select:

  ☐ Pressure
  ☐ Pore size
  ☐ Percent intrusion

Entry	Entry	Entry	Entry

REPORT OPTIONS WORKSHEET

File name	
Calculation range ☐ **Pressure** ☐ **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 *(optional)*				
Penetrometer number *(optional - etched on penetrometer)*				
Sample mass(g). Enter this value on the *Sample Description* tab.				
Sample + penetrometer mass. Load the penetrometer with sample and weigh.				
Penetrometer mass. Subtract the *sample mass* from the *Sample + penetrometer* mass and enter the difference. • If using Basic presentation, enter this value on the *Sample Information* window. • If using Advanced presentation, enter this value on the *Penetrometer Properties* window.				
High pressure port number *(optional)*. Enter the number of the high pressure port where the sample is loaded.				
Assembly mass. The mass of the *sample + penetrometer + mercury*. (Weigh the penetrometer after the low pressure analysis.) Enter this value on the *Start High Pressure Analysis* window.				

Blank Page

J EXPORTED DATA FORMAT

```
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 µL/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 interstatial 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
```

K 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

L ERROR MESSAGES

If the *Action* response indicates to contact a Micromeritics service representative, record the error message, then make backup copies of any files involved in the operation.

2401 | FATAL ERROR.

Cause: An internal processing and / or hardware error has occurred during communication with the analyzer.
Action: Contact your Micromeritics service representative.

2430 | Error accessing file [*n*], error code = [*n*].

Cause A: Media may be damaged.
Action A: Clean the media drive. If this does not eliminate the problem, attempt operation using a backup copy of the file.
Cause B: Hard disk may be damaged.
Cause B: Contact your Micromeritics service representative.
Cause C: A software error occurred when the file was accessed.
Cause C: Contact your Micromeritics service representative.
Cause D: The file name specified contains one or more invalid characters.
Cause D: Enter a valid file name. Do not use characters such as * or ?. Refer to the operating system manual.

2431 | Error writing file [*n*], error code = [*n*].

Cause : Insufficient hard disk to perform the operation.
Action : Copy files not used regularly from the hard disk external media. Delete them from the hard disk, and then try the operation again.

2432 | Invalid response from MMI 'FILE_READ' request.

Cause: An internal processing and/or hardware error has occurred.
Action: Contact a Micromeritics service representative if this error message continues.

2433 | New entries have been found in this directory. Refresh the directory information?

Cause: Several analyzer files (sample information, analysis conditions, adsorptive properties, or report options) have been added to this directory by some function other than the analyzer program.
Action: Click **Yes** to update the directory information with data from each new file. This operation may take a minute.

Click **No** to locate the file manually. This option may be feasible if a large number of files have been copied into the directory and the file name is known.

2434 | File [*n*] — Subset [*n*] wrote wrong [*n*] of data, expected [*n*] bytes.

Cause: An internal processing and/or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2436 | Path specification [*n*] is invalid.

Cause: An invalid path name and / or extension was entered.
Action: Type a valid path name (including the proper extension), then press **Enter**.

2437 | File name [*n*] does not exist.

Cause: The entered file specification does not exist.
Action: Enter an existing file specification, or select a file name from the list box.

2437 | Overlay file [*n*] does not exist.

Cause: The entered file specification does not exist.
Action: Enter an existing file specification, or select a file name from the list box.

2438 | Cannot open scheduling diagnostic data save file: [*n*].

Cause: The file necessary for ***Unit [n] > Diagnostics > Schedule Diagnostic Test*** (*...\Hardware\scheddiagtests.dat*) could not be read. It is likely corrupted.
Action: Exit the application. Delete or rename the file mentioned in the error message and restart the software. A new file will be created.

2439 | Could not register file.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2440 | Subset not found.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2441 | Seek within file failed.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2442 | Bad header in subset file.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2443 | Subset owner denied access.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2444 | Not a valid file format.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2445 | Subset wrote the wrong amount of data.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2446 | Error reading data.

Cause: An unexpected error occurred when trying to access a data file.
Action: Contact your Micromeritics service representative.

2447 | Error writing data.

Cause: An unexpected error occurred when you tried to access a data file.
Action: Contact your Micromeritics service representative.

2448 | Basic Mode default parameter file directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.
Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default Adsorptive Properties directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.
Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default convert sample file directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.
Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default parameter file directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.
Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default report options directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default sample file directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | Default script test file directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | File directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the *Directories* list to move to the correct directory.

2448 | Problem diagnostic directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The export data file directory [*n*] cannot be used. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The library directory [*n*] cannot be used. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The library directory [*n*] does not exist. Please re-install to make use of Windows 7 libraries.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The reference file directory [*n*] cannot be used. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | The z-table file directory [*n*] cannot be used. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2448 | User python script directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

2449 | This field does not contain a valid file specification.

Cause: An invalid file name was entered.

Action: See the description of file naming conventions in a Windows manual, then re-enter the name.

2450 | Sample Defaults may not be edited while this operation is in progress. Do you wish to save and close the Sample Defaults edit session?

Cause: An automatic analysis (an analysis in which sample files are created using the defaults) was processing while editing the defaults.

Action: Finish the edit session of the defaults, close the window, then restart the automatic analysis.

2451 | The specified folder does not exist.

Cause: Informational message only indicating the system is looking for directory entries that cannot be found.

Action: Wait a few moments for the system to finish refreshing, then retry the operation again.

2452 | The instrument is busy performing an operation of which this application is unaware. Do you want to cancel?

Cause: The instrument is performing an operation which is not recognized by the application.

Action: Click **Yes** to have the application cancel the operation. Click **No** to allow the operation to continue.

2456 | Insufficient file handles available. Application cannot continue.

Cause: More than 50 files are open at the same time.

Action: Refer to an operating system manual then set the limit for open files to 50 or greater.

2458 | An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause: An attempt was made to exit the application while the instrument was performing a critical operation. This operation must be completed before the application can be exited.

Action: Wait a short time and attempt to exit the application again.

2459 | An instrument is busy. A delay in restarting this application could result in loss of new data. Continue with program Exit?

Cause: An attempt was made to exit the application while an analysis was in progress. While this is possible, the data collected when the application is inactive will not be permanently recorded until the application is re-started. A power failure to the instrument could cause some data to be lost.

Action: If not concerned with the potential for loss of data should a power failure occur, click **Yes** to continue; otherwise, click **No**.

2460 | Fatal Communications error on [*n*].

Cause: There was a fatal error in communication between the application and the software in the instrument. All displays for that instrument will be closed.

Action: Ensure that the analyzer is connected to the computer on the communications port configured in the *Setup* program. Stop and restart the analyzer software. Contact your Micromeritics service representative.

2461 | No instruments are in operation. This application will unconditionally terminate.

Cause: At least one instrument must be active for the application to operate. The initialization of all of the instruments configured with the Setup program has failed. The application stops.

Action A: Usually this message is preceded by another message giving the reason for the instrument's failure to initialize. Refer to the instructions for that message.

Action B: Ensure that the instrument is attached to the computer on the communications port

configured with the *Setup* program. Verify that the instrument's power switch is in the ON position and that the light on the front panel is illuminated. If the application continues to fail in its attempts to initialize the instrument, contact your Micromeritics service representative.

2477 | [*n*] did not properly initialize.

Cause: The software was unable to initialize this instrument. This is usually caused by one of the conditions listed in the previous error messages.

Action A: Run the *Setup* program and ensure that a valid port is specified; if not, specify a valid one when prompted.

Action B: Reinstall the software, then restart application.

Action C: Contact a Micromeritics service representative if this error message continues.

2478 | Error copying sequential data segment.

Cause: An internal processing and / or hardware error occurred while accessing a portion of a sample file.

Action: Confirm that the media being accessed does not contain errors.. Contact your Micromeritics service representative.

2479 | Cannot open [*n*] because it is a [*n*] file.

Cause: The file cannot be opened because it came from a different Micromeritics application

Action: Select a different file.

2480 | File [*n*] cannot be analyzed. It is currently being edited.

Cause: An attempt was made to start an analysis using a file that is open for editing.

Action: Finish editing the file, save and close it, then start the analysis.

2481 | Error accessing the sample information file [*n*].

Cause: An unexplained error prevented access to this file.

Action: The hard disk drive may be corrupt. Run diagnostics.

2482 | File cannot be opened for writing.

Cause: An attempt was made to open a file currently being used.

Action: Locate the application using the file (in the Micromeritics application, use the Windows menu item to get a list of all open windows, one of which may contain this file).

2483 | An analysis cannot be performed on [*n*]. It is open for editing and contains errors.

Cause: An attempt was made to use a sample file containing errors that is currently open.

Action: Go to the window containing the file, correct the errors, then save it.

2484 | The edit session for [*n*] must be saved before the analysis. Save changes and continue with the analysis?

Cause: An attempt was made to start an analysis using a file that contains unsaved changes and is open for editing.

Action: Click **Yes** to save the changes, then proceed with the analysis. Click **No** to cancel the analysis, then continue editing the Sample Information file.

2485 | The sample file [*n*] has an invalid status and cannot be used for this operation.

Cause: The selected file has a status other than *No Analysis*.

Action: Select a different sample file, or create a new one and use **Replace All** to copy parameters from the file originally selected.

2485 | The service test file has an invalid status and cannot be used for this analysis.

Cause: The selected file has a status other than *No Analysis*.

Action: Select a different service test file, or create a new one and use **Replace All** to copy parameters from the file originally selected.

2486 | Could not construct [*n*] report type. Program will terminate.

Cause A: Full rights to the application's folders and files is required.

Action A: Contact a system administrator to have full rights granted.

Cause B: An internal processing and / or hardware error has occurred.

Action B: Contact your Micromeritics service representative.

2487 | Could not start report generator. Error code [*n*]. Program will terminate.

Cause A: Full rights to the application's folders and files is required.

Action A: Contact a system administrator to have full rights granted.

Cause B: An internal processing and / or hardware error has occurred.

Action B: Contact your Micromeritics service representative.

2488 | File [*n*] cannot be opened for editing.

Cause: The specified file is being used in another edit operation.

Action: Check the Windows list to locate the other edit session.

2489 | File [*n*] cannot be opened for writing.

Cause: The specified file in a *Save As* operation is already open for edit.

Action: Select a different file for the *Save As* operation.

2490 | No '.INI' file present. Application will terminate.

Cause: The ASCII .INI file containing initialization information and system options information used during program startup does not exist.

Action: Run the analyzer *Setup* program (located on the applications CD), select *Change analyzer setup* and enter the pertinent information.

2491 | Highlighted fields contain errors. Please correct the errors before dialog box.

Cause: The highlighted fields contain invalid entries. The window cannot be closed until all errors are corrected.
Action: Check the entries, correct the errors, then close the window.

2492 | This field's entry is invalid.

Cause: The highlighted field contains an invalid entry.
Action: Check the entry and correct the error.

2493 | An entry is required for this field.

Cause: This field requires a valid entry to proceed.
Action: Enter or select an appropriate value.

2494 | Value is out of the valid range.

Cause: The entered value in the highlighted field is outside the valid range of values.
Action: Check the entry, then either enter or select an appropriate value.

2495 | Value is out of the valid range. Enter a value between [*n*] and [*n*].

Cause: The entered value in the highlighted field is outside the valid range of values.
Action: Check the entry, then either enter or select an appropriate value.

2496 | Invalid number.

Cause: An invalid number was entered in the highlighted field.
Action: Check the entry, then either enter or select a valid number.

2497 | This field contains an invalid character.

Cause: An invalid character was entered in the highlighted field.
Action: Check the entry, then enter valid characters.

2498 | The requested change to the Sample's status is invalid at this time.

Cause: A request to change the file's status, for example, from *automatically collected* to *manually entered* could not be done.
Action: Contact your Micromeritics service representative. Record the name of the sample file in which the problem occurred.

2499 | Sequence number must contain at least 3 digits.

Cause: An attempt was made to enter a sequence number that did not contain at least three

digits.

Action: Enter a sequence number that contains at least three digits.

2500 | All sample file names that can be created using the sequence number pattern already exist. You may want to modify the next sequence number.

Cause: No more sample information files can be created using the currently entered file name sequence number.

Action: Go to ***Options > Default Method***, then enter another sequence number.

2501 | System resources have reached a dangerously low level. Please close some windows to avoid the loss of data.

Cause: A large number of windows are open and consuming the system resources available to all applications.

Action: Close one or more windows. Contact your Micromeritics service representative.

2505 | Error logger cannot be initialized. Error code [*n*]. Program will exit.

Cause: An internal processing error has occurred.

Action: Contact your Micromeritics service representative.

2506 | Sample file [*n*] has a *No Analysis* status and cannot be used for this operation.

Cause: The selected sample file does not have collected data and cannot be used for operations, for example, reporting.

Action: Enter the name of a file with a status of *Complete*, *Analyzing*, or *Entered*. Alternatively, select a sample file from the list box.

2507 | The sample has an invalid status and cannot be used for degassing.

Cause: A sample file has been selected which does not have a *No Analysis* or *Prepared* status.

Action: Select a different file with a status of *No Analysis* or *Prepared*.

2508 | The selected file [*n*] does not contain a valid script.

Cause: The selected service test file does not contain any steps or the file has become corrupted.

Action: If the service test file has no steps, open the service test file and add steps to the file using the *Service Test Script* tab .

2513 | Unable to read the calibration file [*n*].

Cause: An invalid calibration file was selected or cannot be read.

Action: Ensure the media containing the calibration file has no problems.

2514 | Unable to write the calibration file [*n*].

Cause: An attempt to save calibration data has failed due to possible media problems.
Action A: Ensure the destination location has no problems.
Action B: Choose an alternate media to save the calibration data.

2515 | Warning: Changing the calibration information will affect the performance of the instrument. Only qualified service personnel should do this. Do you wish to proceed?

Cause: The process of performing a calibration operation was started.
Action: Calibration operations should only be done by or under the direction of qualified service personnel.

2516 | Warning: Keeping a backup copy of the calibration data is recommended by Micromeritics. Would you like to do so now?

Cause: A calibration operation was performed and a backup copy is recommended.
Action: Go to ***Unit [n] > Calibration > Save to File*** to perform a calibration save operation.

2517 | Canceling this dialog will reset the calibration state to what it was when this dialog was first opened. Are you sure you want to cancel?

Cause: The calibration has not been accepted.
Action: If the calibration operation was successful, click **Accept**.

2520 | No data points available for reporting.

Cause: The selected sample file does not have collected data and cannot be used for reporting.
Action: Select a different sample file.

2521 | Unable to program controller.

Cause: A hardware malfunction has occurred.
Action: Contact your Micromeritics service representative.

2522 | Invalid controller application file.

Cause: The application's control file has been corrupted or deleted.
Action: Reinstall the analysis program.

2523 | Programming controller failed.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2524 | CRC check failed on programming controller.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2525 | Unknown error programming controller.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2526 | Controller download was not successful.

Cause: An internal processing and / or hardware error has occurred.
Action A: Contact your Micromeritics service representative.

2527 | Controller CRC error on boot block.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2528 | Controller DRAM error.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2529 | Controller Com 1: error.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2530 | Controller Com 2: error.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2531 | Controller debug port error.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2532 | The instrument contains a different software version. Do you want to reset it?

Cause: The application has discovered a different version of software operating in the analyzer.
Action: If there are no analyzers other than the one connected to the computer, click **Yes**, then allow the updated software to load.

2533 | Analyzer initialization failed.

Cause: An internal processing and / or hardware error has occurred.
Action: Contact your Micromeritics service representative.

2534 | Unable to establish the TCP connection with [*n*].

Cause: There was a problem establishing communication with the analyzer.

Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection.

Ensure that no other Micromeritics application is initializing another instrument. If there is another Micromeritics application open and initializing an instrument, wait until the instrument initialization completes or is canceled.

Contact a Micromeritics service representative if this error message continues.

2548 | System status 1 [*n*].

Cause: There was a problem establishing communication with the analyzer.
Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2548 | System status 2 [*n*].

Cause: There was a problem establishing communication with the analyzer.
Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2549 | Error accessing online manual file [*n*].

Cause: The operator's manual file could not be located.
Action A: Reinstall the application.
Action B: Copy the contents of the manual folder from the setup CD to the application directory.

2550 | Attempts to acquire the instrument's status timed out.

Cause: There was a problem establishing communication with the analyzer.
Action: Ensure that the communications cable is seated firmly in the Ethernet slot at the analyzer connection and the computer connection. Contact your Micromeritics service representative.

2551 | Cannot access web page [*n*].

Cause: The Micromeritics web page for DFT models cannot be accessed. This could be caused by an ISP problem of high internet traffic.
Action: Try the operation later.

2552 | Configured serial number does not match instrument.

Cause: An instrument was substituted without properly changing the instrument serial number.
Action: Use the installation program to add or move devices as necessary.

2553 | Dialog ID [*n*] can not be created!

Cause: A required window could not be found by the software.
Action: Re-install the software.

2554 | File [*n*] does not contain any report data.

Cause: The selected sample file has no reports selected for printing.
Action: Edit the sample file and select reports to print in Report Options.

2556 | File [*n*] cannot be opened. It is currently selected for an analysis.

Cause: The sample file is currently selected and is undergoing a critical operation.
Action: Open the sample file after the critical operation has completed.

2557 | Directory [*n*] does not exist.

Cause: The directory entered in a file selector is not valid.
Action: Enter a valid directory.

2558 | The instrument is busy. The requested operation cannot be executed.

Cause: The instrument is analyzing and cannot be interrupted.
Action: Try the operation later.

2559 | SPC directory [*n*] does not exist.

Cause: A directory in one or more of the files selected in a SPC Report is invalid.
Action: Click **Samples** in the SPC report and verify the locations of all sample files.

2560 | File [*n*] cannot be created or opened. It has an unrecognized extension.

Cause: The extension specified in the file you are trying to create is not one which is recognized by the application.
Action: Change the extension of the file in the file name field of the file selector.

2560 | File [*n*] cannot be created. It has an unrecognized extension.

Cause: The extension specified in the file you are trying to create is not one which is recognized by the application.
Action: Change the extension of the file in the file name field of the file selector.

2563 | Cannot write. File or directory [*n*] read only.

Cause: The specified file name is marked as read-only
Action: Select a different file name.

2564 | Directory database [*n*] error [*n*] .

Cause: There is a problem creating the directory file used in file selectors.
Action: Verify the directory specified in this message is not marked read-only.

2570 | Instrument log database could not be opened: [*n*].

Cause: There is a problem opening the instrument log file [*SN.LOG*].

Action: Exit the software. Go to the \HARDWARE subdirectory of the instrument's program directory and rename the [SN.LOG] file where SN is the serial number of the instrument. A new log file will be created.

2571 | Instrument log database [*n*] error [*n*].

Cause: There is a problem opening the instrument log file [*SN.LOG*].

Action: Exit the software. Go to the \HARDWARE subdirectory of the instrument's program directory and rename the [SN.LOG] file where SN is the serial number of the instrument. A new log file will be created.

2572 | File [*n*] already selected for the analysis.

Cause: The file selected for analysis on one port has already been specified for another port.

Action: Choose a different sample file for each port.

2573 | A maximum of [*n*] files may be selected for analysis.

Cause: When using one file selector to select samples for all ports, the number of samples selected exceeds the number of ports on the instrument.

Action: Choose the proper number of samples or pick the sample for each port individually.

2573 | A maximum of [*n*] files may be selected.

Cause: When using one file selector to select samples for all ports, the number of samples selected exceeds the number of ports on the instrument.

Action: Choose the proper number of samples or pick the sample for each port individually.

2574 | No samples, or completed samples selected.

Cause: When using one file selector to select samples for all ports, either no samples were selected or a file with *Complete* status was selected.

Action: Select samples that do not have a *Complete* status.

2576 | The instrument [*n*] is not calibrated.

Cause: The analyzer application is in the process of initializing the instrument and is unable to locate the calibration files.

Action A: Click **OK**. Go to ***Unit [n] > Calibration > Load from File***, then select a file containing calibration data.

Action B: Click **OK**. Close the application, then use the *Setup* program to reinstall calibration files.

2577 | The python directory is missing or some of its contents have been removed.

Cause: When using Advanced reports, a necessary component is missing.

Action: Re-install the software.

2578 | Not enough available resources. Application will have reduced functionality. Reduce the number of open files or report windows.

Cause: There is not enough memory available to open the requested window.
Action: Close any unnecessary applications, then try the operation again.

2579 | The selected file has a status that is not compatible with the operation requested.

Cause: The selected sample file cannot be used for the selected operation. For example, a sample file with a *Complete* status cannot be used for analysis.
Action: Select a different sample file.

2580 | Problem encountered trying to load dbghelp.dll.

Cause: Files necessary for the application are missing or have been corrupted.
Action: Re-install the software.

2581 | Problem encountered trying to create the trace file.

Cause: Files necessary for the application are missing or have been corrupted.
Action: Re-install the software.

2582 | Bad MiniDumpWrite function found in dbghelp.dll.

Cause: Files necessary for the application are missing or have been corrupted.
Action: Re-install the software.

2583 | Error writing trace.

Cause: Files necessary for the application are missing or have been corrupted.
Action: Re-install the software.

2584 | The application encountered an unexpected error and will be halted.

Cause: Files necessary for the application are missing or have been corrupted.
Action: Re-install the software.

2585 | The following libraries are missing: [*n*]

Cause: This message is triggered on application start up if any of the library files used by an application, do not exist on disk.
Action: Add the library into the libraries.

2586 | Sample file [*n*] has no pressure table entries.

Cause: Trying to analyze a sample file with no pressure entries in *Analysis Conditions*.
Action: Either edit the *Analysis Conditions* and enter the pressure points to be used for analysis, or choose a sample file that already has the pressure points entered.

2588 | Sample file [*n*] is a [*n*] file and can not be analyzed on this instrument.

Cause: The specified sample file is from another Micromeritics application which is not usable with this application.
Action: Select a different sample file.

2589 | Sample file [*n*] can not be analyzed on this instrument.

Cause: The file specified for analysis cannot be used.
Action: Select a different file.

2590 | The default sample file [*n*] cannot be selected.

Cause: The default sample file (default method) cannot be used in this operation (e.g., as a sample file for analysis).
Action: Select a different sample file from the data directory.

2590 | The default sample file [*n*] can not be overwritten.

Cause: The default sample file (default method) cannot be used in this operation (e.g., as a sample file for analysis).
Action: Select a different sample file from the data directory.

2590 | An error occurred accessing file or directory [*n*].

Cause: The default sample file (default method) cannot be used in this operation (for example — as a sample file for analysis).
Action: Select a different sample file.

2591 | Cannot open problem diagnostic data save file: [*n*]

Cause: The file necessary for ***Unit [n] > Diagnostics > Schedule Diagnostic Test*** (*\Hardware\probdiag.dat*) could not be read. It is likely corrupted.
Action: Exit the application. Delete or rename the file mentioned in the error message and restart the software. A new file will be created.

2592 | The selected file has an extension that is not supported by this operation.

Cause: The selected file does not have a supported file extension.
Action: Open the adsorptive properties file. Open the FPI file selector and select another file with a supported file extension.

2593 | Warning: The selected file uses Unicode [*n*] or [*n*] encoding with non-ASCII characters.

Cause: The Advanced report is not saved in ASCII format, which is required by Python.
Action: Edit the file and save it in ASCII (ANSI) format.

2594 | The selected file uses Unicode [*n*] or [*n*] encoding and could not be read.

Cause: The Advanced report is not saved in ASCII format, which is required by Python.
Action: Edit the file and save it in ASCII (ANSI) format.

2595 | The selected file is too large (maximum allowed size is [*n*] MB).

Cause: The *Advanced* report is too large.
Action: Edit the file and reduce the size.

6000 | An instrument is performing a critical operation. Wait a few moments before exiting the application.

Cause: An attempt was made to exit the AutoPore application while the instrument is performing a critical operation. This operation must be completed before the application can be stopped.
Action: Wait a short time and attempt to stop the AutoPore application again.

6001 | An instrument is busy. Continue with program Exit?

Cause: An attempt was made to exit the AutoPore application while an analysis or calibration is in progress. While this is possible, the data collected while the AutoPore application is inactive will not be permanently recorded until the application is restarted. A power failure to the instrument could cause some data to be lost.
Action: If you are not concerned with the potential loss of data due to a power failure, click **Yes** to continue; otherwise click **No**.

6002 | No active instruments. Application will stop.

Cause: At least one instrument must be active for the AutoPore application to operate. The initialization of all of the instruments configured with the *Setup* program has failed. The AutoPore application must stop.
Action A: Usually this message is preceded by another message giving the reason for the instrument's failure to initialize. See that message's description.
Action B: Check that the instrument is attached to the computer on the communications port configured with the *Setup* program. If the AutoPore continues to fail in its attempts to initialize the instrument, contact your Micromeritics Service Representative.

6008 | File cannot be opened for writing. It is already in use.

Cause: You attempted a *Save As* operation to a file which is already in use. The save could not be completed.
Action: Wait until the selected file is no longer being used or select a new target name for the *Save As* operation.

6009 | Basic-Mode default parameter file directory [*n*] is invalid. Resetting to the installation directory.

Cause: A working directory specified in the .INI file is invalid, has been moved, or has been deleted.

Action: The installation directory will be substituted. The next time a file is opened, use the directories list to move to the correct directory.

6015 | No common pressure ranges were found in the file selected for averaging.

Cause: Samples selected for averaging do not have any cycles with common pressure ranges.
Action : Select samples that were analyzed with similar pressures.

6034 | Temperature was above warning level [*n*]. Current = [*n*], Max = [*n*], Warning Level = [*n*].

Cause: Temperature was above warning level.
Action: Shut down the instrument and vacate the area. Fumes may be present.

6034 | Current temperature is above warning level [*n*]. Current = [*n*], Max = [*n*], Warning Level = [*n*].

Cause: Temperature is above warning level.
Action: Shut down the instrument and vacate the area. Fumes may be present.

6048 | Analysis cannot proceed until caps are installed on the following low pressure ports [*n*].

Cause: The detected configuration does not match any known 96xx configuration.
Action: You may continue, which will use the 9620 configuration for the available ports, or you may call your Micromeritics service representative.

6060 | Instrument [*n*] is not calibrated.

Cause: The calibration was read, but is for a different instrument. It needs to be redone.
Action : If you have saved calibration files, go to ***Unit [n] > Calibration > Load From File*** to load a known, good calibration. Or go to ***Unit [n] > Calibration > Signals*** and ***Unit [n] > Calibration > Low Pressure Servo*** to calibrate the instrument. This should be done with caution or call your Micromeritics Service Representative.

6061 | Low pressure servo calibration failed.

Cause : Fewer than 2 reference points are available for the low pressure servo.
Action : Go to ***Unit [n] > Calibration > Low Pressure Servo*** to calibrate the instrument.

6064 | High pressure system is overpressurized.

Cause: The high pressure system was instructed to increase the pressure when it is already at too great a pressure.
Action: Reduce the pressure in the high pressure system. If this error was part of an automatic analysis, the pressure will be reduced automatically by returning the intensifier to the lower limit switch.

6065 | Intensifier reached upper limit switch.

Cause: During the course of a high pressure analysis, the intensifier reached its upper limit switch without achieving the desired pressure. The analysis cannot continue and was canceled. All data collected prior to this problem are stored in the sample file.

Action: Usually this error only occurs if the vent valve on the top of the pressure chamber was left open or did not seal properly. If this is not the case, it may indicate a problem with the equipment. Click **OK** to acknowledge the message.

6066 | Intensifier reached lower limit switch.

Cause: During the course of a high pressure analysis, the intensifier reached its lower limit switch without achieving the desired pressure. The analysis cannot continue and was canceled. All data collected prior to this problem are stored in the data file.

Action: Usually this error occurs if very low pressures (below 30 psia) are requested during extrusion. Because of the heat generated during pressurization, it may not be possible to de-pressurize the sample below 30 psia. If the problem occurs at reasonable extrusion pressures, the instrument may require calibration. Alternatively, the vent valve on the pressure vessel may not have been opened to atmosphere when the analysis was started. Click **OK** to acknowledge the message.

6067 | High pressure system pump has overheated.

Cause: The high pressure pump has been on too long at a high power, possibly due to extensive cycling at very high pressures.

Action: Allow the pump to cool and return it to the lower limit switch. Avoid allowing the pump to operate for extended periods at high power.

6068 | Low pressure analysis canceled. Mercury has overfilled.

Cause: During the execution of the low pressure analysis, the mercury reservoir overfilled. The analysis cannot continue and was canceled. The data already collected is stored in the sample file.

Action: Click **OK** to acknowledge the message. Consult your Micromeritics Service Representative to correct the overfill condition.

6069 | Low pressure analysis canceled. Mercury failed to drain.

Cause: During the execution of the low pressure analysis, the mercury did not drain. The analysis cannot continue and was canceled. The data already collected are stored in the sample file.

Action: Click **OK** to acknowledge the message. Consult your Micromeritics Service Representative.

6070 | Low pressure analysis canceled. No mercury available.

Cause: You attempted to start a low-pressure analysis, but there was not enough mercury to complete it.

Action: Add mercury to the system and try again.

6071 | Warning, the instrument is not calibrated.

Cause: An analysis is being attempted on a unit which has not been calibrated..

Action : If you have saved calibration files, go to ***Unit [n] > Calibration > Load From File*** to load a known, good calibration. Or go to ***Unit [n] > Calibration > Signals*** and ***Unit [n] > Calibration > Low Pressure Servo*** to calibrate the instrument. This should be done with caution or call your Micromeritics Service Representative.

6072 | Samples [*n*] and [*n*] do not have compatible analysis conditions.

Cause: The analysis conditions used on one port in a simultaneous analysis are incompatible with those used on another port.

Action: Edit the sample file and use compatible analysis conditions. Using the same selection in the analysis conditions drop-down list is an easy way to do this.

6073 | You must select sample files for both ports to do a differential analysis.

Cause: A high pressure differential analysis was selected, but only one port was used.

Action: A differential analysis requires a sample file for each port. Choose another sample file for the other port or change the current analysis to a standard one.

6074 | Warning these analysis conditions contain pressures that are below the fill pressure or too high for the high pressure system of this instrument. Do you want to proceed without the high pressures?

Cause: The analysis conditions selected contain pressure points higher than the instrument is capable of.

Action : Select a different sample file or continue with the currently selected file for a low pressure analysis or edit the Analysis Conditions file and remove the pressures that are too high.

6074 | These analysis conditions contain pressures that are out of range for the high pressure system of this instrument.

Cause: The analysis conditions selected contain pressure points higher than the instrument is capable of.

Action : Select a different sample file or continue with the currently selected file for a low pressure analysis or edit the Analysis Conditions file and remove the pressures that are too high.

6075 | Instrument [*n*]: The calibration is for a different type of instrument and has been reset.

Cause: The calibration was read, but is for a different instrument. It needs to be redone.

Action : If you have saved calibration files, go to ***Unit [n] > Calibration > Load From File*** to load a known, good calibration. Or go to ***Unit [n] > Calibration > Signals*** and ***Unit [n] > Calibration > Low Pressure Servo*** to calibrate the instrument. This should be done with caution or call your Micromeritics Service Representative.

6076 | Low pressure analysis canceled. Evacuation timeout.

Cause: The pressure did not drop to 7 psia in a reasonable amount of time during a low pressure evacuation.

Action: Ensure that the penetrometers are installed properly and that there is no leak in the system; then try again.

6077 | Low pressure analysis canceled. Capacitance detector removed while manifold was pressurized.

Cause: Analysis was canceled because a critical component was removed while the analysis was in progress.

Action: Ensure all components are in place then start a new analysis.

6078 | Low pressure analysis suspended. Install penetrometers or rods and capacitance detectors on all low pressure ports before resuming.

Cause: Low pressure analysis is suspended because some low pressure ports do not have the necessary components installed in order to start.

Action: Ensure that penetrometers or rods, and capacitance detectors are installed on all ports then resume the analysis.

6079 | A hardware interface error has occurred. Operation canceled.

Cause: A malfunction has occurred in the instrument electronics.

Action: Contact your Micromeritics service representative.

6080 | Low pressure manifold outgas rate measured (interval: [*n*}, rate: [*n*].4f mmHg/min)

Cause: The low pressure manifold outgas rate has been measured and the result has been recorded in the instrument log.

Action: No action is required.

6081 | Low pressure manifold leak rate measured rate: [*n*] mmHg/min

Cause: The low pressure manifold leak rate has been measured and the result has been recorded in the instrument log.

Action: No action is required.

6082 | Low pressure analysis canceled. Mercury reservoir failed to evacuate.

Cause: The mercury reservoir could not be evacuated as required for draining mercury.

Action: Ensure the cap is properly installed on the mercury filling port. Contact a Micromeritics service representative if this error message continues.

6083 | The number of pressure table points [*n*] has exceeded the maximum of [*n*]. Reduce the number of points specified in the analysis conditions pressure table.

Cause: More than 2500 pressure table points have been selected for analysis.

Action: Edit the analysis conditions of the sample file and reduce the number of pressure table points.

6084 | Duplicate penetrometer selections found. Unable to save volumes for penetrometers on the following ports: [*n*]

Cause: The same *Penetrometer Properties* file is used for more than one port in the analysis.

Action: Use different *Penetrometer Properties* files in the samples to be used for analysis.

6085 | Unable to save volumes for penetrometers on the following ports: [*n*]. Discard volume measurements for those penetrometers?

Cause: There was a problem reading the sample file on the ports specified in the message.

Action: Use new sample files on the ports identified.

6086 | The instrument is not calibrated. Continue?

Cause: Trying to save a calibration file for an instrument that is not calibrated.

Action : If you have saved calibration files, go to ***Unit [n] > Calibration > Load From File*** to load a known, good calibration. Or go to ***Unit [n] > Calibration > Signals*** and ***Unit [n] > Calibration > Low Pressure Servo*** to calibrate the instrument. This should be done with caution or call your Micromeritics Service Representative.

6087 | Analysis cannot proceed until caps are installed on the following low pressure ports: [*n*].

Cause: For a low pressure analysis, at least one of the low pressure caps has been left off of one of the selected ports.

Action: Install low pressure caps on all selected ports.

Blank Page

INDEX

A

B

C

D

E

F

G

H

I

K

L

M

S

T

U

V

W