AutoPore® V Series

Mercury Intrusion Porosimeter

Operator Manual

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(Rev E)

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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.

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

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CORPORATE PROFILE

Micromeritics Instrument Corporation is the world's leading supplier of high-performance systems to characterize particles, powders and porous materials with a focus on physical properties, chemical activity, and flow properties. Our technology portfolio includes: pycnometry, adsorption, dynamic chemisorption, particle size, intrusion porosimetry, powder rheology, and activity testing of catalysts. The company has R&D and manufacturing sites in the USA, UK, and Spain, and direct sales and service operations throughout the Americas, Europe, and Asia. Micromeritics systems are the instruments-of-choice in more than 10,000 laboratories of the world's most innovative companies and prestigious government and academic institutions. Our world-class scientists and responsive support teams enable customer success by applying Micromeritics technology to the most demanding applications. For more information, please visit www.Micromeritics.com.

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About this Manual

The following can be found on the Micromeritics web page (www.Micromeritics.com).

- Calculations document (PDF)
- Error Messages document (PDF)
- Operator Manual (PDF)
- Parts and Accessories
- Vacuum Pump Guide (PDF)

The following symbols or icons indicate safety precautions and/or supplemental information and may appear in this manual:

NOTE — Notes contain important information applicable to the topic.

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

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

General Safety

Do not modify this instrument without the authorization of Micromeritics Service Personnel.

The AutoPore uses mercury which is an extremely toxic substance. Read the Material Safety Data Sheet (MSDS) and be aware of the hazards of mercury and know what to do in the event of a spill or an exposure incident.

Any piece of laboratory equipment can become dangerous to personnel when improperly operated or poorly maintained. All employees operating and maintaining Micromeritics instruments should be familiar with its operation and should be thoroughly trained and instructed on safety.

- Read the operator manual for any special operational instructions for the instrument.
- Know how the instrument functions and understand the operating processes.

- Wear the appropriate personal protective equipment when operating this instrument — such as eye protection, lab coat, protective gloves, etc.
- When lifting or relocating the instrument, use proper lifting and transporting devices for heavy instruments. Ensure that sufficient personnel are available to assist in moving the instrument. The AutoPore V 9600 weighs approximately 227 - 250 kg (500 - 550 lb) depending on configuration.
- Always pay attention to the safety instructions provided on each label affixed to the instrument and do not alter or remove the labels. When inspecting the instrument, ensure that the safety labels have not become worn or damaged.
- Proper maintenance is critical to personnel safety and smooth instrument operation and performance. Instruments require regular maintenance to help promote safety, provide an optimum end test result, and to prevent costly down time. Failure to practice proper maintenance procedures can lead to unsafe conditions and shorten the life of the instrument.
- Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage to the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

INTENDED USE

The AutoPore V Series 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 you to calculate 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 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.

The instrument is intended to be operated by trained personnel familiar with the proper operation of the equipment recommended by the manufacturer and as well as relevant hazards involved and prevention methods. Other than what is described in this manual, all use is seen as unintended use and can cause a safety hazard.

The instrument is intended to be used as per applicable local and national regulations.

TRAINING

It is the customer's responsibility to ensure that all personnel operating or maintaining the equipment participate in training and instruction sessions. All personnel operating, inspecting, servicing, or cleaning this instrument must be properly trained in operation and machine safety before operating this instrument.

ENVIRONMENTALLY FRIENDLY USE PERIOD

Hazardous Substances Table

Part Name	Hazardous Substances					
	Lead (Pb)	Mercury (Hg)	Cadmium (Cd)	Hexavalent Chromium (Cr (VI))	Polybrominated biphenyls (PBB)	Polybrominated diphenyl ethers (PBDE)
Metal Parts	o	o	o	o	o	o
Power Entry	o	o	o	o	o	o
Thermo-couple Cables	x	o	o	o	o	o
Electronic Components	x	o	o	o	o	o
Gas Fittings, Hoses and Filter	x	o	o	o	o	o

o Hazardous substance is below the specified limits as described in SJ/T11363-2006.

x Hazardous substance is above the specified limits as described in SJ/T11363-2006.

The Environmentally Friendly Use Period (EFUP) for all enclosed products and their parts are per the symbol shown here unless otherwise marked. Certain parts may have a different EFUP (for example, battery modules) and are marked to reflect such. The Environmentally Friendly Use Period is valid only when the product is operated under the conditions defined in the product manual.

SYMBOLS THAT MAY APPEAR ON YOUR INSTRUMENT

The following symbols or icons indicate safety precautions and/or supplemental information and may appear on your instrument:

Use extreme caution when working on the instrument where one of these symbols may be displayed. These symbols indicate the part may be hot and cause serious burns.

Use the cotton gloves provided in the accessory when handling heated surfaces. These cotton gloves are not intended to protect hands when heated surfaces are above 60 °C.

When working on the instrument where this symbol is displayed, refer to your Micromeritics' instruction manual for additional information.

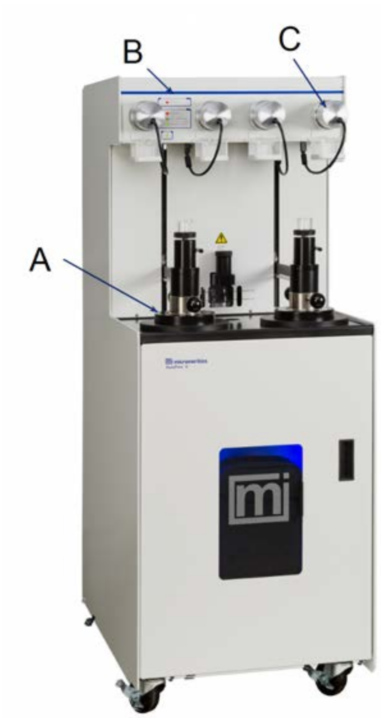
When this symbol is displayed, toxic or flammable gases require proper venting of exhaust.

This symbol can also indicate the instrument uses mercury which is an extremely toxic substance. Read the Material Safety Data Sheet (MSDS) and be aware of the hazards of mercury and know what to do in the event of a spill or an exposure incident mi micromeritics®

Table of Contents

1 Analyzer Components for AutoPore

Front Panel

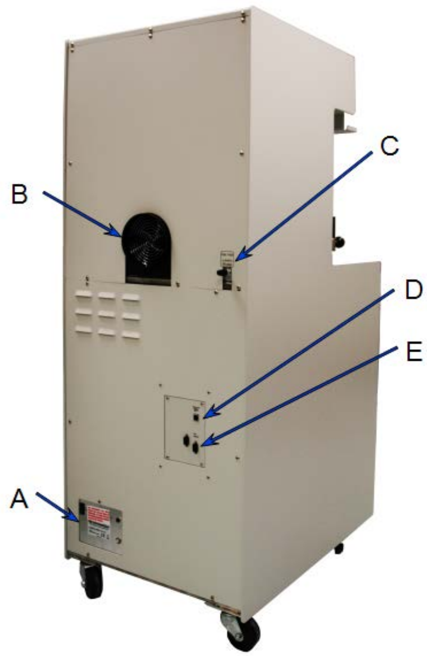


A. High pressure ports (2)
B. LED display
C. Low pressure ports (4)

Front Panel Components

Component	Description
High Pressure Ports [*LED light*]	**Pressurized.** Illuminates when the high pressure system is pressurized, when the computer system is not connected, or when the computer is connected but the application is not running.
Low Pressure Ports [*LED light*]	**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 [*LED light*]	Illuminates when power is supplied to the analyzer.

Rear Panel



A. Power
B. Ventilation
C. External gas supply port
D. Ethernet port
E. Aux RS-232 port

Rear Panel Components

Component	Description
Power	Power cord connector, power supply, and power switch. Reset buttons: ■ **10 Amp.** Resets the internal vacuum pump. ■ **25 Amp.** Resets the internal vacuum pump in a high pressure analyzer.
Ventilation	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.
External Gas Supply	Used for connecting an external gas supply to the analyzer when generating pressures in the low pressure system.
Ethernet Port	Port for a shielded Ethernet cable allowing communication between the analyzer and the computer.
Aux RS-232 port	Used by service personnel.

MERCURY PRECAUTIONS

Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

At all times practice strict chemical hygiene when working with mercury:

- Read the MSDS for mercury hazards
- Store mercury in a cool location to minimize mercury vaporization
- Instruments should be installed in a cool location
- Wear safety glasses, protective gloves, and appropriate clothing (long lab coat, closed toe shoes)
- Avoid skin and eye contact.
- Wash hands and face after handling mercury
- Do not eat or drink in the lab where mercury is present
- Dispose of contaminated waste in an appropriate manner

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 analyzer 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.

THE BASIC WORKFLOW

Before a sample can be analyzed, it must be:

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

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

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

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

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

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

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

Theory for Mercury Porosimetry on page E - 1 provides a thorough discussion of the theory of porosimetry.

SPECIFICATIONS FOR AUTOPORE V

Electrical

Frequency	50 to 60 Hz
Power	500 VA maximum
Voltage	100/120/220/240V~ ±10% 220V applications should use the 230V setting.
Overvoltage category	II

Gas

Nitrogen or other clean, dry gas at 50 psig (345 kPa)

Environment

Temperature	10 °C to 35 °C (50 °F to 95 °F), operating 0 °C to 50 °C (32 °F to 122 °F), non-operating Maximum rate of change of 2 °C per hour
Humidity	10% to 80% relative, non-condensing
Indoor or Outdoor use	Indoor only (not suitable for wet locations) Altitude: 2000 m max (6500 ft) Pollution degree of the intended environment: 2
Location	Instrument should be located in a dust-free, vibration free environment, away from exposure to direct sunlight and direct air drafts.
Degree of Ingress Protection	IPXO

High Pressure Model 9600 and 9605

Measurement	From atmospheric pressure to 33,000 psia (228 MPa)
Pore Diameter Range	6 to 0.005 µm
Resolution	0.165 psi (1400 Pa) from 3300 psia (21 MPa) to 33,000 psia
Servo Control Precision	0.05% of target, min 5 psia, no overshoot
Transducer Accuracy	±0.1% of full scale (transducer manufacturer's specifications)
Transducer Hysteresis	0.05% of full scale

High Pressure Model 9610 and 9620

Measurement	From atmospheric pressure to 60,000 psia (414 MPa)
Pore Diameter Range	6 to 0.003 µm
Resolution	0.3 psia from atmospheric pressure to 60,000 psia
Servo Control Precision	0.05% of target, min 5 psia, no overshoot
Transducer Accuracy	± 0.1% of full scale (transducer manufacturer's specifications)
Transducer Hysteresis	0.05% of full scale

Intrusion

Accuracy	±0.1% of maximum penetrometer stem volume
Resolution	Better than 0.1 µL

Low Pressure

Measurement	0.2 to 50 psia (345 kPa)
Pore Diameter Range	500 to 3.6 µm
Resolution	0.00025 psi
Servo Control Precision	1% of target, min. 0.05 psia, no overshoot
Transducer Accuracy	±0.1% of full scale (transducer manufacturer’s specifications)

Penetrometers

Sample Size	Maximum: a cylinder 2.54 cm diameter × 2.54 cm long (1 in. diameter × 1 in. long)
Stem Intrusion Volumes	0.38, 1.1, 1.7, 3.1, and 3.9 mL

Physical

Height	143 cm (56.25 in.)
Width	54.3 cm (21.38 in.)
Depth	78 cm (30.75 in.)
Weight (9600 and 9610)	227 kg (500 lb)
Weight (9605 and 9620)	250 kg (550 lb)

Computer Requirements

Operating System	Windows 10 or higher operating system is required.
Desktop Installation Required	The application should not be installed on a network drive with shared access. Multiple users cannot operate the application at the same time. Ensure the "Sleep" setting on the desktop is set to "Never" to avoid interruption while running an analysis. If this occurs, the application loses network connectivity with the instrument and a communications error will be reported. A restart of the Windows application may be required if automatic reconnection is not successful.
10 Base T or 100 Base T Ethernet Port	If the computer is to be connected to a network, two Ethernet ports are required. If more than one Ethernet-based unit is connected to the same computer, an Ethernet switch will also be required.
Read/Write Permissions	All application users will need Read/Write permission to all directories and subdirectories where the application is installed.
Drives	USB port

Due to continuous improvements, specifications are subject to change without notice.

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2 About the Software

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

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

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

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

Data can be manipulated and displayed interactively using MicroActive reports.

Menu Structure

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

Main Menu Bar Options

Selections	Description
File	Use to manage files used by the application — such as sample files, analysis conditions files, report options files, etc.
Unit [*n*]	Use to perform analyses, calibrations, and other analyzer operations. *Unit [n]* displays on the menu bar for each analyzer attached to the computer.
Reports	Use to start or initiate reports and view the results.
Options	Use to change presentation options, set the method and active metals defaults, configure signal calibration, manage libraries, select units, and create report styles.
Window	Use to manage open windows and display a list of open windows. A checkmark appears to the left of the active window.
Help	Use to access the embedded operator manual, Micromeritics web page, and information about the application.

COMMON FIELDS AND BUTTONS

The fields and buttons in the following table are located in multiple windows throughout the analyzer application and have the same description or function. Fields and button descriptions not listed in this table are found in tables in their respective sections. All entry fields will accept information when using a bar code reader.

Common Fields and Buttons

Selections	Description
Add	Adds an item to the list.
Add Log Entry	Use to enter information that will display in the sample log report that cannot be recorded automatically through the application. Click the button again to enter multiple log entries.
Append	Use to insert one row at the end of a table.
Autoscale	When enabled on report parameters windows, allows the x- and y-axes to be scaled automatically. *Autoscale* means that the x- and y- ranges will be set to show all the data. If *Autoscale* is not selected, the entered range is used.
Axis Range	On report parameters windows, the *From* / *To* fields are enabled when *Autoscale* options are not selected. Enter the starting and ending values for the x- and/or y-axes.
Bar Code *(default field label name)*	Use to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Browse	Searches for a file.
Cancel	Discards any changes or cancels the current process.
Clear	Use to clear the table entries and display only one default value.
Close	Closes the active window and displays a prompt to either accept or reject changes.
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 to display in the report header about the sample or analysis.
Copies	Selects the number of copies to print. This field is only enabled when *Print* is selected.
Delete	When working with tables, deletes the selected information.
Destination	Selects the report destination.

Common Fields and Buttons (continued)

Selections	Description
Edit	When working with report parameters, highlight the item in the *Selected Reports* list box and click **Edit** to modify the report details.
Exit	Exits the application. If a file is open with unsaved changes, a prompt displays the option to save the changes and exit or exit the application without saving the changes. If an analyzer is currently operating, an additional prompt displays to confirm exiting from the software.
Export	Exports data in a sample file as a .TXT, .XML or .XLS file. When saved to a file, the data can be imported into other applications.
File	Selects the destination directory. Enter a new file name in the *File name* field or accept the default. Select to save the file as a spreadsheet (.XLS), a portable document format (.PDF), or an ASCII text (.TXT) file format.
File name	Selects a file name from the list shown or enter a file name. If the required file type is not shown, select the type of file from the list.
From / To	Indicates the *From* and *To* range for x- and/or y-axes when working with report parameters windows.
Insert	Inserts one row above the selected row in the table.
List	Creates a list of samples or other types of files. The list will contain the file name, date/time the file was created or last edited, file identification, and file status.
Name	Contains a list of files in the selected directory or library.
Next	Moves 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	Moves 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	Removes the selected file or files from the list.
Replace	Selects another file where the values will replace the current file's values.

Common Fields and Buttons (continued)

Selections	Description
Replace All	Selects another .SMP file where the values will replace all values for the active sample file. The original file will remain unchanged. No analysis data is added to the file. The only information added is sample information, material properties, liquid properties, analysis, and reporting parameters.
Report	Displays a window to specify report output options.
Save	Saves changes.
Save As	Saves a file in the active window under a different file name. A portion can be saved as a separate, stand-alone file, such as Analysis Conditions or Report Options, when saving sample information.
Start	Starts the report, test, analysis, or operation.
Start Date	Displays a calendar to select the start date for the report.
View	**Operation**. Displays the data from the current analysis. **Instrument Log**. Displays recent analyses, calibrations, errors, or messages. Enabled only in Service Test Mode. **Instrument Schematic**. Displays a schematic of the analyzer system.

FILE STATUS

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

File Status

File Status	Description
Analyzing	Sample files that are currently used for analysis.
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 files that have not been used to perform an analysis.

File Type and File Name Extension

File Type	File Name Extension
Analysis Conditions	.ANC
Methods	.MTH
Penetrometer	.PEN
Report Options	.RPO
Sample Information	.SMP

File Types for Printing or Exporting

File Type	File Name Extension
Portable document format	.PDF
Report	.REP
Spreadsheet	.XLS
Unicode	.TXT
Extensible markup language	.XML

KEYBOARD SHORTCUTS

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

Certain menus or functions can also be accessed using the **Alt** key plus the underlined letter in the menu command. For example, to access the *File* menu, press **Alt + F**, then press the underlined letter on the submenu (such as pressing **Alt + F**) then pressing **O** to open the *File Selector*).

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

Keyboard Shortcuts

Selections	Description
Alt +*[Unit n]*	Opens the *Unit [n]* 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.
Ctrl + N	Opens a new sample file.
Ctrl + O	Opens the *File Selector* window.
Ctrl + P	Opens the *File Selector* to start a report from a selected .SMP file.
Ctrl + S	Saves the open file.
F1	Opens the online help operator manual.
F2	Opens the *File Selector* window.
F3	When in the *File Selector* window, opens the file search box.
F4	When in the *File Selector* window, opens the address bar.
F6	Cascades open windows.
F7	Tiles all open application windows.
F8	Opens the *File Selector* to start a report from a selected .SMP file.
F9	Closes all open reports.
Shift + F9	Opens the shortcut menu of either the selected component on the analyzer schematic when manual control is enabled or the onscreen reports.

Option Presentation

Options > Option Presentation

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

Option Presentation Display

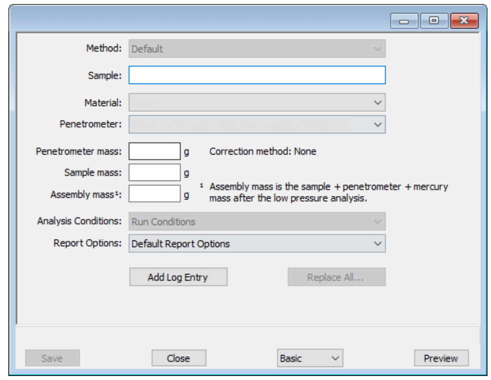
Presentation Display	Description
Advanced	Displays all parts of sample and parameter files. Navigate to parameter windows by selecting the tabs across the top of the window.
Basic	Displays sample information in a single window. This display option is used after the parameter files have been created. The previously entered or default parameter files are then accessible using drop-down lists.
Restricted	Displays the sample file in a single window like 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 — such as the pharmaceutical industry — where analysis conditions must remain constant. The *Advanced* option is not available in the view selector at the bottom of the window when using the *Restricted* display option.
Show Splash Screen	Enables (or disables) the splash screen upon application startup.

To change the view for the selected window, use the drop-down list at the bottom of the sample file editor.

The following examples show the same sample file in *Advanced* and *Basic* display. *Basic* and *Restricted* displays will look the same. A password is required if using *Restricted* format.

Option Presentation Examples

Advanced view



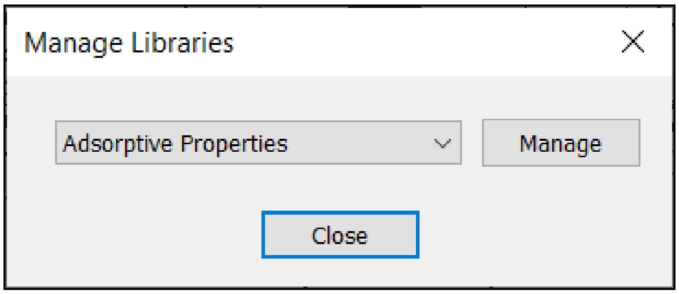
Basic or Restricted view

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

LIBRARIES

Options > Manage Libraries

This feature is not available when using *Restricted* option presentation.



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

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

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

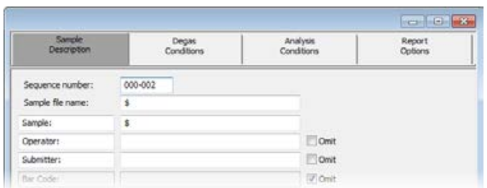
When *removing* a folder from a library, the folder and its contents are not deleted from the original file storage location. However, when *deleting* files or folders from within a library, they are deleted from their original file storage location. Deleted files and folders can be recovered from the Recycle Bin located on the Windows desktop.

METHODS

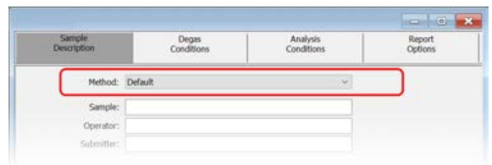
File > New Method
Options > Default Method
File > Open > [.MTH File]

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

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



Default Method



Sample Information file

Default Methods

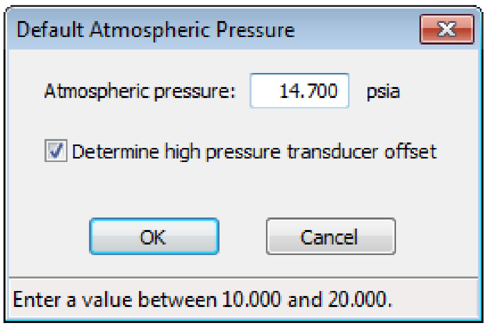
Selections	Description
Sample file name [*text box*]	Enter a format for the sample identification. The entry in this field becomes a part of the saved sample file name. Include the $ symbol to have the sample file number included as part of the identification.
Sample **Operator** **Submitter** **Bar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

Configure the Analyzer

Atmospheric Pressure

Options > Atmospheric Pressure

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



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

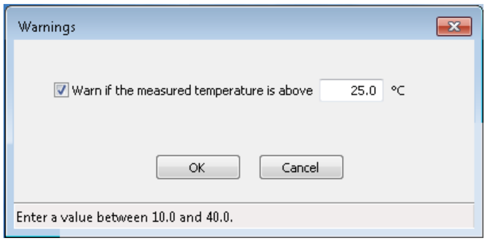
Select *Determine high pressure transducer offset* to enable the system to determine automatically the high pressure transducer offset.

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

MEASURED TEMPERATURE WARNING

Options > Warnings

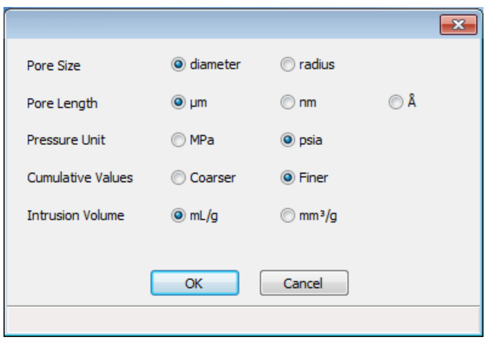
Enable this option to have the analyzer automatically display a warning if the measured temperature rises above the entered temperature.



UNIT SELECTION

Options > Units

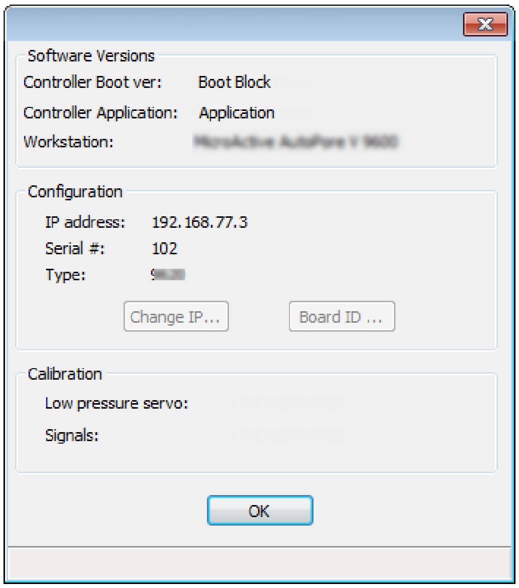
Use to specify how data should appear on the application windows and reports. This menu option is not available if using *Restricted* option presentation in a standard installation environment.



Unit Configuration

Unit [n] > Unit Configuration

Use to display and confirm hardware and software configurations and calibrations of the analyzer.



Unit Configuration

Selections	Description
Calibration [*group box*]	Displays calibration information for analyzer components.
Configuration [*group box*]	Displays the IP address used by the analysis program, serial number, and type of analyzer. **IP address.** Displays the IP address of the analyzer. **Change IP.** Displays the Board ID dialog, which describes the circuit boards in the analyzer. Use the Board drop-down list to select a board to view. **Board ID.** Click to display information from the circuit boards in the analyzer. Use the drop-down list to select a board to view. The parameters shown cannot be edited.
Software Versions [*group box*]	Displays the software versions of the controller start up code, analysis program, and type of analyzer.

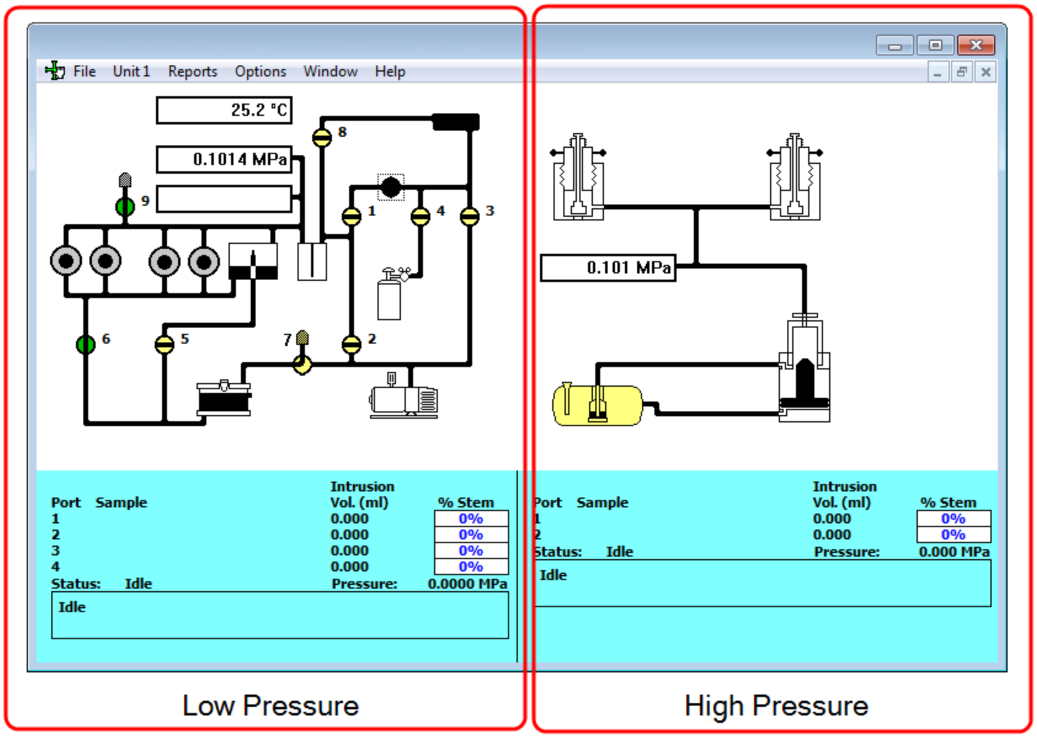
For fields and buttons not listed in this table; see ***Common Fields and Buttons on page 2 - 2*****.**

INSTRUMENT STATUS

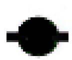
SHOW INSTRUMENT SCHEMATIC

Unit [n] > Show Instrument Schematic

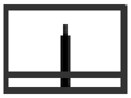
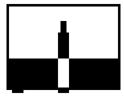
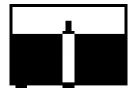
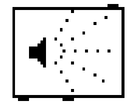
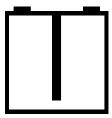
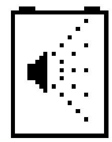
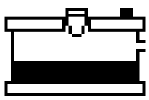
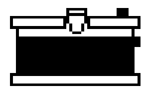
To operate the valves and elevator from the schematic, manual control must be enabled (***Unit [n] > Enable Manual Control***).



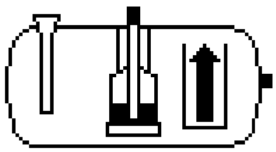
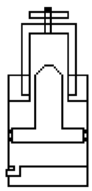
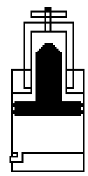
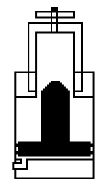
Analyzer Schematic Icons

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

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 - 16*.		 Level is OK.	
Hg Reservoir Vacuum Switch			
		When illuminated, indicates that mercury reservoir has been evacuated.	
Low Pressure Ports		Indicates that the mercury fill sensor detects mercury.	
		Indicates a rod and capacitance detector are present (detector reading is less than -5 pF).	
		Indicates there is no mercury, but the capacitance detector is on with either a rod or a penetrometer.	
		Indicates there is no capacitance detector and penetrometer or rod in place.	

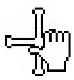
High Pressure Schematic Icons

<table>
<tr><th colspan="3">Hydraulic Pump</th></tr>
<tr><td></td><td colspan="2">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.</td></tr>
<tr><td colspan="3">Intensifier. Displays the state of the intensifier limit switches.</td></tr>
<tr><td>
Midway</td><td>
Top</td><td>
Bottom</td></tr>
</table>

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.

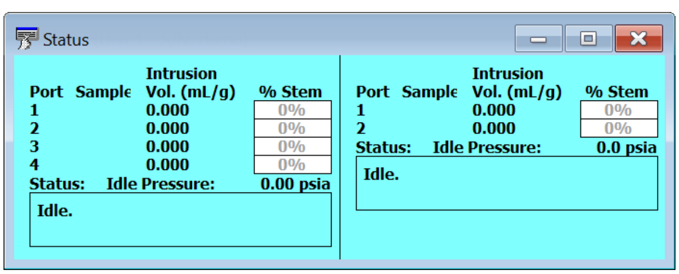
Schematic Shortcuts

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 displays.

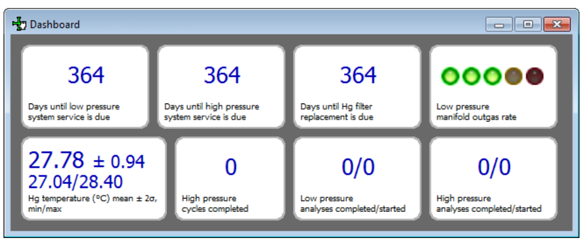
Show Dashboard

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

Selections	Description
Days until high pressure system service is due	The number of days until service is required on the high pressure system. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.
Days until Hg filter replacement is due	The number of days until the mercury filter replacement is due. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.
Days until low pressure system service is due	The number of days until service is required on the low pressure system. When the displayed value is 30 or less, the value is displayed in red. Red negative numbers display if maintenance is past due.

Dashboard Gauges (continued)

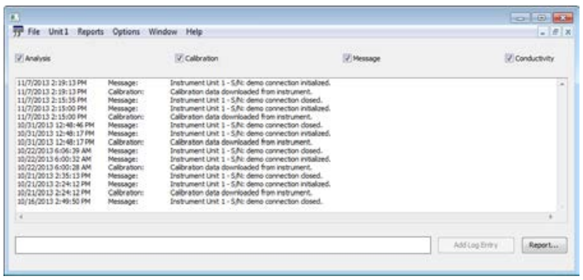
Selections	Description
Hg temperature	Displays the statistics of the manifold temperature reading. The mean, the value at two standard deviations, the minimum, and the maximum display.
High pressure analyses completed / started	Displays N/M where N is the number of high pressure analyses that have finished 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

Selections	Description
Add Log Entry	To manually enter information into the log file, enter the text into the text field to the left of the **Add Log Entry** button, then click **Add Log Entry**.
Analysis / Calibration / Message / Conductivity	Select the logs to display.

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

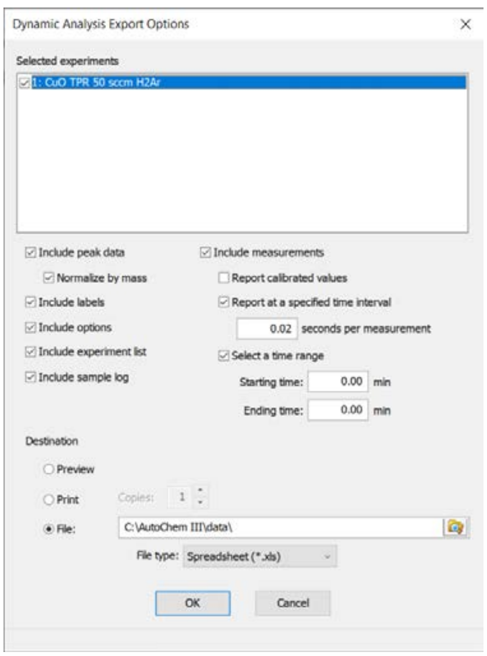
EXPORT FILES

File > Export

Exported Data Example on page H - 1

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

1. Click ***List*** and open an .SMP file.
2. Select an experiment and the applicable options.
3. Click **OK**.



LIST FILES

File > List

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

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

Example of File List

File Listing

No.	File Name	Date	Time	Description	Status
1	13x with CO2 at 0C Port 1B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 1	Complete
2	13x with CO2 at 0C Port 2B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 2	Complete
3	13x with CO2 at 0C Port 3B.SMP	8/10/2020	3:53:54 PM	13x with CO2 Port 3	Complete
4	13x with N2 and TranSeal Port 2.SMP	8/10/2020	3:53:54 PM	13X Zeol Tube 2 w/ FS @ end of analysis, Port 2	Complete
5	13x with N2 and TranSeal Port 3.SMP	8/10/2020	3:53:54 PM	13X Zeol Tube 1A w/ FS @ end of analysis, Port 3	Complete
6	Activated Carbon with Butane C3 Port 1.SMP	8/10/2020	3:53:55 PM	Activated Carbon Tube C3 Butane Port 1	Complete
7	Activated Carbon with Butane C4 Port 3.SMP	8/10/2020	3:53:55 PM	Activated Carbon Tube C4 Butane Port 3	Complete

SOFTWARE UNINSTALL

The software can be uninstalled in two ways. Either method removes only the files required to run the software, not the analysis files.

- Click the Windows *Start* icon. Scroll to the Micromeritics entry. Select the *Uninstall [analyzer]* option, then follow the prompts.
- Locate the *uninstall.exe* file in *C:\Program Files (x86)\Micromeritics\[analyzer name]* (or wherever the application was installed). Double-click the *uninstall.exe* file, then follow the screen prompts.

SOFTWARE UPDATES

> A User Account Control in the Windows operating system must be enabled to ensure all components of the Micromeritics application are correctly installed. If UAC is not enabled, right-click the *setup.exe* installer file and select *Run as administrator*.

The most current version of the instrument software can be found on the Micromeritics web page (www.Micromeritics.com).

When performing a software update, existing data files are not overwritten.

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

3 Sample Files

Option Presentation on page 2 - 8

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

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

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

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

To change the view for the selected window, use the drop-down list at the bottom of the sample file editor.

CREATE SAMPLE FILES

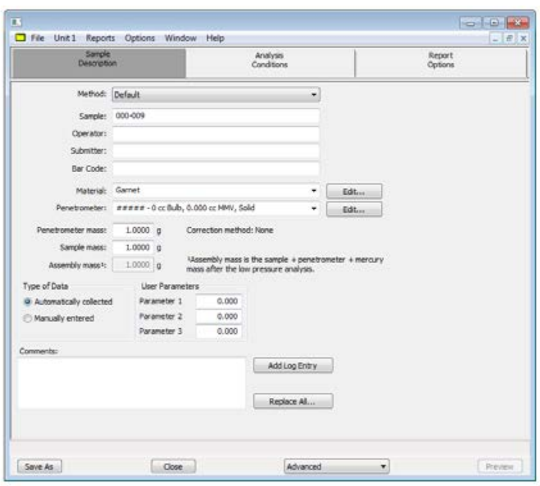
Options > Option Presentation > [Advanced / Basic / Restricted]
File > New Sample
File > Open > [.SMP File]

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

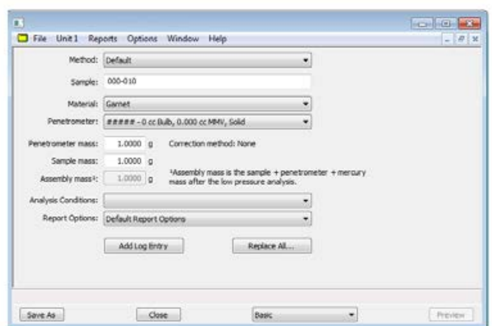
Specify or change the option presentation by selecting ***Options > Option Presentation,*** or use the view selector drop-down list at the bottom of the window.

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

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



Advanced option presentation



Basic option presentation

Sample Files

Selections	Description
Assembly mass [*text box*]	This field is entered after the low pressure analysis is completed. Sample + penetrometer + mercury mass after the low pressure analysis.
Bar Code [*text box*]	Use to enter additional information about the sample, such as a sample lot number, sample ID, etc.
Comments [*text box*]	Enter comments to display in the report header about the sample or analysis.
Material [*drop-down box*]	Select the material to be analyzed from the drop-down list.
Method [*drop-down box*]	Select a method from the drop-down list.
Operator [*text box*]	Enter operator identification information. This field label may have been renamed or may not display if modified in ***Options > Default Methods.***
Penetrometer [*drop-down box*]	Select a penetrometer file from the drop-down list, or click **Edit** to modify or create a new Penetrometer file.
Penetrometer mass [*text box*]	Enter the mass of the penetrometer.
Sample [*text box*]	Enter a sample description.
Sample mass [*text box*]	Enter the mass of the sample.
Submitter [*text box*]	Enter submitter identification information. This text box may have been renamed or may not display if modified in ***Options > Default Methods.***
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.
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 *SPC Report*.

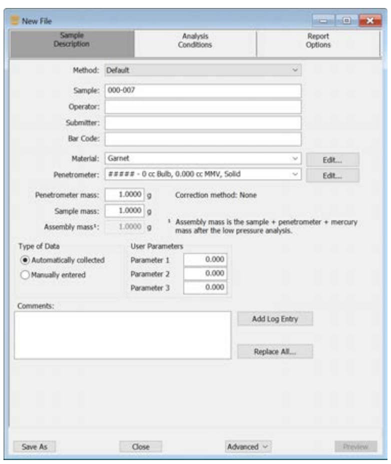
For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 2*.

OPEN A SAMPLE FILE

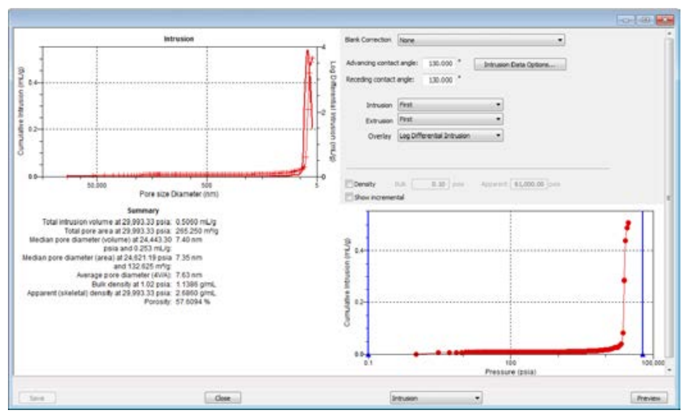
File > Open > [.SMP File]

When working with an existing sample file, consider copying the sample file to maintain the original configuration options.

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



Tabbed file editor in Advanced view



Example of a Report window

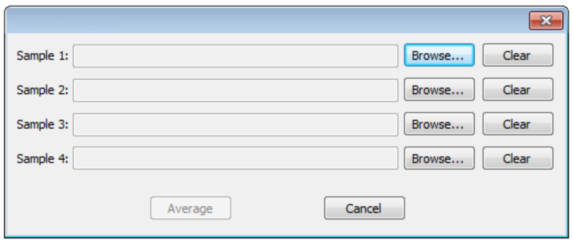
To view the tabbed file editor for a sample file with a *Complete* status, select *Advanced* from the view selector drop-down list at the bottom of the window.

SAMPLE AVERAGING

File > Average

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

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



BASELINE ERROR CORRECTION METHODS

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

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

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

- formula correction, and
- blank correction

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

CREATE BLANK CORRECTION FILE

File > Open > [.SMP file name]

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

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

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

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

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

1. Create the sample file. Enter zero for the sample mass.
2. Perform the low and high pressure analysis.

An alternative method for creating the blank correction file is to perform a *High Pressure Differential* analysis which analyzes the blank and sample simultaneously.

Formula Correction

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

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

Combine a Penetrometer Calibration and Blank Correction File

1. Calibrate the penetrometer. 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 record the assembly mass. Use a syringe to withdraw a small amount of mercury from the tip of the capillary so that the mercury column recedes approximately 1/8 in. to 1/4 in. into the stem. This enables measurement of any rise or fall of mercury in the stem during high pressure analysis and prevents spilling of mercury into the high pressure chamber. Then install the penetrometer in a high pressure port.
3. Create blank correction files by completing the high pressure run.
4. Create a new *Penetrometer Properties* file for this penetrometer.. From the file selection list, choose the default file for this type of penetrometer. Change the identifier to include the penetrometer serial number. Enter the calibrated volume and save the file. The *Penetrometer Properties* file now includes the calibrated penetrometer volume. A blank correction file has also been created for this penetrometer.

Manually Enter Data

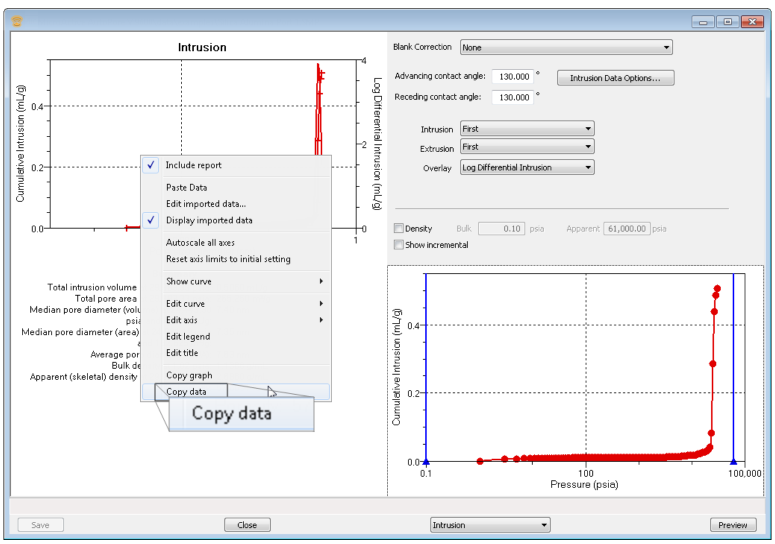
This process allows the manual entry of pressure data from a sample file with a *Complete* status. There are two methods for manually entering data into a sample file:

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

Copy and Paste Manually Entered Data

> To display the file status in a search window, go to ***File > Open***. Right-click the column header then click *More...* Scroll to the *MIC* entries and enable *MIC Status*.

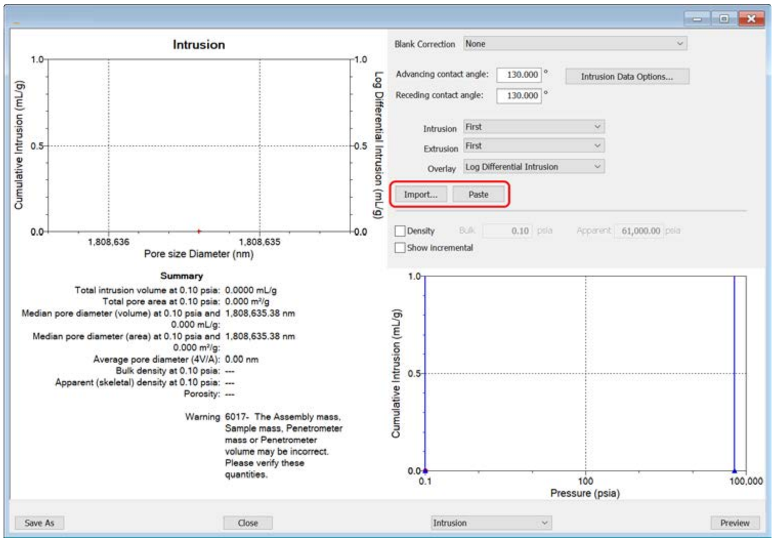
1. Open a sample file with a *Complete* status. The file will open in the interactive reports window.
2. Right-click in the graph area of the interactive reports window, then select *Copy data*.



Example of Report window

3. Open another sample file using the *Advanced* option presentation.
4. On the *Sample Description* tab, select *Manually entered* in the *Type of Data* group box.

5. In the view selector drop-down list at the bottom of the window, click *Advanced*, then select *Intrusion*.



6. Ensure that all parameter fields are set appropriately, then click **Paste**.

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:

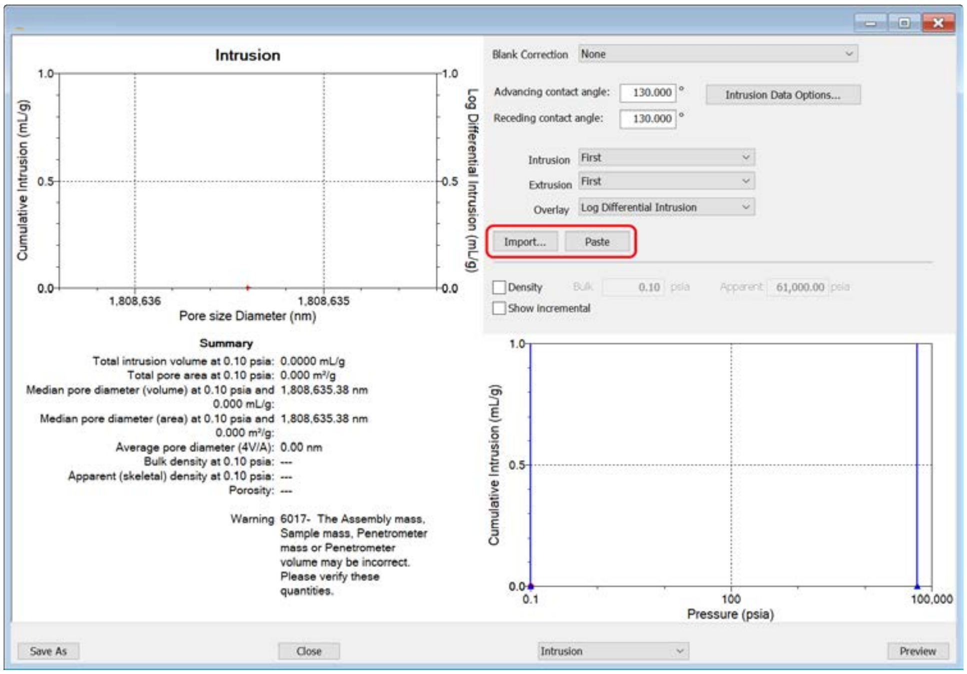
- Pressure (psi)
- Pressure (MPa)
- Pressure (kPa)
- Pressure (Pa)
- Pressure (bar)
- Pressure (mbar)
- Pressure (Atm)
- Intrusion (cm^3)
- Intrusion (cm3)
- Intrusion (mL)
- Intrusion (mm^3)
- Intrusion (mm^3)

Sample Mercury Porosimetry ASCII Text File

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

To import the ASCII text file

1. Open a new sample file in *Advanced* option presentation.
2. On the *Sample Description* tab, select *Manually entered*.
3. In the view selector drop-down list at the bottom of the window, click *Advanced*, then select *Intrusion*.



4. Ensure that all parameter fields are set appropriately, then click **Import**.
5. Open the .TXT file. The data from the original sample file is imported and displayed. If an error message displays instead, verify that the .TXT file format is correct.

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4 PARAMETER FILES

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

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

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

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

Parameter File Types

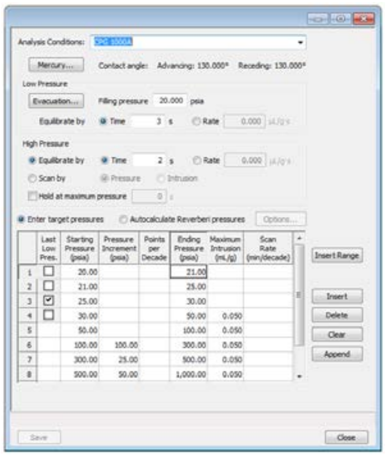
File Type	File Extension
Analysis Conditions	.ANC
Material Properties	.MTP
Method	.MTH
Penetrometer Properties	.PEN
Report Options	.RPO

ANALYSIS CONDITIONS

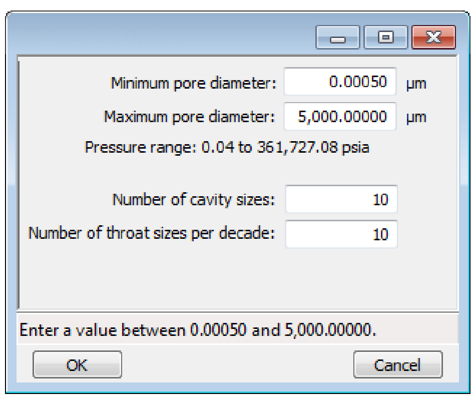
File > Open > [.ANC File]

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

Analysis conditions specify the parameters used to guide an analysis.



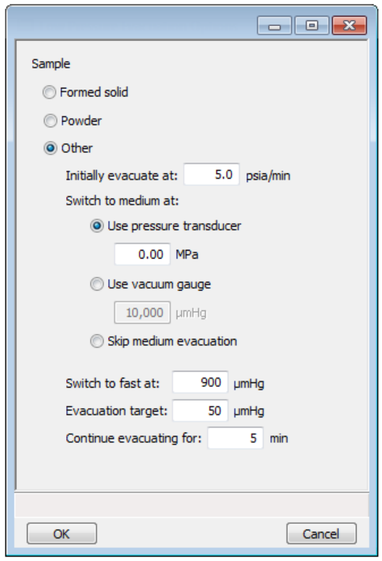
Analysis Conditions

Button	Use to Specify...
Analysis Conditions [*drop-down box***]**	Use to browse for an *Analysis Conditions* file that contains analysis condition parameters to be used in the analysis.
Autocalculate Reverberi pressures [*selection***]**	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 [*selection***]**	Select to manually enter pressures into the table.

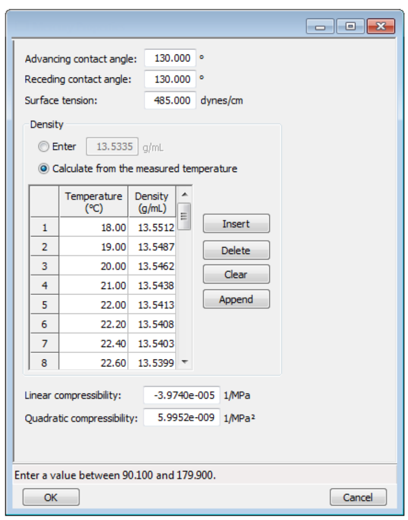
Analysis Conditions (continued)

Button	Use to Specify...
High Pressure **[*group box*]**	**Equilibrate by.** Select the option for equilibration based on elapsed *Time* (in seconds) or decrease in *Rate* of intrusion (or extrusion). Scan by: ■ **Pressure**. The analyzer goes through a sequence of segments with each segment starting at the end of the previous one. Each segment ends at the specified pressure. The pressure is programmed to increase or decrease at a rate to give a constant time per decade of pressure. Along the way, the analyzer takes intrusion points at the specified number of points per decade ending at the specified ending pressure. Also, any points in the pressure table as well as points separated by the maximum intrusion volume, are collected. ■ **Intrusion**. The analyzer goes through a sequence of segments, with each segment starting at the end of the previous one. Each segment ends at the specified pressure The pressure rate is the maximum achievable safe rate (up to 0.5 min/decade) and is programmed to increase or decrease at a rate to give a constant intrusion/extrusion rate. The analyzer takes intrusion points at the specified number of points per decade ending at the specified ending pressure. Also collected are points in the pressure table as well as points separated by the maximum intrusion volume. 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.

Analysis Conditions (continued)

Button	Use to Specify...
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 ◦ 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 mercury at this pressure. It is recommended to set the filling pressure slightly lower than the first low pressure point on the pressure table.

Analysis Conditions (continued)

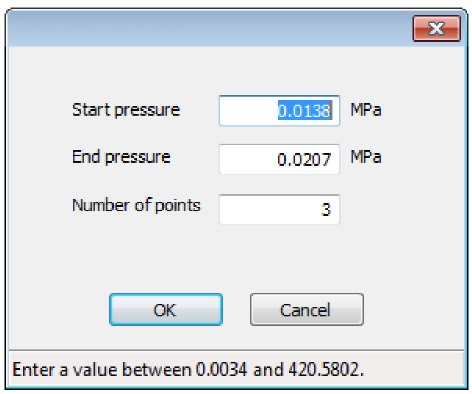
Button	Use to Specify...
	A filling pressure of at least 0.5 psia is recommended. Because mercury generates pressure and because fill pressures less than 0.5 psia can fail to fill the corner radii and gaps between the glass and sample in the pen-etrometer, using a lower pressures may reduce the accuracy 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 extrusion) in mL/g per second.
Mercury [*button*]	Enter the mercury properties. Mercury properties may change with variations in temperature.  **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.**

Analysis Conditions (continued)

Button	Use to Specify...
	■ **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 row 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. If both columns are blank, only the *Ending Pressure* will be used. **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 analyzer automatically takes additional readings between points on the pressure table when this volume of additional intrusion is detected. Enter the intrusion volume per gram of sample that must be reached in order for additional data pair readings to be recorded. Use 0 to prevent readings between pressure points. See ***Maximum Intrusion Volume Option on page F - 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

Analysis Conditions (continued)

Button	Use to Specify...
	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**. [*button*] 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.

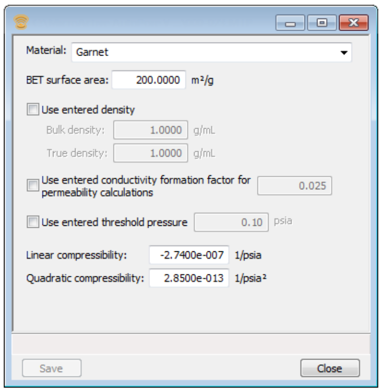


For fields and buttons not listed in this table; see ***Common Fields and Buttons on page 2 - 2*****.**

MATERIAL PROPERTIES

File > Open > [.MTP File]

Material properties specify the properties of the material to be used in an analysis. This information is only required if it is to be used in a report.



Material Properties

Selections	Description
BET Surface Area **[*text box*]**	Enter the BET surface area.
Linear compressibility **[*text box*]**	Enter the linear compressibility coefficient.
Quadratic compressibility **[*text box*]**	Enter the quadratic compressibility coefficient.
Use entered conductivity formation factor... [*check box*]	Select if using the entered conductivity formation factor for permeability calculations and enter the factor in the text box.

Material Properties (continued)

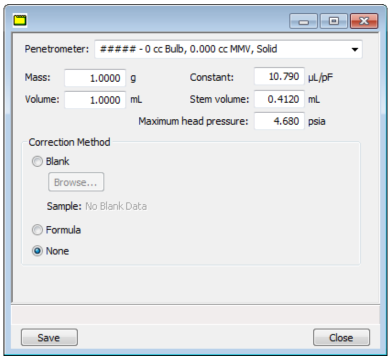
Selections	Description
Use entered density [*check box*]	Select if using the entered density and enter the bulk and/or true density in the appropriate text boxes. The entered values are used in the intrusion summary results, Mayer-Store report, and the pore structure report. The Mayer-Stowe particle size calculation has meaning mostly in the range of interstitial filling. For larger pressures after which the interstitial space is filled, the particle size calculation has less meaning.
Use entered threshold pressure [*check box*]	Select if using the entered threshold pressure and enter the threshold pressure in the text box.

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

PENETROMETER PROPERTIES

File > Open > [.PEN File]

Penetrometer properties specify the properties of the penetrometer to be used in an analysis. It is recommended to use a copy of the system penetrometer file that corresponds to the penetrometer bowl.



Penetrometer Properties

Selections	Description
Constant [*text box*]	Enter the penetrometer constant provided with the penetrometer. Verify the field contents if the *Replace* option has been used.
Correction Method [*selection*]	**Blank.** If using the blank correction method, click **Browse** to select a sample file. **Formula.** **None.**
Mass [*text box*]	Mass of the empty, assembled penetrometer (excluding the spacer).
Maximum head pressure [*text box*]	Enter the maximum head pressure.

Penetrometer Properties (continued)

Selections	Description
Penetrometer [*drop-down box***]**	Enter identifying information for this file. This information can consist of the last two digits of the base part number and the 4 digit penetromer serial number. For example: **Penetrometer part number:** 950-61708-00 **Serial number:** 1420 **Suggested identification number:** 08-1420
Stem volume [*text box***]**	Enter the penetrometer stem volume provided with the pen- etrometer.
Volume [*text box***]**	Enter the volume of the penetrometer. This is required to calculate density or when using the blank correction formula.

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

REPORT OPTIONS

File > Open > [.RPO File]

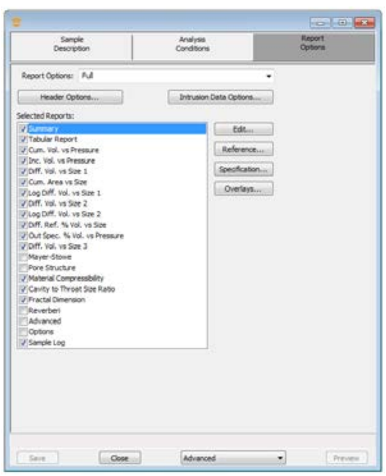
(or click the *Report Options* tab when in *Advanced* format)

Additional reports are available using the *Reports* menu.

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

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

Report Options files may be defined to include overlay options. This system allows the overlay of up to 25 plots of different samples onto a plot of the same type or overlay one plot type onto a different plot type from the same analysis.

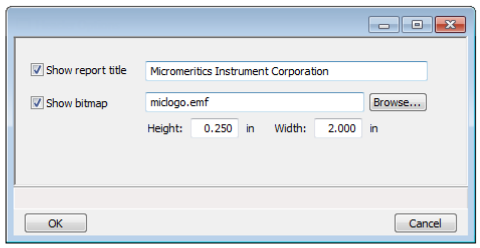


Report Options

<table>
<tr><th>Selections</th><th>Description</th></tr>
<tr><td>Edit [button]</td><td>Edit graph options.

Plot Points. Select to plot points on the graph.

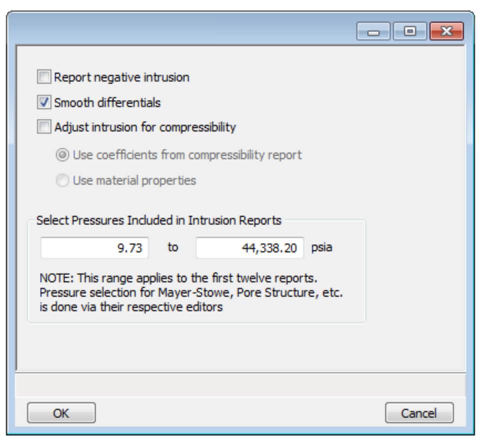
Plot Curve. Select to plot curves on the graph.

Show Histogram. Select to show the graph as a histogram. When selected, the Plot Points and Plot Curve selections are disabled.</td></tr>
<tr><td>Header Options [button]</td><td>

Show report title. Enter a report title to appear on the report header.

Show bitmap. Displays the selected graphic on the report header.

Height / Width. Enter the height and width of the selected graphic. These values determine the graphic appearance on the generated report.</td></tr>
<tr><td>Intrusion Data Options [button]</td><td>This button is also located on Intrusion reports.



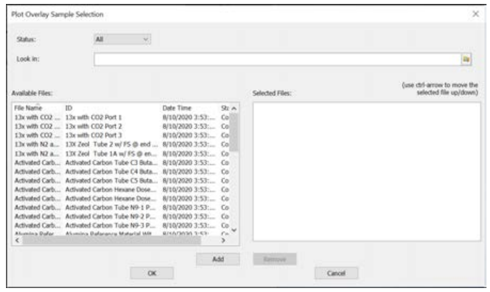
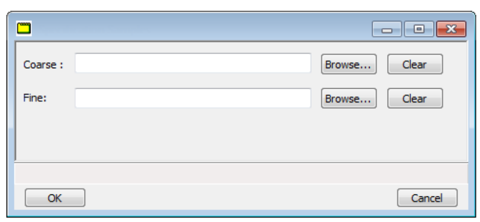
Adjust intrusion for compressibility:

■ Use coefficients from compressibility report. Select to have the application use the coefficients from the Material Com-</td></tr>
</table>

Report Options (continued)

Selections	Description
	pressibility report rather than from the *Material Properties*. ■ **Use material 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 analyzer malfunction. **Select Data Included in Intrusion Reports.** Enter range of pressure. This range applies to the first twelve reports on the options list. When these selections are changed in the intrusion report, the lower right graph x-axis and the intrusion data in the graph will change accordingly. The two blue range bars can then be used to select the required range of data. ■ **Pressure.** The default behavior of the application is to select data using pressure as this is the quantity which is varied during mercury porosimetry analysis. The maximal range of data (all data) is selected by default. It is advisable to use this default setting unless there is a specific goal is required. ■ **Pore size.** Uses the Washburn equation to translate the user entered pore size range to a corresponding pressure range used for data selection. ■ **Particle size.**This option is less commonly used. In the *Material Properties* window, select the checkbox *Use entered density* and complete the *Bulk density* and effective *Particle density* fields. The effective *Particle density* and *Bulk density* quantities are necessary to compute the interstitial porosity of the powder used in the Mayer-Stowe calculation. **Smooth differentials.** Select to apply smoothing to any differentials reported in tables or graphs.

Report Options (continued)

Selections	Description
Overlays [*button*]	Select the files to overlay. 
Reference [*button*]	Select a sample file to compare analysis results with the current sample. 
Selected Reports [*check box*]	Select the report names to include in the report.
Specification [*button*]	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 ***Common Fields and Buttons on page 2 - 2***.

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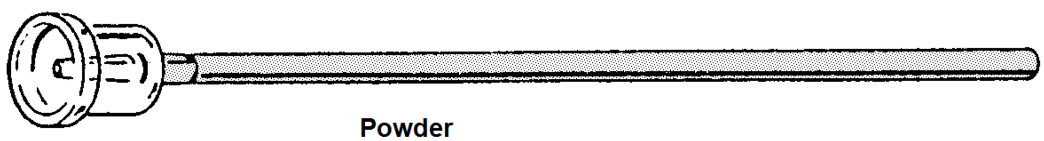
5 Perform an Analysis

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

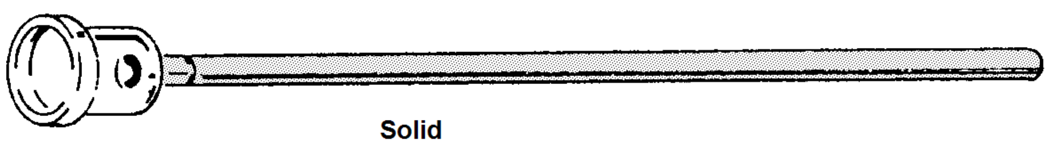
Select a Penetrometer

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

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



Powder



Solid

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

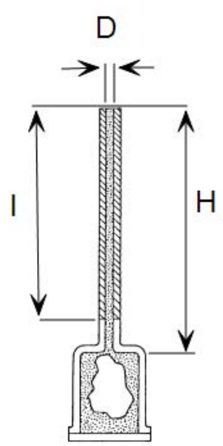
Penetrometer Selection Guide

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

Penetrometer Selection Guide (continued)

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

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



- Maximum measurable volume = [(3.14)(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 analyzer 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. ***Clean the Penetrometer below***
2. ***Load the Sample on the next page***
3. ***Seal the Penetrometer on page 5 - 7***
4. ***Weigh the Assembled Penetrometer with Sample on page 5 - 9***
5. ***Install the Penetrometer on page 5 - 9***

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 - 34***.
5. Turn the penetrometer over and pour remaining sample into the waste container.
6. Immerse the penetrometer in the detergent solution. Clean the outside of the penetrometer stem and the bulb with a brush. Then clean the inside of the stem with one of the smaller brushes.

7. Rinse the penetrometer with warm water. Hold the penetrometer upright and 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 G - 8***.
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.

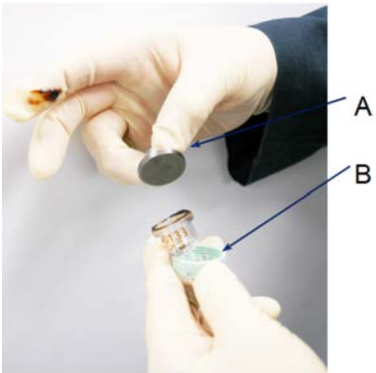
1. A vacuum-tight seal is required. Therefore, vacuum grease (Apiezon H) must be used to fill the inevitable roughness of the ground glass lip and polished surface of the cap. To apply grease:

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

Lightly grease the tip of the bulb

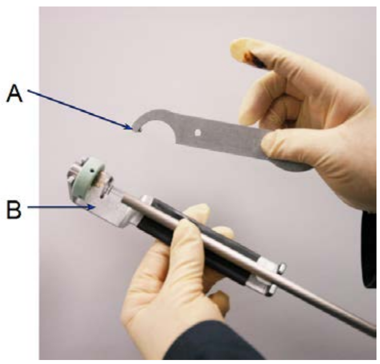
   a. Use a fingertip to apply a light coating of grease to the lip of the bulb.
   b. Smooth the grease evenly around the lip of the bulb.
   c. Remove all excess grease from both the inside and outside of the bulb.

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



A. Place a cap over the greased bulb and turn 1/2 turn to seat
B. Slide the nut over the penetrometer stem and finger tighten

3. Place the penetrometer tool over the nut.



A. Wrench peg
B. Place penetrometer tool over the nut and cap

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.

A. Wrench peg
B. Place penetrometer tool over the nut and cap

5. Weigh the penetrometer before installing it in the low pressure port.

WEIGH THE ASSEMBLED PENETROMETER WITH SAMPLE

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

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

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

INSTALL THE PENETROMETER

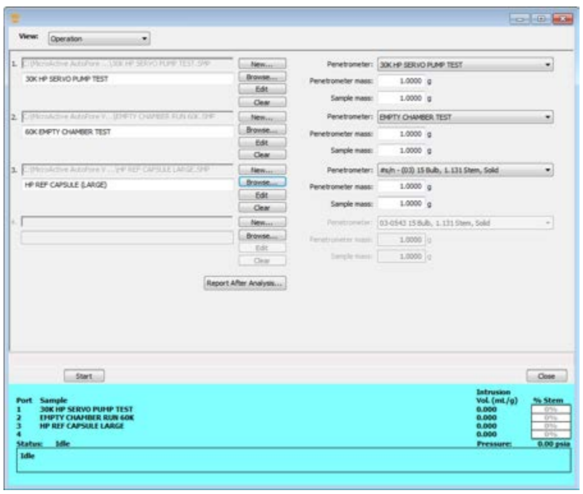
Install a Penetrometer in a High Pressure Chamber on page 10-6
Install a Penetrometer in a Low Pressure Port on page 10-8
Remove a Penetrometer from a High Pressure Chamber on page 10-10
Remove a Penetrometer from a Low Pressure Port on page 10-11

Perform a Low Pressure Analysis

Unit [n] > Low Pressure Analysis

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

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



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

4. Click **Start** to start the analysis. A window displays data as they are collected. A short delay is encountered before the port status at the bottom of the window changes from the *Idle* state.

- 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

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

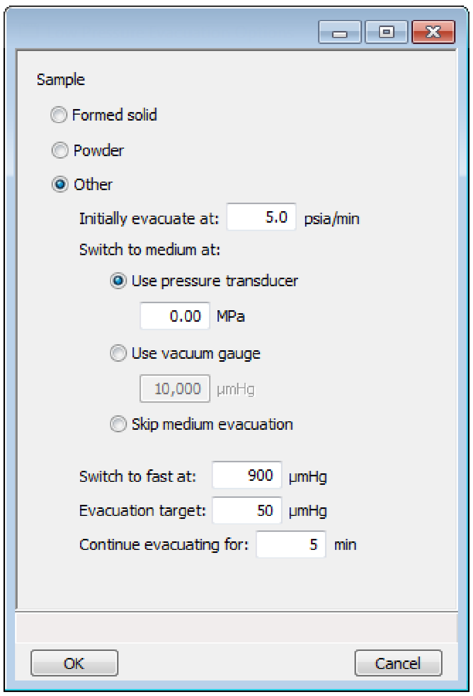
For fields and buttons not listed in this table; see ***Common Fields and Buttons on page 2 - 2*****.**

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.



Evacuate Low Pressure

Selections	**Description**
Initially evacuate at	Enter the initial maximum evacuation rate.
Switch to medium at	Enter the method and pressure the system must reach before medium evacuation begins. ■ Use pressure transducer ■ User vacuum gauge ■ Skip medium evacuation .
Switch to fast at	Enter the pressure the system must reach before fast evacuation begins.

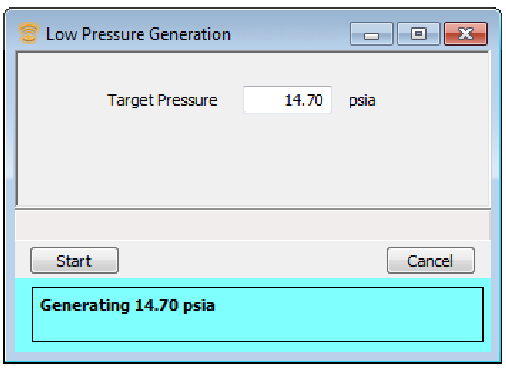
Evacuate Low Pressure (continued)

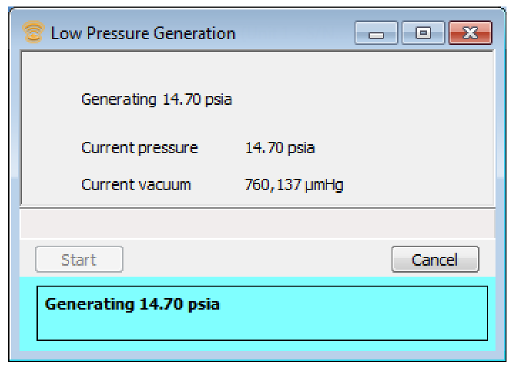
Selections	Description
Evacuation target	Enter the evacuation pressure.
Continue evacuating for	Enter the evacuation duration.

Generate Low Pressure

Unit [n] > Generate Low Pressure

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





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

Monitor a Low Pressure Analysis

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

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

When monitoring a low pressure analysis, the analyzer schematic can be displayed to show the state of the low pressure system components. See ***Show Instrument Schematic on page 2 - 15***.

Low Pressure Analysis

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

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

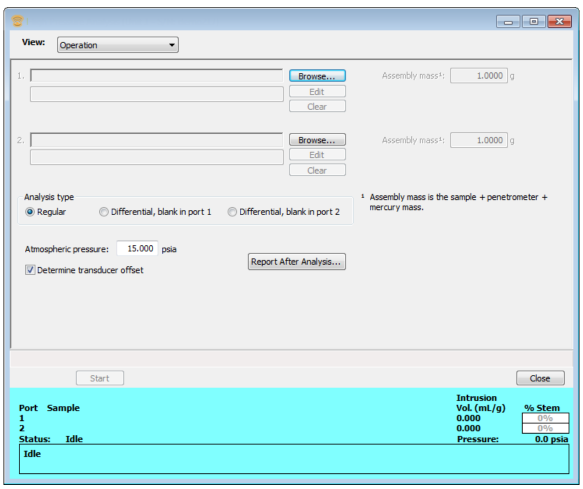
PERFORM A HIGH PRESSURE ANALYSIS

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

Unit [n] > High Pressure Analysis

Low and high pressure analyses can be performed simultaneously.

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



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

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

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

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

2. Install a Penetrometer in a High Pressure Port.

3. Click **Start** to start the analysis immediately. A live graph of the analysis displays as data are being collected.
4. Remove the penetrometer after the analysis

> 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

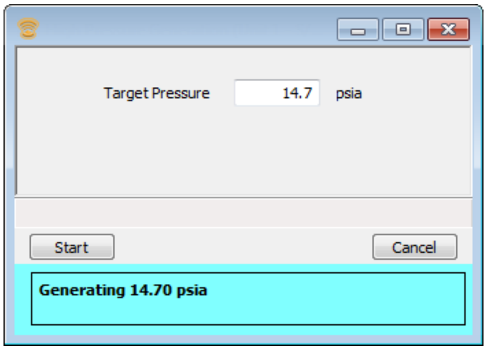
Selections	Description
Analysis type	**Regular.** Performs a differential analysis with the blank penetrometer in port 1. **Differential, blank in port 1.** Performs a differential analysis with the blank penetrometer in port 1. **Differential, blank in port 2.** Performs a differential analysis with the blank penetrometer in port 2.
Assembly mass	Sample + penetrometer + mercury mass.
Atmospheric pressure	Enter the atmospheric pressure if it differs from the default entry. The default entry is pulled from the information entered in ***Options > Atmospheric Pressure***.
Determine transducer offset	Select to allow the system to determine the transducer offset. The default entry is pulled from the information entered in ***Options > Atmospheric Pressure***.

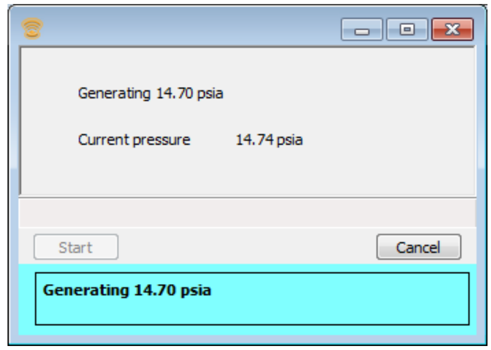
> **For fields and buttons not listed in this table; see** ***Common Fields and Buttons on page 2 - 2*****.**

GENERATE HIGH PRESSURE

Unit [n] > Generate High Pressure

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





MONITOR A HIGH PRESSURE ANALYSIS

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

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

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

High Pressure Analysis

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

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

HIGH PRESSURE DIFFERENTIAL ANALYSIS

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

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

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

1. Create a sample file. Also create a sample file for the blank penetrometer using *None* for the correction method.
2. Run the low pressure analysis on both files.
3. In the high pressure analysis wizard, select *Differential, blank in port 1* or *Differential, blank in port 2* as the analysis type.
4. Start the high pressure analysis.

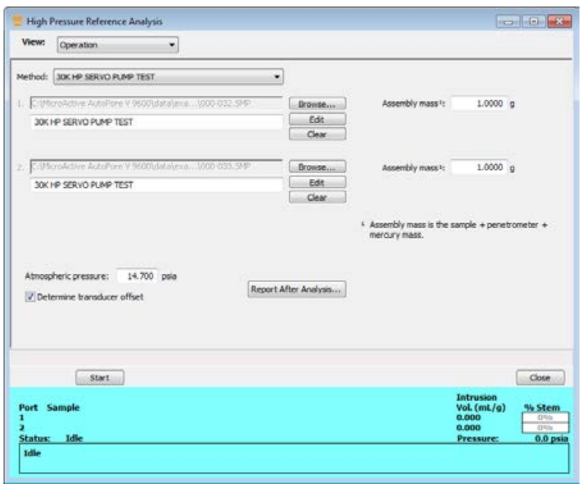
Reference Material Analysis

High Pressure Reference Material Analysis

Unit [n] > High Pressure Reference Analysis

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

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



High Pressure Reference Material Analysis

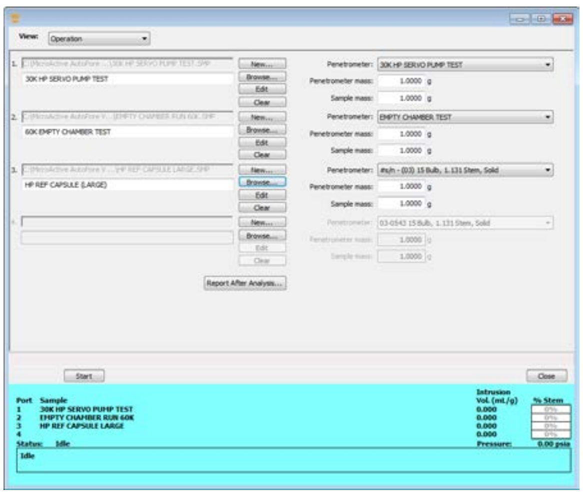
Selections	Description
Assembly mass	This information is pulled from the selected sample file. Sample + penetrometer + mercury mass.
	For fields and buttons not listed in this table; see *Common Fields and Buttons on page 2 - 2*.

LOW PRESSURE REFERENCE MATERIAL ANALYSIS

Unit [n] > Low Pressure Reference Analysis

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

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



Low Pressure Material Reference Material Analysis

Selections	Description
Penetrometer	Penetrometer description from the selected sample file.
Penetrometer mass	Penetrometer mass from the selected sample file.
Sample mass	Sample mass from the selected sample file.

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

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 worksheet 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

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

Reports > Start Report

Generates a report on a sample analysis.

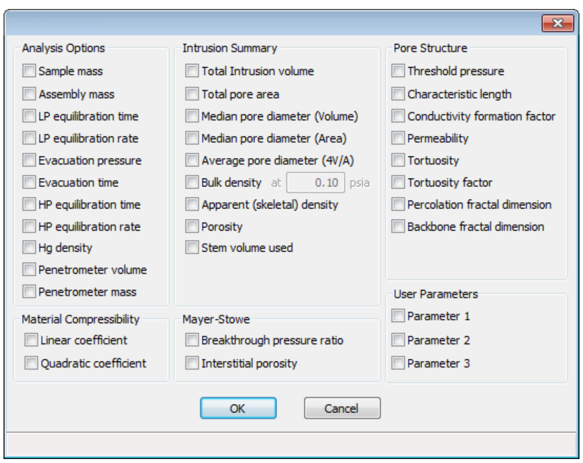
Reports > Close Reports

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

SPC Report

Reports > SPC Report Options

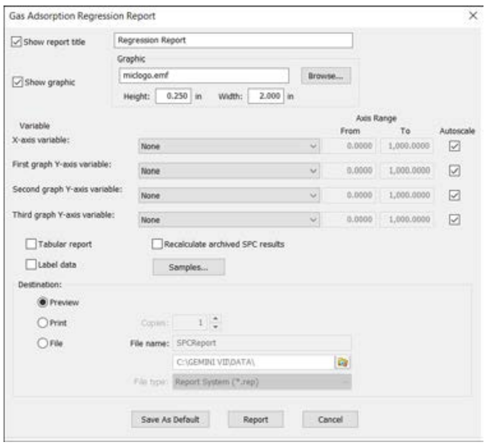
Use to generate reports with various *SPC* (Statistical Process Control) options. All selected variables must be computed for each sample file used in an SPC report; therefore, it is more efficient to select only the necessary variables.



REGRESSION REPORT

Reports > Regression Report

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



Regression Report

Selections	Description
Autoscale [*check box*]	When enabled, allows the x- and y-axes to be scaled automatically.
Axis Range [*text box*]	Enter the beginning and ending values for the x- and y-axis ranges. These fields are disabled if *Autoscale* is selected.
Label data [*check box*]	Use to label the points on the plot to correspond with the values in the sample files.
Recalculate archived SPC results [*check box*]	If selected, SPC results are archived in the sample files when a report is generated. If deselected, previously archived results are used to save time when generating reports. Since this option updates and saves sample files, do not use it with sample files that need to remain compatible with other applications.
Report [*button*]	Generates the report.
Samples [*button*]	Select additional sample files to add to the report.
Save as Default [*button*]	Click to save selected report options as default report settings.

Regression Report (continued)

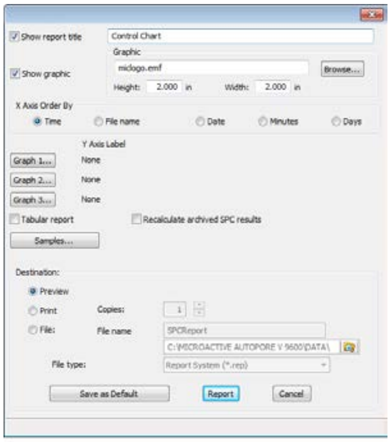
Selections	Description
Show graphic [*check box*]	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic's appearance on the generated report.
Show report title [*check box*]	Select then enter a report title to appear on the report header.
Tabular report [*check box*]	Generates a tabular report of the included samples that contains the numeric values contributed by each sample.
X- and Y-Axis variable [*drop-down box*]	Designates the x- and y-axes variables. The variables in the drop-down lists are those selected in the ***Reports > SPC Report Options*** window. Use these options to plot the regression of up to three y-axis variables against the x-axis variable.

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

CONTROL CHART REPORT

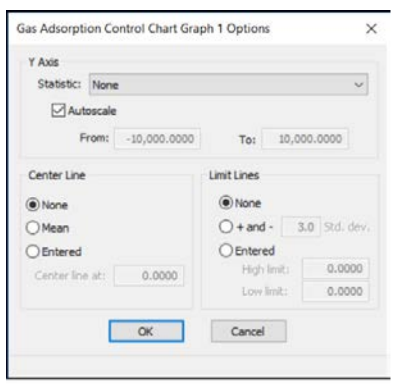
Reports > Control Chart

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



Control Chart Report

<table>
<tr><th>Selections</th><th>Description</th></tr>
<tr><td>Graph [n] [button]</td><td>
Defines the y-axis of each graph.



Statistic. Displays the SPC variables selected on the Reports > SPC Report Options window. The selected variable will be plotted for each selected sample. This selection also becomes the y-axis label.

Autoscale. Allows the y-axis to be scaled automatically. To specify a range, deselect this option and enter a range in the From and To fields.
</td></tr>
</table>

Control Chart Report (continued)

Selections	Description
	Center Line. Displays placement options for the center line in the graph. Select *Entered* to specify placement of the line or *Mean* to place the center line at the calculated mean value for the selected samples. **Limit Lines.** Displays limiting lines options. Lines can be placed at some multiple of the standard deviation or at specified positions (*Entered*). When *Entered* is selected, enter the *High limit* and *Low limit* fields with appropriate values.
Recalculate archived SPC results [*check box***]**	If selected, SPC results are archived in the sample files when a report is generated. If deselected, previously archived results are used to save time when generating reports. Since this option updates and saves sample files, do not use it with sample files that need to remain compatible with other applications.
Report [*button***]**	Generates the report.
Samples [*button***]**	Select additional sample files to add to the report.
Show graphic [*check box***]**	Use to show a graphic on the report header. **Height/Width.** Enter the height and width of the selected graphic. These values determine the graphic's appearance on the generated report.
Show report title [*check box***]**	Select then enter a report title to appear on the report header.
Tabular report [*check box***]**	Generates a tabular report of the included samples that contains the numeric values contributed by each sample.

Control Chart Report (continued)

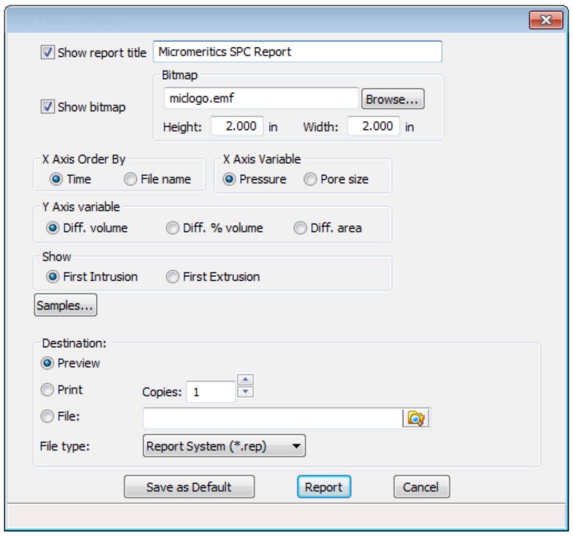
Selections	Description
X Axis Order By **[***group box***]**	Select the order in which x-axis statistics are placed. Sort by: **Time.** Time the files were analyzed. **File name.** Alphanumeric order. **Date.** Date the files were analyzed. **Minutes.** Minutes elapsed from the first file placed on the list, which is the earliest-analyzed file. **Days.** Number of days elapsed from the first file placed on the list, which is the earliest-analyzed file.

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

PSD HISTORY REPORT

Reports > PSD History

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



PSD History Report

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

PSD History Report (continued)

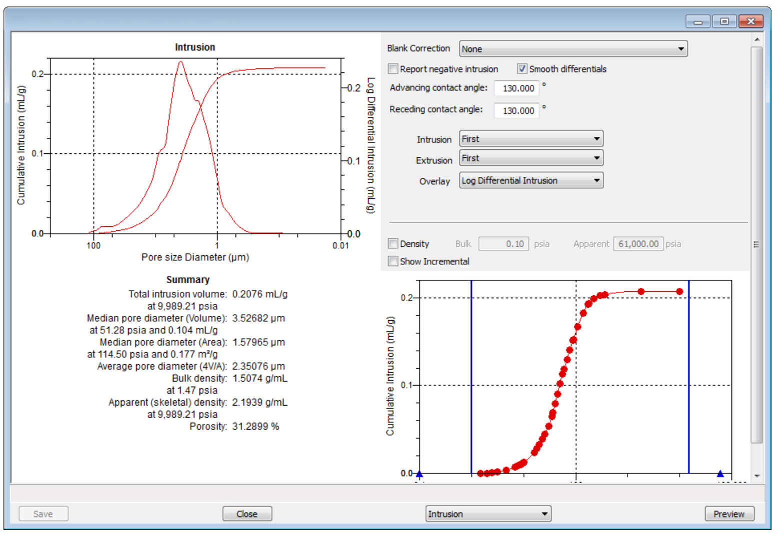
Selections	Description
X Axis Order By **[***selection***]**	Select one of the following: **Time.** Time the files were analyzed. **File name.** Alphanumeric order.
X Axis variable **[***selection***]**	Select one of the following to display on the graph: **Pressure.** **Pore size.**
Y Axis variable **[***selection***]**	Select one of the following to display on the graph: **Diff. volume.** **Diff. % volume.** **Diff. area.**

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

INTERACTIVE REPORTS

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

1. When opening a sample file that contains analysis data, a window with the following information will display:
   - 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 intrusion and/or extrusion segments 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 analysis, click **Preview**.
5. From the view selector drop-down list at the bottom of the window, do either of the following:
   - Change the option presentation of the sample description window to either *Basic* or *Advanced* to modify certain file parameters.
   - 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. Click **Save**.

MICROACTIVE REPORTS

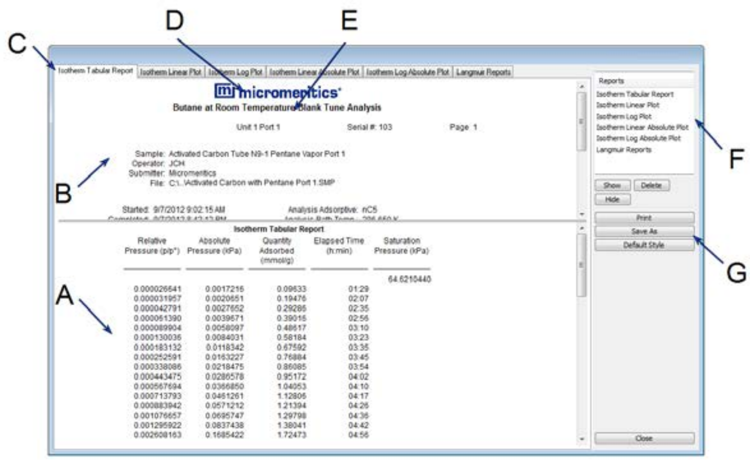
MicroActive reports are generated automatically after an analysis is performed. This feature provides a quick and easy way to investigate and manipulate analysis data using a variety of reporting methods.

When a report is opened, plots and summary data are displayed, and in some reports certain parameters (for example, permeability constant and pore shape exponent) 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.

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.



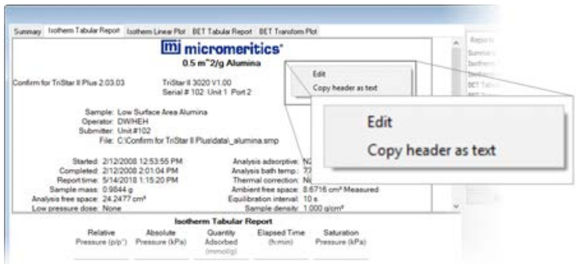
A. Data display (graph or text)
B. Header
C. Generated tabs
D. Graphic
E. Title
F. List box
G. Toolbar

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.

Report Header Shortcuts

Right-click in the report header to display header shortcuts.

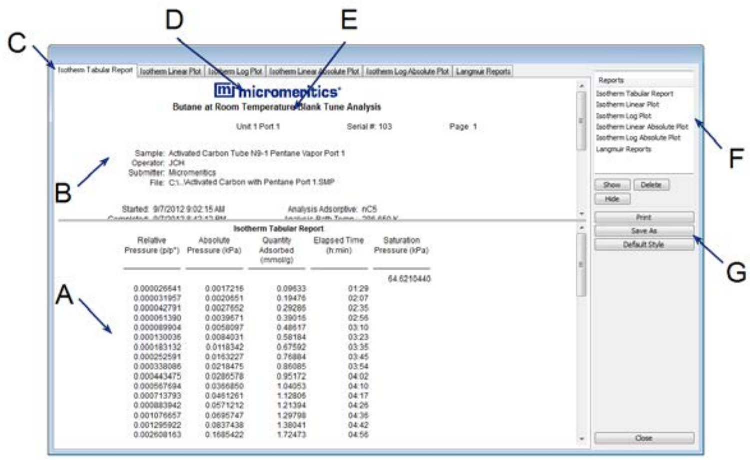


Report Header Shortcuts

Selections	Description
Copy header as text	Copies the report header as text. Text is copied to the clipboard and then can be pasted into other documents.
Edit	Opens a dialog box for editing the report title.

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**.



A. Data display (graph or text)
B. Header
C. Generated tabs
D. Graphic
E. Title
F. List box
G. Toolbar

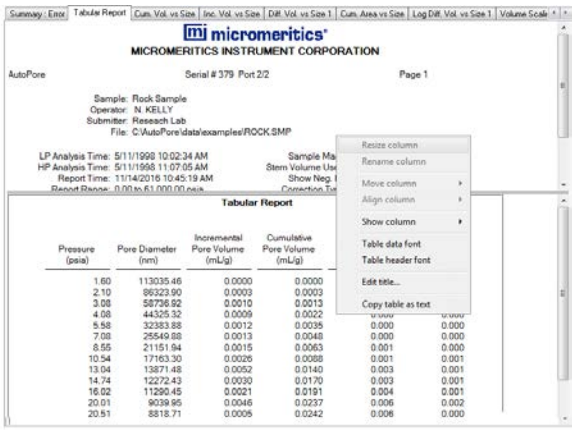
Report Toolbar

Selections	Description
Default Style [*button*]	Specifies default report parameters for fonts and curve properties.
Delete [*button*]	Deletes the selected report in the *Reports* list box. Deleted reports will have to be regenerated if deleted in error.
Hide [*button*]	Hides (or temporarily removes) the selected report from the tabbed view. The report name remains in the *Reports* list box.
Print [*button*]	Displays the *Print* window for report output.
Reports [*group box*]	Contains a list of all generated reports. The same reports display as tabs at the top of the report header unless the report has been hidden using the **Hide** button.
Show [*button*]	Displays the selected or hidden report in the *Reports* list box.

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

TABULAR REPORT FEATURES AND SHORTCUTS

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



Tabular Report Shortcuts

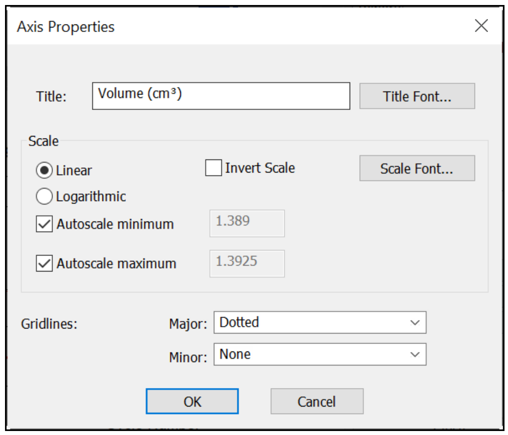
Selections	Description
Align column	Changes the column alignment to either left, right, or centered.
Copy table as text	Copies the report contents to the clipboard as tab-delimited text. It can then be pasted into another document.
Edit title	Edits the report title and/or title font attributes. Click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

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

GRAPH FEATURES AND SHORTCUTS

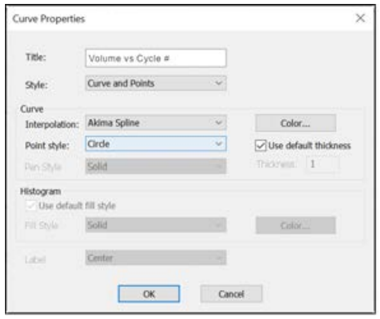
Right-click in the graph area to display graph report shortcuts.

Graph Shortcut Options

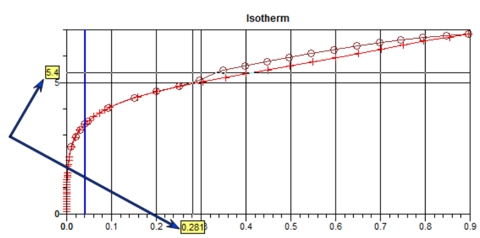
Selections	Description
Autoscale all axes	Returns the report to full view after using the zoom feature.
Edit axis	Edits the selected axis properties.  **Gridlines.** Changes how to display major / minor grid lines. **Scale.** ■ **Autoscale minimum/maximum.** To manually specify minimum / maximum autoscale, deselect the option and enter the new amount in the text box. ■ **Invert scale.** Inverts the scale. ■ **Linear/Logarithmic.** Scales the graph as linear or logarithmic. ■ **Scale font.** Modifies the font for the scale label. Deselect *Use default font* to enable font options. **Title.** Edits the selected axis label. **Title font.** Modifies the font for the selected axis label. Deselect *Use default font*. Select new font attributes for report data. Enable *Use default font* to reset default fonts.

Graph Shortcut Options (continued)

<table>
<tr><th>Selections</th><th>Description</th></tr>
<tr><td>Edit curve</td><td>
Edits selected curve properties.

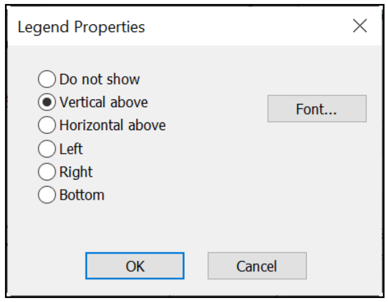


Color. Changes the curve color.
Curve. Changes the interpolation, point style, and pen style for the selected curve. These options are disabled if Use default fill style is selected in the Histogram group box.
Histogram. Enabled only if Histogram is selected in the Style drop-down list. Specifies the type of fill, fill color, and label position for the selected curve.
Label. Designates where the graph point labels will display (left, right, center, etc.) on the SPC report.



Style. Selects another style for the collected data curve.
Title. Changes the title of the selected curve.
Use default thickness. Uses the default curve thickness. Deselect to enter a new thickness number in the Thickness text box.
</td></tr>
</table>

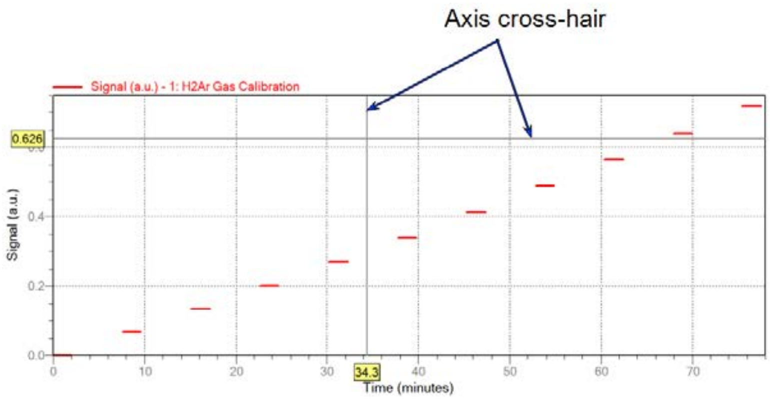
Graph Shortcut Options (continued)

Selections	Description
Edit legend	Changes the legend location and font. 
Edit title	Changes the report title.
Paste Data	Used with pore distribution data reports only. Pastes ASCII text data from the clipboard onto 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.

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

AXIS CROSS-HAIR

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



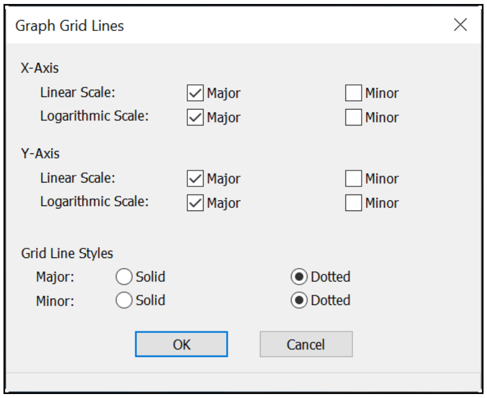
Example of Axis cross-hair feature

ZOOM FEATURE

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

GRAPH GRID LINES

Options > Graph Grid Lines



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

Graph Grid Lines

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

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

GRAPH AND SAMPLE OVERLAYS

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

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

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

GENERATE PORE SIZE DISTRIBUTION GRAPH OVERLAYS

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

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

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

Pore size distribution report overlays menu selections are:

- Copy data
- Paste data
- Edit Imported Data
- Display Imported Data

IMPORT ASCII TEXT DATA

ASCII text file format rules

- The header must consist of one line to include title, two unit specifications, and distribution type:
    - Accepted pore dimension units are: A, nm, um
    - Accepted pore volume units are: cm^3/g, cm^3/g, mL/g
    - Accepted distribution types are: cumulative, incremental

  Two examples of a header format:

  My Title (A, cm^3/g, incremental)
  My Title (A, cm^3/g, cumulative)

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

Sample ASCII Text File

```
silica alumina bjh (A, cm3/g,
cumulative)

456.657        0.0133559
444.847        0.0546427
429.168        0.0869924
425.419        0.119721
419.629        0.132681
360.634        0.156611
340.859        0.197672
326.601        0.233092
```

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

1. Create the ASCII text file using the proper format as indicated above.
2. In the analyzer application, go to ***File > Open***.
3. Select a sample file to overlay graphs on to.
4. Click **Open** (or double-click the file name).
5. Right-click in the graph area and select *Edit imported data*.

6. In the *Select Imported Overlays* window, if the ASCII text file does not display, click **Import** to locate the file.
7. Select the ASCII text file in the *Select Imported Overlays* window, then click **OK**. If an error message is displayed, verify that the .TXT file format is in the correct format.
8. To include the overlay data in a printed report, see *Print Pore Size Distribution Overlay Data in Reports below*.

OVERLAY PORE SIZE DISTRIBUTION DATA USING COPY/PASTE

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

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

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

Print Pore Size Distribution Overlay Data in Reports

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

Import ASCII Pore Distribution Data

[*Manually Enter Data on page 3 - 8*]

Import an ASCII Text File using Graph Shortcuts

1. Create an ASCII text file.
2. Open a report with a *Complete* status.
3. Select a pore-size distribution report from the view selector drop-down list at the bottom of the window.
4. Right-click on the graph and select *Edit imported data* on the shortcut menu.
5. If the ASCII text file does not display on the *Selected Imported Overlays* window, click **Import**.
6. Locate and select the file, then click **Open**. Header information from the ASCII text file will appear in the *Select Imported Overlays* window.
7. Select the entry, then click **OK**. If an error message appears, verify that the .TXT file format is correct.
8. To hide or show imported data, right-click in the graph area and use the *Display imported data* option on the shortcut menu.

Copy/Paste an ASCII Text File using Graph Shortcuts

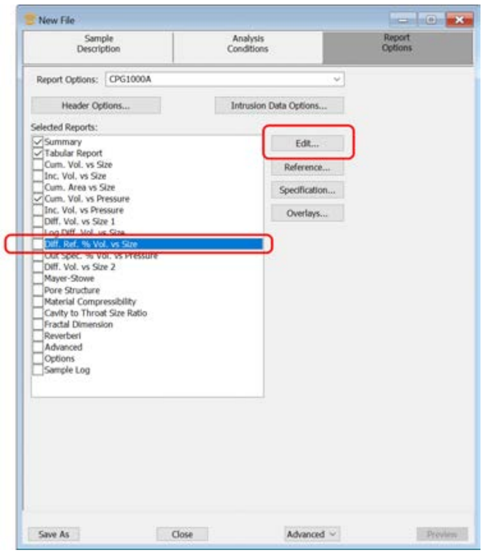
1. Create an ASCII text file.
2. Copy the ASCII text data to the clipboard.
3. Open a report with a *Complete* status.
4. Select a pore-size distribution report from the view selector drop-down list at the bottom of the window.
5. Right-click on the graph and select *Paste data* on the shortcut menu.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

Copy/Paste Graph Data from Another Graph

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

Overlay Multiple Graph Options

1. Go to ***File > Open***.
2. Select the .SMP file, then click **Open**.
3. Select *Advanced* from the view selector 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**.

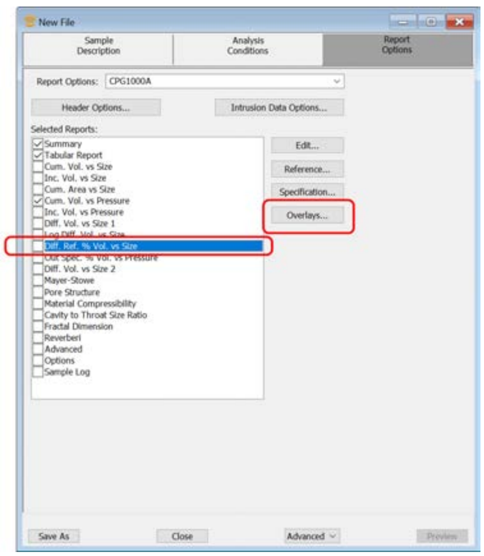


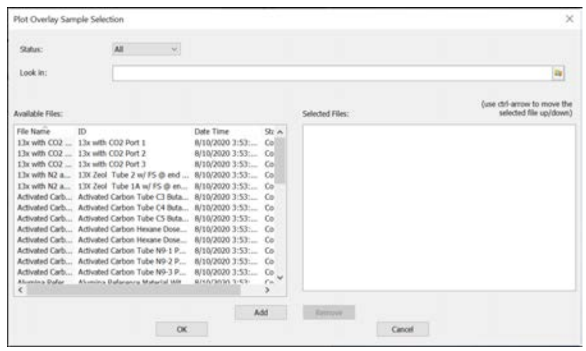
6. In the *Y-Axis* group box, select *Variable* and/or *Overlay* options.
7. Click **OK** to return to the *Report Options* tab.
8. Click **Save**, **Save As**, or **Preview**.

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 view selector drop-down list at the bottom of the window to display the *Report Options* tab.
3. Click the *Report Options* tab.
4. In the *Reports* list box, highlight a plot report, then click **Overlays**.

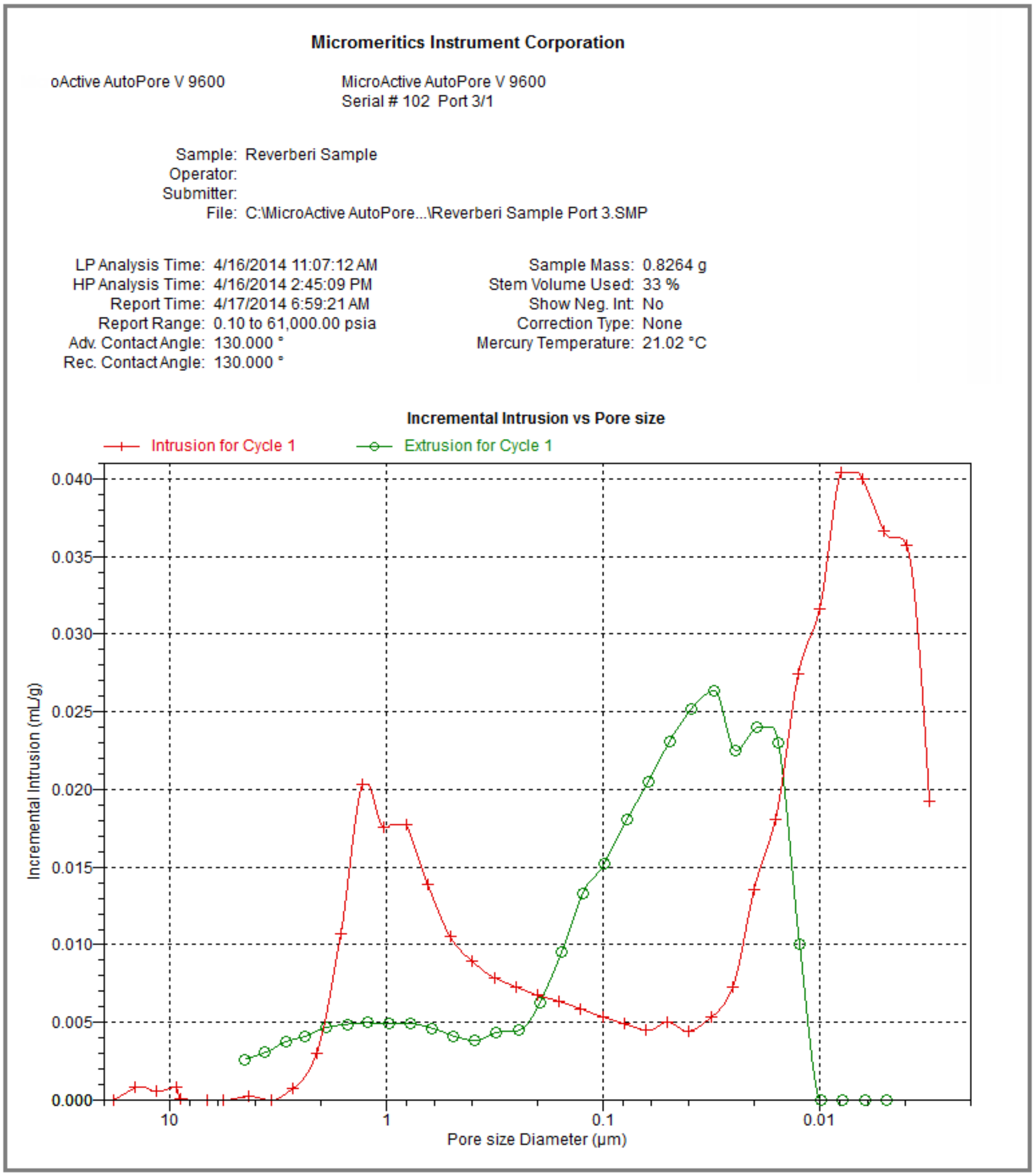




5. On the *Plot Overlay Sample Selection* window, move files from the *Available Files* box to the *Selected Files* box.
6. Click **Save**, **Save As**, or **Preview**.

Report Examples

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

Silica Alumina Reference Material Report

Micromeritics Instrument Corporation

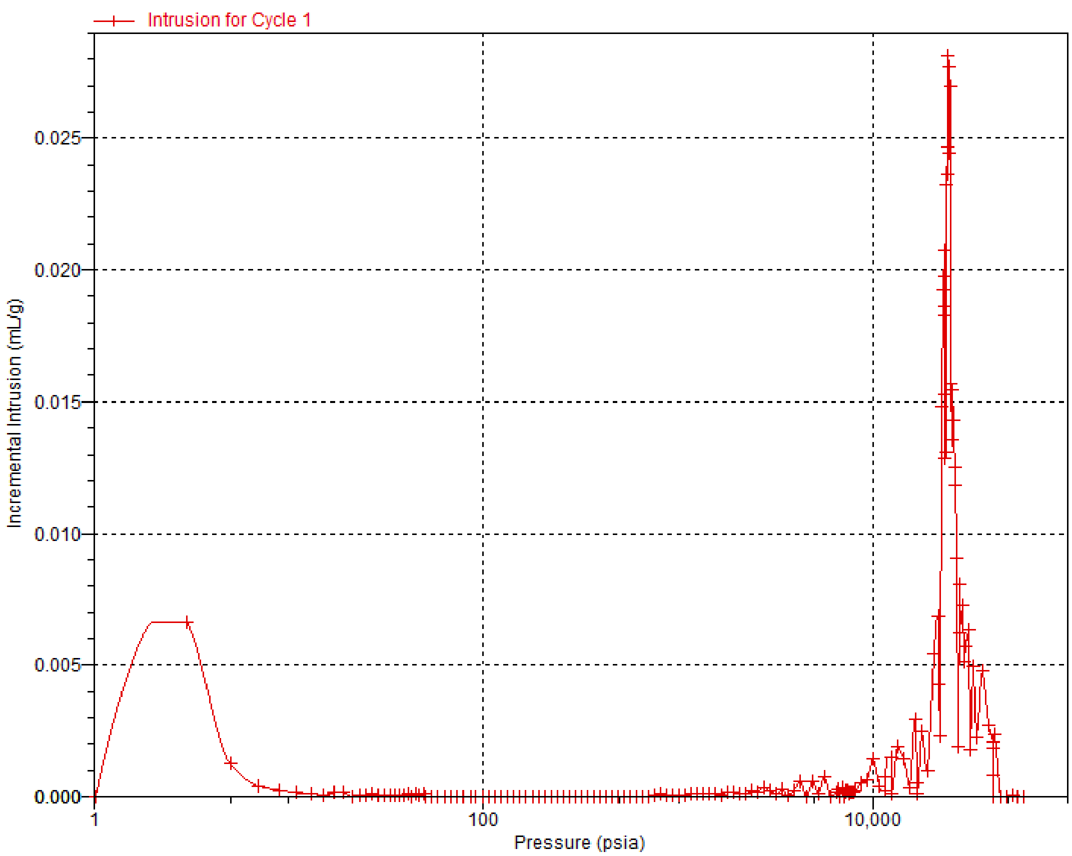
MicroActive AutoPore V 9600

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

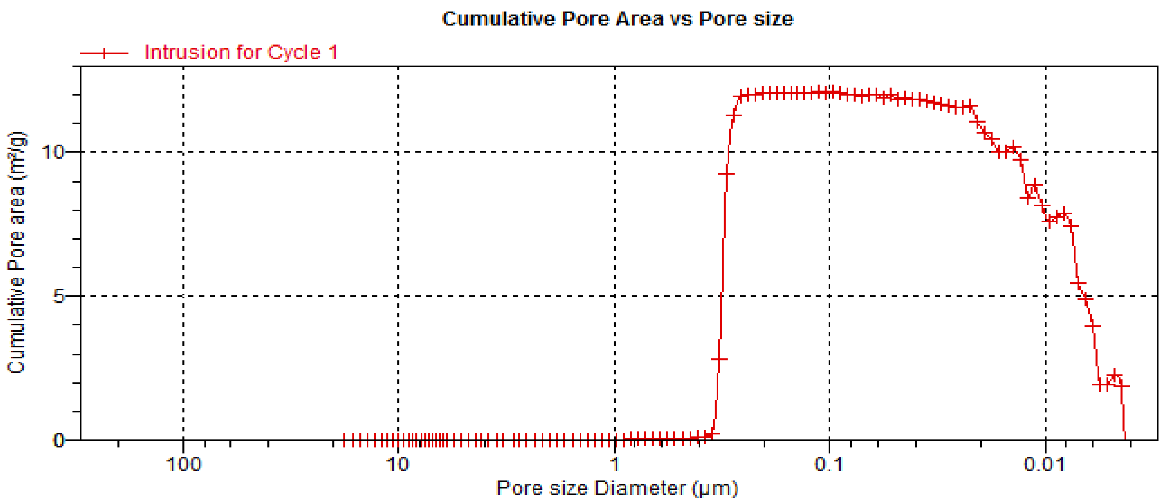
CONTROLLED PORE GLASS

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 °		



7 Selected Reports

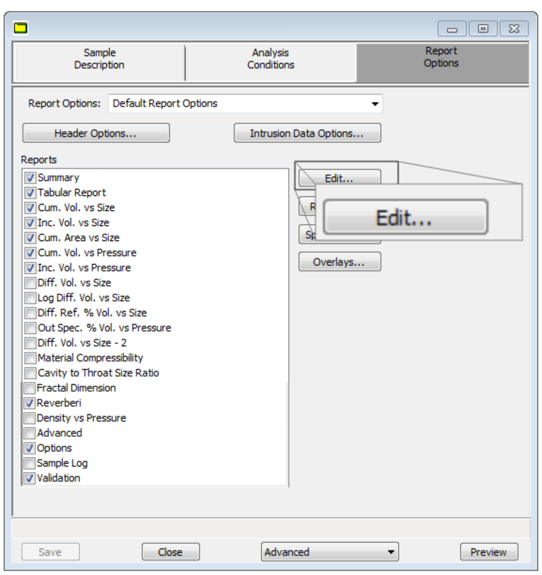
To edit reports, open the *Sample* file then select the *Report Options* tab. Highlight the report name in the *Selected Reports* list box and click **Edit**.

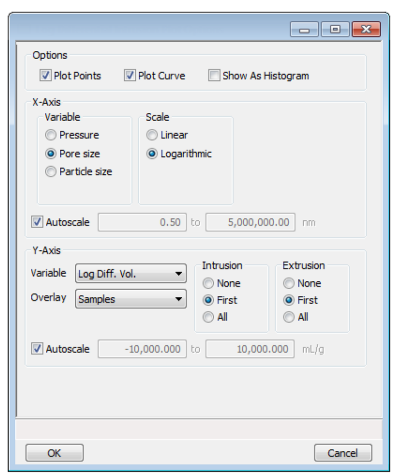
Advanced Reports

Advanced Reports - Python Module on page A - 1

Graph Report Options

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



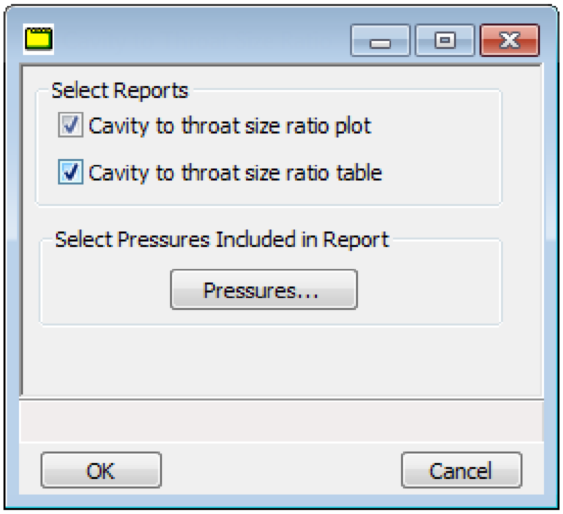


Graph Reports

Selections	Description
Autoscale [*check box*]	When enabled on the report parameters windows, allows the x- and y- axes to be scaled automatically. *Autoscale* means that the x- and y- ranges will be set so that all the data is shown. If Autoscale is not selected, the entered range is used.
Options [*check box*]	**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*]	Select sample files that contain data to be overlayed onto the selected plot.
Reference [*button*]	Select a sample file to compare analysis results of the current sample.
Specification [*button*]	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 [*group box*]	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 *Common Fields and Buttons on page 2 - 2*.

CAVITY TO THROAT SIZE RATIO REPORT OPTIONS



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

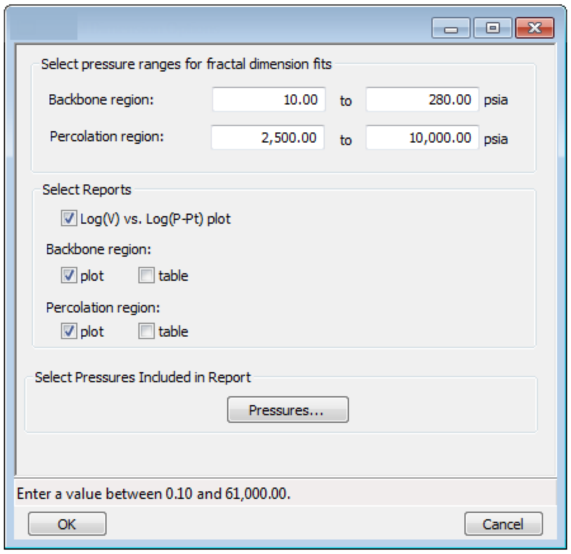
Cavity to Throat Size Ratio Reports

Selections	Description
Cavity to throat size ratio plot [*check box*]	Displays the ratio plot.
Cavity to throat size ratio table [*check box*]	Displays the ratio table.
Pressures [*button*]	Enter the minimum and maximum calculation pressure range.

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

FRACTAL DIMENSION REPORT OPTIONS

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



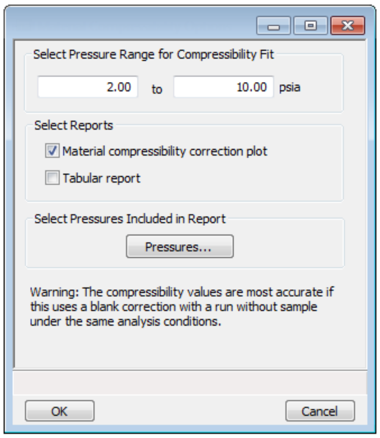
Fractal Dimension Reports

Selections	Description
Backbone region [*text box*]	Enter the pressure at which the calculations are to be performed.
Percolation region [*text box*]	Enter the pressure at which the calculations are to be performed.
Pressures [*button*]	Select to enter minimum and maximum pressures.
Select Reports [*check box*]	**Show Log(V) vs Log (P-Pt) graph.** Select to generate an additional graph to help select linear range for calculations. **Backbone region.** Select the type of report to generate. **Percolation region**. Select the type of report to generate.

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

MATERIAL COMPRESSIBILITY REPORT OPTIONS

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

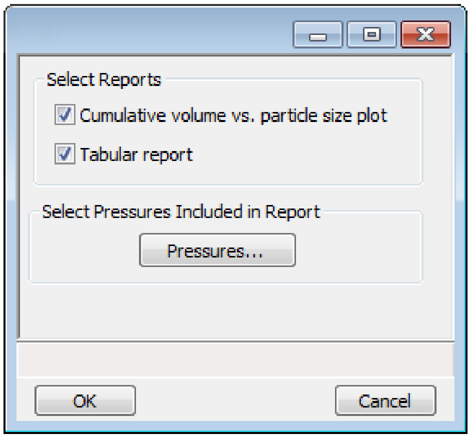


Material Compressibility Reports

Selections	Description
Pressures [*button*]	Enter the calculation pressure range.
Range [*text box*]	**P1.** Enter the beginning pressure. **P2.** Enter the ending pressure.
Select Reports [*group box*]	**Material compressibility correction plot.** Displays the results in graph format. The graph plots pressure on a log scale on the x-axis and the volume compressed readings as points on the y-axis, with the theoretical curve based on the calculated values overlaid. **Tabular report**. Displays the results in table format. The table displays pressure, volume compressed, predicted volume compressed, and error.

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

MAYER-STOWE REPORT OPTIONS



Mayer-Stowe Reports

Selections	Description
Cumulative volume vs particle size plot [*check box*]	Select to show the ratio plot.
Pressures [*button*]	Select to enter the minimum and maximum calculation pressure range.
Tabular report [*check box*]	Select to show the ratio table

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

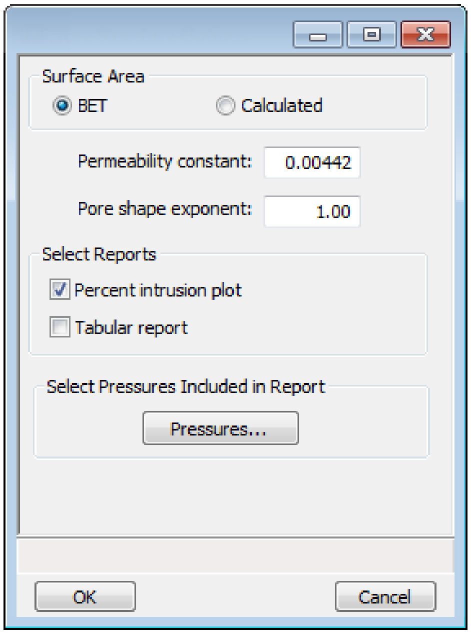
OPTIONS REPORT

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

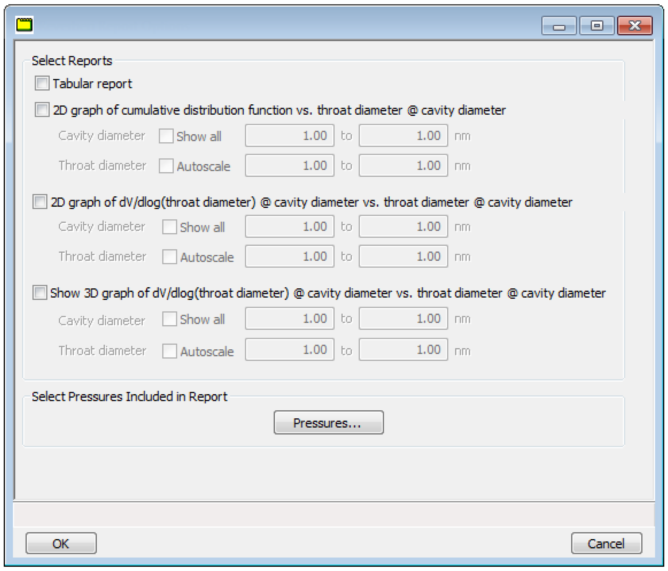


Pore Structure Reports

Selections	Description
Permeability constant [*text box*]	Viewed on Report Summary.
Pore shape exponent [*text box*]	Viewed on Report Summary.
Pressures [*button*]	Select to enter minimum and maximum pressures.
Select Reports [*group box*]	Select the report type.
Surface Area [*group box*]	Select the type of Surface Area to reference.

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

REVERBERI REPORT OPTIONS



Reverberi Reports

Selections	Description
Cavity diameter [*check box*]	Select *Show all* to display all diameters or enter a specific range to display.
Show 2D graph of cumulative distribution ... [*check box*]	Displays a 2D graph with this description.
Show 2D graph of dV/d-log (throat diameter) ... [*check box*]	Displays a 2D graph with this description.
Show 3D graph of dV/d-log (throat diameter) ... [*check box*]	Displays a 3D graph with this description.
Show table [*check box*]	Displays a table in the report.
Throat diameter [*check box*]	Select *Autoscale* to autoscale the throat diameter or enter a specific range to display.

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

Sample Log Report

Sample Log reports cannot be edited.

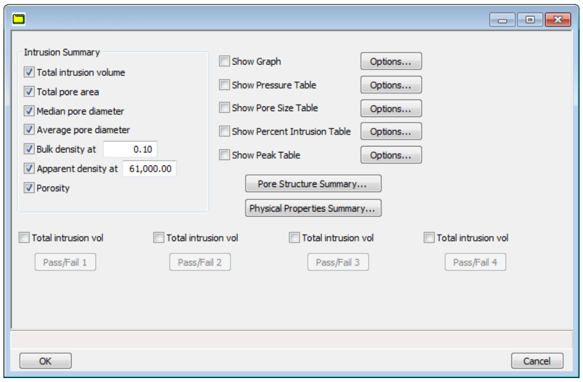
Inserts a log of sample operations in the reports.

This report provides information on:

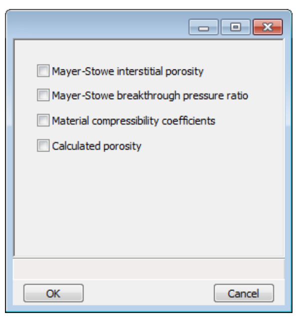
- Manual control operations performed during analysis.
- Information entered using *Add Log Entry* on the sample file editor.
- Warnings and/or errors which occurred during analysis.

SUMMARY REPORT

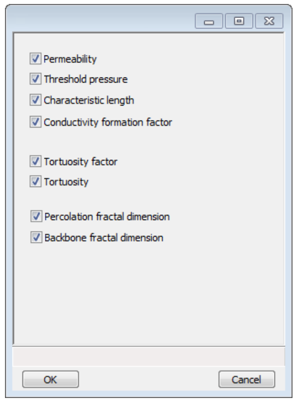
The *Summary Report* provides a condensed listing of selected data results.



Summary Report

Selections	**Description**
Intrusion Summary [*group box*]	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 [*button*]	Select the physical properties to display on the report. 

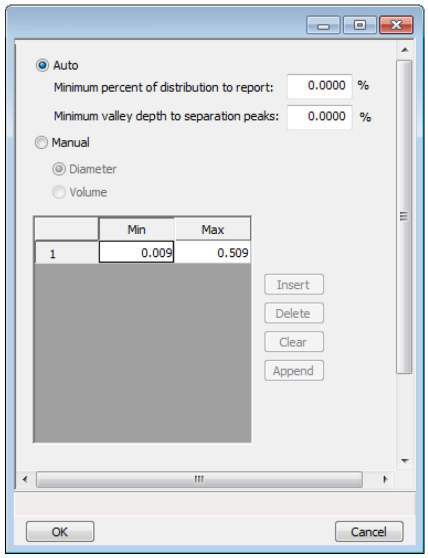
Summary Report (continued)

<table>
<tr><th>Selections</th><th>Description</th></tr>
<tr><td>Pore Structure Summary [button]</td><td>Select the pore structure to be included in the report.
</td></tr>
<tr><td>Show Graph [check box]</td><td>Displays the report in graph format. Click Options to select how the graph should display.

See Report Options on page 4 - 12 for a description of fields and buttons on this window.</td></tr>
</table>

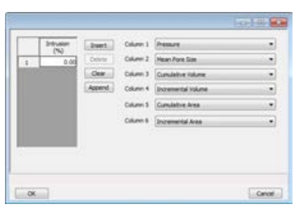
Summary Report (continued)

<table>
<tr><th>Selections</th><th>Description</th></tr>
<tr><td>Show Peak Table
[check box]</td><td>Displays the peak table in the report. Click Options to select how the graph should display.

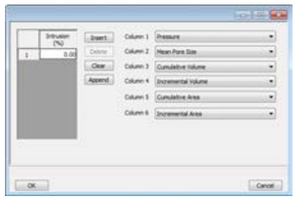
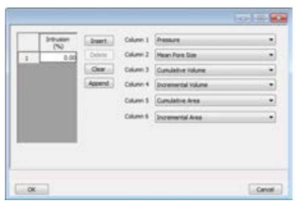
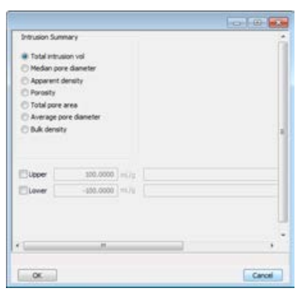


Auto. Select to have the system automatically identify peaks based on the entered minimum valley depth to separation peaks. Enter the minimum settings.

Manual. Select to manually enter the minimum and maximum diameter or volume for each peak in the table.</td></tr>
<tr><td>Show Percent Intrusion
[table]</td><td>Click Options to enter the percentile intrusion for the report.

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

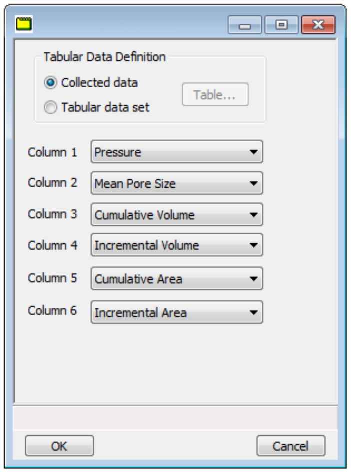
Summary Report (continued)

Selections	Description
Show Pore Size Table [*check box*]	Select to show pore size in the report. Click **Options** to select points to display. 
Show Pressure Table [*check box*]	Select to display pressure points in the report. Click **Options** to select points to display. Use the drop-down fields to specify the data to appear in the specified columns for report generation. 
Total intrusion volume [*check box*]	Enables the **Pass/Fail** button.  **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.

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

TABULAR REPORT OPTIONS

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



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

Tabular Reports

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

Tabular Reports (continued)

Selections	Description
	 ■ **Data Set**. Select either *Pressure*, *Pore size*, or *Percent intrusion* as the data set. Click **Insert** to insert a data point immediately before the selected point. To complete the table quickly, enter the highest value in the set, then click **Insert** to enter points below that value.

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

8 Diagnostics

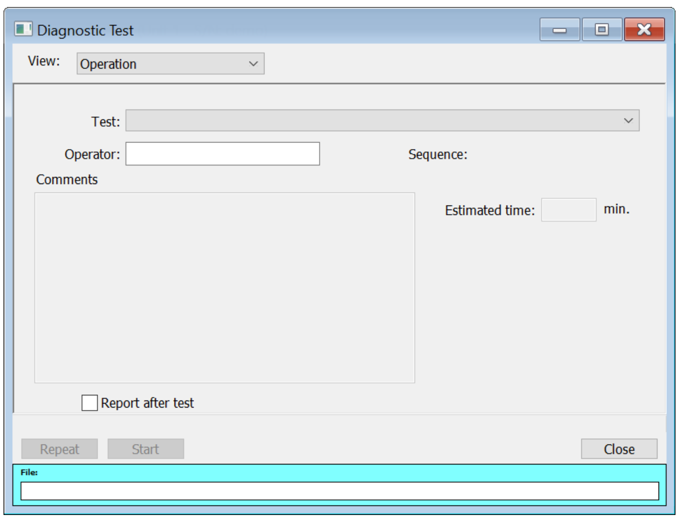
Unit [n] > Diagnostics

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

Start Diagnostic Test

Unit [n] > Diagnostics > Start Diagnostic Test

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



Start Diagnostic Test

Selections	Description
Comments [*text box*]	Displays comments from the selected diagnostic test.
Estimated time (min.) [*text box*]	Approximate time for test completion.
File [*group box*]	Shows a status bar of steps complete once the test begins.
Next [*button*]	Starts the next test.
Operator [*text box*]	Enter information to identify the person running the service test.
Repeat [*button*]	Repeats the selected diagnostic test.

Start Diagnostic Test (continued)

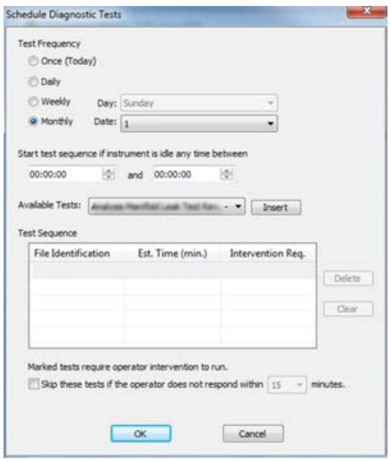
Selections	Description
Report after test [*check box*]	Automatically generates reports to the selected destination when the test is complete.
Sequence	Sequence number assigned to the test.
Start [*button*]	Starts the diagnostic test.
Test [*drop-down box*]	Select the diagnostic test to be performed.

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

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

Selections	Description
Available Tests [*drop-down box*]	Select one or more tests to run unattended.
Insert [*button*]	Inserts the selected test in the *Available Tests* drop-down list.
Skip these tests if the operator does not respond within [*n*] minutes [*check box*]	Check this option if any test requiring operator intervention should be omitted if the operator does not respond within the specified time.

Schedule Diagnostics Test Frequency (continued)

Selections	Description
Start test sequence if instrument is idle any time between 00:00:00 and 00:00:00 [*text box***]**	Enter a from and to time for an unattended test to begin if the instrument is idle at any time during the entered time frame.
Test Frequency [*selection***]**	Select how often the test is to run unattended.
Test Sequence [*group box***]**	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**. 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**.

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

DIAGNOSTIC TEST REPORT

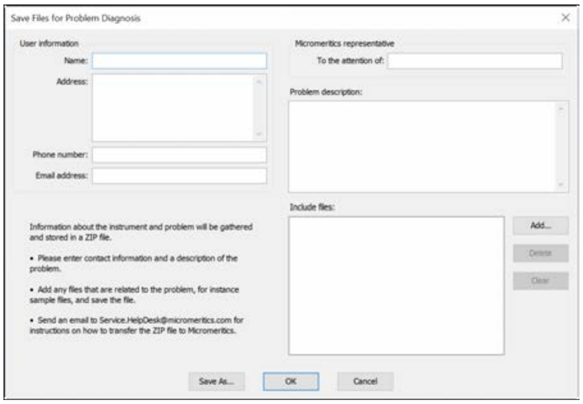
Unit [n] > Diagnostics > Diagnostic Test Report

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

SAVE FILES FOR PROBLEM DIAGNOSIS

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

Use to compress pertinent diagnostic information into a single zip file. This file can be sent to a Micromeritics Service Representative for problem resolution.



1. Complete the form. A default file named *Diagnostics-[date].zip* is created unless another file name is specified.
2. Add any files that are related to the problem — such as sample files — and save the file.
3. Send an email to Service.Helpdesk@Micromeritics.com for instructions on how to transfer the .ZIP files to Micromeritics.

Save Files for Problem Diagnostics

Selections	Description
Problem description [*text box***]**	Enter information that would be helpful to the Micromeritics representative.
Include Files [*button***]**	■ **Add.** Click to select additional files to send with this problem diagnosis. ■ **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 [*button***]**	Click to specify the name and location of the compressed file. Make a note of the file name and location. This file will need to be sent to your Micromeritics representative for problem resolution.
Micromeritics representative [*text box***]**	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).

Save Files for Problem Diagnostics (continued)

Selections	Description
User Information **[***text box***]**	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 ***Common Fields and Buttons on page 2 - 2*****.**

9 Calibration

Unit [n] > Calibration

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

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

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

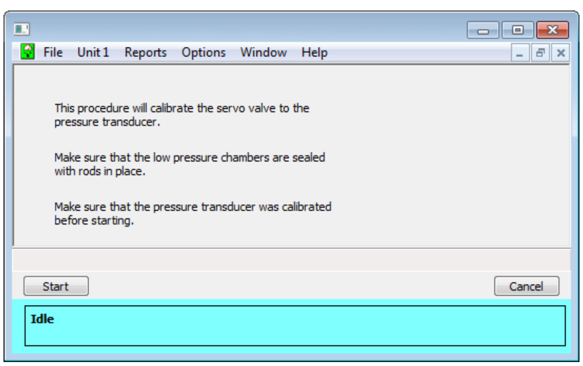
Generally, it will not be necessary to change the data in the calibration file. However, if a condition occurs during the operational verification that requires changes to the calibration data, changes should be saved in a file. Calibration data files are retained in the analyzer history file and can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

Calibrate the Low Pressure Servo

Unit [n] > Calibration > Low Pressure Servo

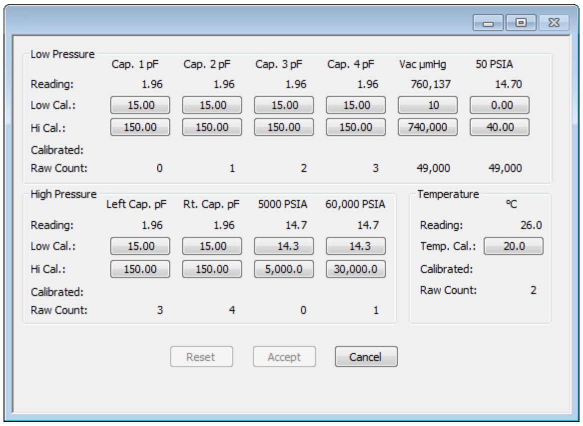
Ensure the pressure transducer was calibrated by a service technician before starting this process.

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



CALIBRATE SIGNALS

Unit [n] > Calibration > Signals



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

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

Calibration Signals

Selections	Description
Low Pressure	**Reading.** Displays the current values from each capacitance detector. **Low Cal**. Displays the capacitance of the reference device with the lower value. **Hi Cal.** Displays the capacitance of the reference device with the higher value. **Calibrated.** The date the capacitance detector was last calibrated. **Raw Count**. The digital value produced by the analyzer for each capacitance detector. **Vac µmHg.** ■ **Reading.** The current value from the vacuum gauge. ■ **Low Cal.** The pressure value read on a reference vacuum gauge after vacuum is achieved. ■ **High Cal.** The pressure value read on a reference vacuum gauge

Calibration Signals (continued)

Selections	Description
	when higher pressure is applied. ■ **Calibrated.** The date the vacuum gauge was last calibrated. ■ **Raw Count.** The digital value produced by the analyzer for the vacuum gauge. **50 PSIA** ■ **Reading.** The current value from the 50 psia transducer. ■ **Low Cal.** The pressure value read on a reference transducer after vacuum is achieved. ■ **High Cal.** The pressure value read on a reference transducer when higher pressure is applied. ■ **Calibrated.** The date the 50 psia transducer was last calibrated. ■ **Raw Count.** The digital value produced by the analyzer for the 50 psia transducer .
High Pressure	**Reading.** Displays the current values for the left and right capacitance detectors. **Low Cal**. Displays the left and right capacitance detectors of the reference device with the lower value. **Hi Cal.** Displays the capacitance of the left and right capacitance detectors with the higher value. **Calibrated.** The date the left and right capacitance detectors were last calibrated. **Raw Count**. The digital value produced by the analyzer for left and right capacitance detectors.
Temperature	**Reading**. The transducer's current temperature. **Temp Cal**. The calibration setting for the transducer. Click to reset the temperature. **Calibrated.** The date the transducer was last calibrated. **Raw Count**. The digital value produced by the analyzer for the transducers.

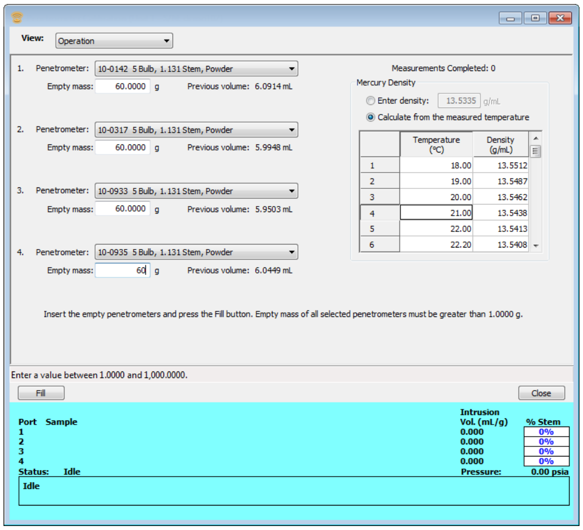
For fields and buttons not listed in this table; see ***Common Fields and Buttons on page 2 - 2***.

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.



1. 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. To create a new *Penetrometer Properties* file, click **Browse** from the drop-down list and enter a new file name.
2. 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.
3. 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.

4. Prepare and load the penetrometers as for a low pressure analysis. 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.

5. 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.

LOAD CALIBRATION FROM FILE

Use to load a previously saved calibration file.

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

Changing the calibration may affect the analyzer’s performance.

SAVE CALIBRATION TO FILE

Use to save the current calibration settings to a backup file which can later be reloaded using the 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	Analyzer serial number
2013-04-25	Date the calibration file was saved
.CAL	File name extension

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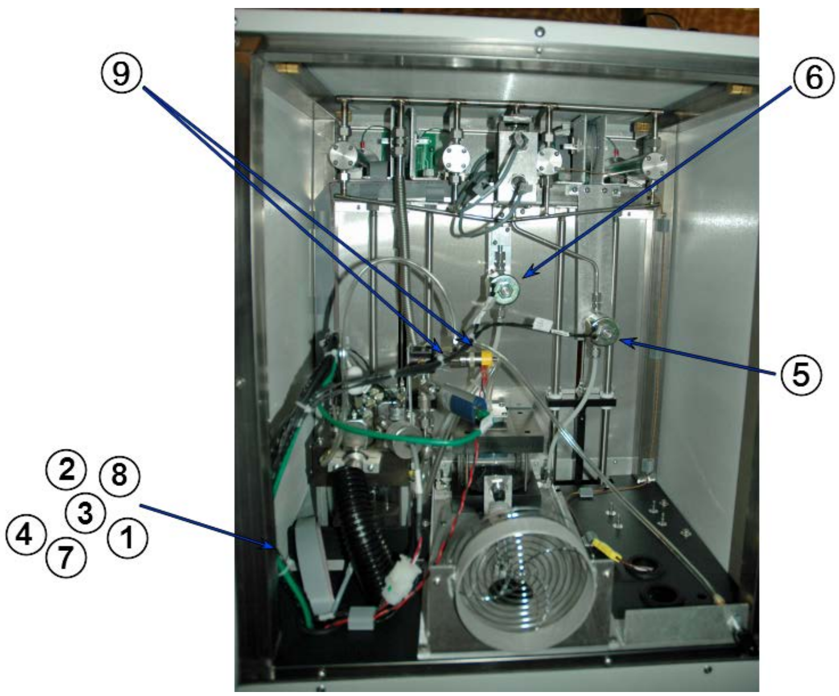
10 Hardware Components and Accessory Installation

System Components

Low Pressure System Components



Back view

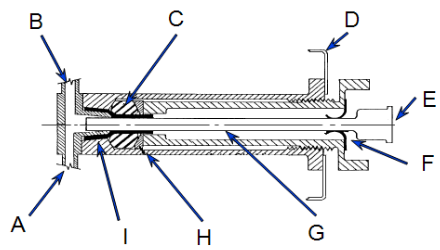
Low Pressure System Valve Locations

Valve	Description
Servo isolation valve [1]	Ensures no leaking occurs through the servo. It is open when the servo is in use and closed when the servo is off.
Fast evacuation valve [2]	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.

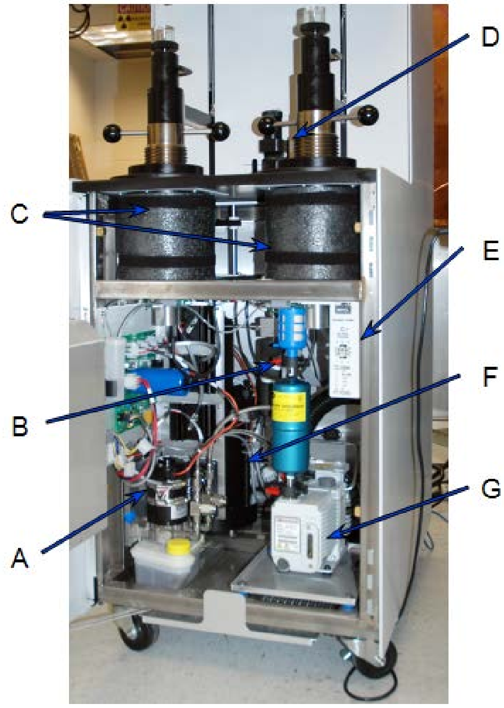
Low Pressure System Valve Locations (continued)

Valve	Description
Vacuum valve [3]	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).
Gas inlet valve [4]	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.
Mercury fill valve [5]	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.
Drain valves [6]	See Mercury fill valve [5].
Mercury reservoir evacuation valve [7]	Connects the vacuum line to the mercury reservoir.
Evacuation reservoir valve [8]	See Vacuum valve [3].
Vent valve [9]	If a cap detector is removed when the pressure is above atmospheric pressure, the low pressure system will be vented to atmosphere with this valve.

Low Pressure System Valves

Component	Description
Low pressure ports	A sample encased in a penetrometer is first evacuated, filled with mercury, then pressurized to between 15 and 50 psia.  A. Mercury B. Vacuum C. Soft gum rubber D. Front panel E. Penetrometer F. Contact G. Metal cladding H. Thrust I. Rubber tube 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.

HIGH PRESSURE SYSTEM COMPONENTS



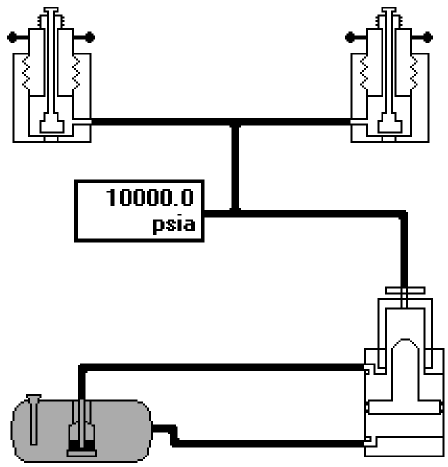
Front View

A. Hydraulic pump
B. Transducer
C. High pressure chambers
D. Mercury storage reservoir
E. Diagnostic panel
F. Pressure intensifier
G. Vacuum pump

High Pressure System Components

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

High Pressure System Components (continued)

Component	Description
	
Hydraulic pump	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 trans- ducer. The signal from this transducer is processed by electronic cir- cuitry to yield two pressure output signals.

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.

A. Penetrometer
B. Vent valve
C. High pressure plug

1. Turn the vent valve slowly counter-clockwise to release excess pressure.
2. Unscrew the chamber plug assembly by turning the arms counter-clockwise. Lift the chamber plug assembly as far as it will go. The chamber assembly contains a latching device which automatically locks into place when the assembly is in the topmost position.
3. Gently lower the penetrometer assembly, bulb down, into the chamber until it rests firmly on the banana plug.
4. Verify that the oil in the high pressure chamber just covers the ledge inside the chamber. Add more oil, if needed.

5. Pull the latch release down to unlock the chamber plug assembly. Gently lower the assembly while ensuring that the penetrometer is inserted into the assembly hole. After making sure the penetrometer goes into the hole, lower the assembly the rest of the way and screw the chamber plug in.

Latch release

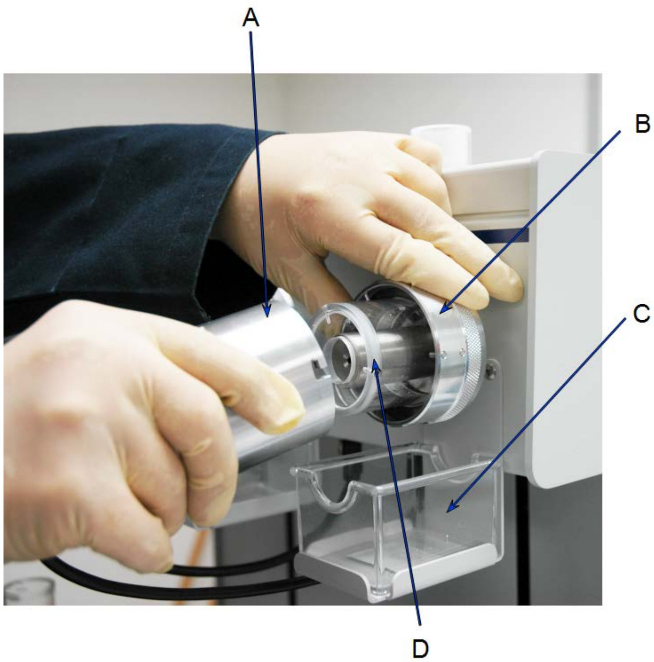
6. After the chamber plug is screwed, check the fluid level in the plastic cup above the vent valve. to ensure all the air bubbles are out of the chamber, and sealing the vent valve.

Install a Penetrometer in a Low Pressure Port

If fewer than four samples are to be analyzed, a steel rod with a receptacle for the cap detector must be installed in each unused low pressure port. The cap detector must see that a rod is installed.

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

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

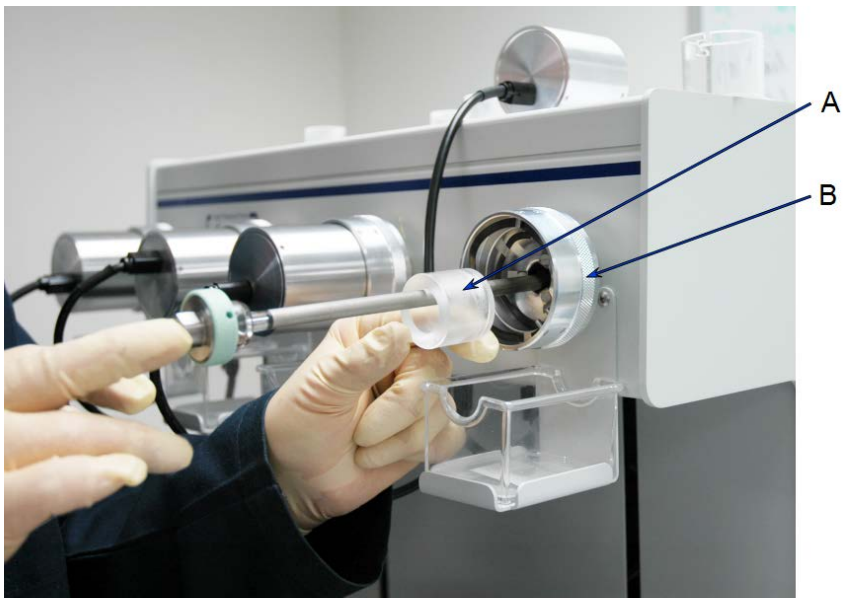


A. Capacitance detector
B. Retaining knob
C. Mercury spill tray
D. Plastic cap

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, remove the penetrometer from the low pressure port.
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.



A. Spacer is required only for the 3 cc penetrometer
B. Retaining knob

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.

A. Penetrometer
B. Vent valve
C. High pressure plug

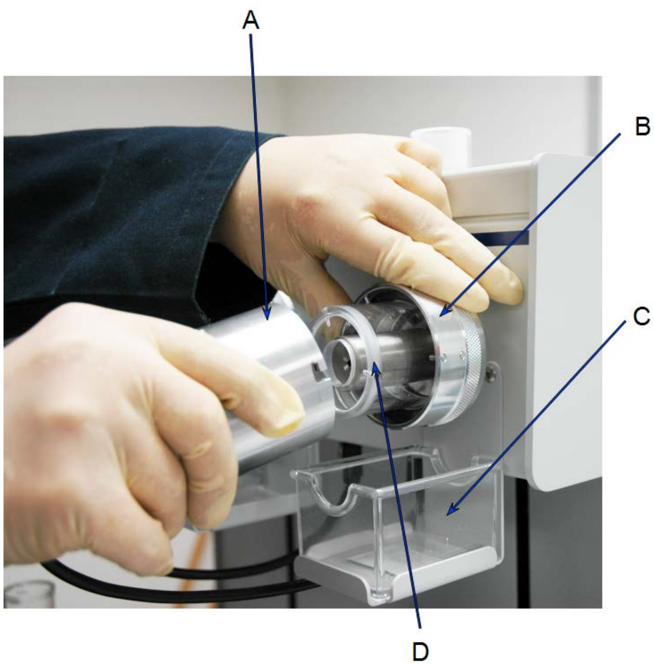
2. Unscrew the chamber plug by turning the arms counter-clockwise. Lift the plug assembly as far as it will go. Fluid begins to drain from the vent valve. Pause a few seconds to allow fluid to drain back into the chamber.
3. Remove the penetrometer assembly. Hold it over the chamber for a few moments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

REMOVE A PENETROMETER FROM A LOW PRESSURE PORT

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

Removing penetrometers when the *Hg Drained* indicator is not illuminated may allow mercury to spill from the port.

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



A. Capacitance detector
B. Retaining knob
C. Mercury spill tray
D. Plastic cap

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. The rack for full penetrometers [*part number 962-25827-00*] can be used for this purpose.

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.

11 Maintenance and Troubleshooting

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

The following can be found on the Micromeritics web page (www.Micromeritics.com).

- Error Messages document (PDF)
- Parts and Accessories
- Vacuum Pump Guide (PDF)

Improper handling, disposing of, or transporting potentially hazardous materials can cause serious bodily harm or damage to the instrument. Always refer to the MSDS when handling hazardous materials. Safe operation and handling of the instrument, supplies, and accessories is the responsibility of the operator.

Do not modify this instrument without the authorization of Micromeritics Service Personnel.

When lifting or relocating the instrument, use proper lifting and transporting devices for heavy instruments. Ensure that sufficient personnel are available to assist in moving the instrument. The AutoPore V 9600 weighs approximately 227 - 250 kg (500 - 550 lb) depending on configuration.

Use of a power cord or power supply not provided with the instrument could cause personal injury or damage to the equipment. If a replacement is needed, contact your Micromeritics Service Representative. Detachable power supply cords with an inadequate rating could cause significant instrument damage or physical harm.

Do not add anything between the power cord and the power source that would compromise the earth ground.

Do not remove or disable the grounding prong on the instrument power cord.

If the equipment needs to be relocated, check with your Micromeritics service representative. The equipment must be positioned such that the mains supply is not obstructed and is easily accessible to disconnect the equipment from the AC main power supply.

> **Warning:** Prior to moving the instrument, disconnect and remove all glassware from the instrument. Ensure all gas shut-off valves on the gas cylinder have been closed and gas lines disconnected from the instrument. Contact your Micromeritics Service Representative.

Status display is too large for computer monitor.

Cause: Monitor resolution is set below 800 × 600.

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

Difficulty attaining adequate vacuum conditions during low pressure analyses.

Cause A: Vacuum pump oil is low.

Action A: Add vacuum pump oil.

Cause B: Vacuum hose not properly connected.

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

Cause C: Sample contains excess moisture adsorbed from atmosphere.

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

> **CAUTION:** Never heat a sample that has contacted mercury.

Cause D: Proper sealing not achieved on penetrometers

Action D:

1. Use blank rods to test the ports to eliminate the possibility of having a leaking penetrometer cap / bulb seal.
2. Lightly grease the rods with a high grade vacuum grease, then insert the rods in the low pressure ports.
3. Tighten the retaining knobs on the low pressure ports. If vacuum conditions are satisfactory, check the penetrometer for scratches or chips in the bulb.

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

Action E:

1. Polish the lip of the bulb and the cap using 600 grit emery paper or crocus cloth.
2. Place the paper or cloth on a flat surface, grit-side up.

3. Clean all grease from the surface to be polished with solvent.
4. Lightly press the surface down on the grit and rub in a circular motion. A minute of this polishing action is usually sufficient, but it must be continued until the surface is free from flaws. Examination of the surface under low magnification (approximately 20X) helps determine when the surface is free from flaws.
5. Clean the ground surface before regreasing.

Cause F: Leaking valves.

Action F: Check the valves for leaks. See ***Check Valves for Leaks on page 11 - 28***.

Cause G: Moisture has accumulated in the system.

Action G: Remove the accumulated moisture. See ***Remove Moisture from the Analyzer on page 11 - 31***.

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

Cause A: Indicator is not working properly.

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

If the status display window shows that the mercury has overfilled (the alarm state), see ***Handling Mercury Overfill on page 11 - 23***. If the status display shows that the mercury degasser is either filled or partially filled, see ***Drain the Low Pressure System on page 11 - 8***.

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

Action B: See ***Drain the Low Pressure System on page 11 - 8***.

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

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

Action: See ***Handling Mercury Overfill on page 11 - 23***.

Mercury warning buzzer sounds with continuous tone.

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

Action: Drain excess mercury into the reservoir. See ***Handling Mercury Overfill on page 11 - 23***. Remove the plug extending down from the mercury trap. The plug is reached through the front door. Facing the analyzer, the plug is in the upper back right corner. See ***Dish for Spilled Mercury on page 11 - 18***. Position a container beneath the trap before removing the plug.

The high pressure system failed to attain the specified pressure.

Cause: Low fluid level in the hydraulic pump.

Action: Add fluid to the hydraulic pump. See ***Maintain Hydraulic Pump Fluid Level on page 11 - 26***.

High pressure system failed to retain a reasonably constant pressure.

Cause: Leakage around the high pressure chamber cap.

Action: Replace the cup seals and backup rings on the high pressure chamber cap. See ***Replace Chamber Cup Seals on page 11 - 24***.

Cannot attain low pressure data points above atmospheric pressure.

Cause: Gas supply pressure too low.

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

SAFE SERVICING

Do not modify this instrument without the authorization of Micromeritics Service Personnel.

To ensure safe servicing and continued safety of the instrument after servicing, service personnel should be aware of the following risks:

Product specific risks that may affect service personnel:

- **Electrical**. Servicing or repair could require opening the outer panels and exposing energized electrical components.
- **Fuses.** Only use fuses rated as:

Power Source	Required Fuses
100-120V	4.0A, Slo-Blo, AG
220-240V *	2.0A, Slo-Blo, 5x20mm

* 220V applications should use the 230V setting.

- Understand how to use the Mercury Vapor Kit installed in the front panel of the AutoPore.

Protective measures for these risks:

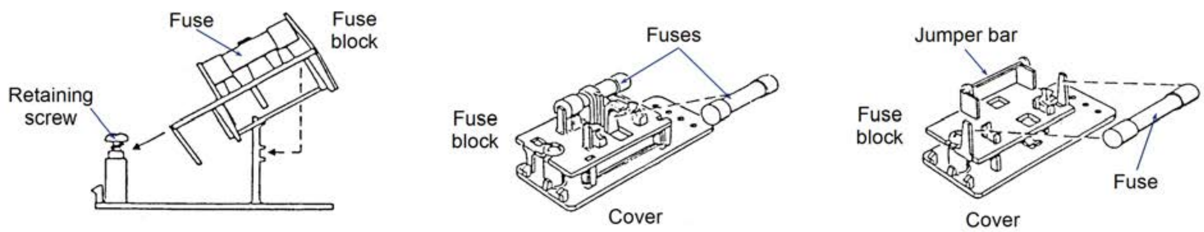
- **Electrical**. The electrical components operate at low voltage (24V or less) and pose low risk when energized. Maintenance, troubleshooting, and repairs should be performed with the instrument de-energized whenever possible, in accordance with standard electrical safety guidelines.
- **Fuses**. Use of improperly rated fuses could cause damage to the equipment.
- Power off and unplug the analyzer from the power outlet prior to servicing.

Verification of the safe state of the instrument after repair:

- The low pressure and high pressure systems readings must be at atmospheric pressure.
- The steel rods must be inserted inside each low pressure port with the cap detectors in place.
- The high pressure chambers must be opened.

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.

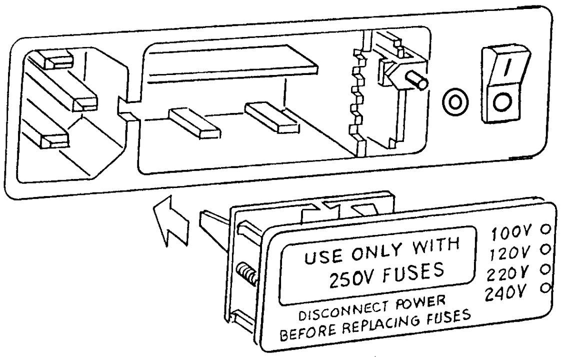
1. Reposition the fuse block, if necessary:
    a. Remove the fuse block retaining screw.
    b. Lift the fuse block from the cover.
    c. Rotate the fuse block.
    d. Mount the fuse block to the cover.
    e. Replace the retaining screw.
2. Insert appropriate fuse for the input power source. Refer 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	Required Fuses
100-120V	4.0A, Slo-Blo, AG
220-240V *	2.0A, Slo-Blo, 5x20mm

* 220V applications should use the 230V setting.

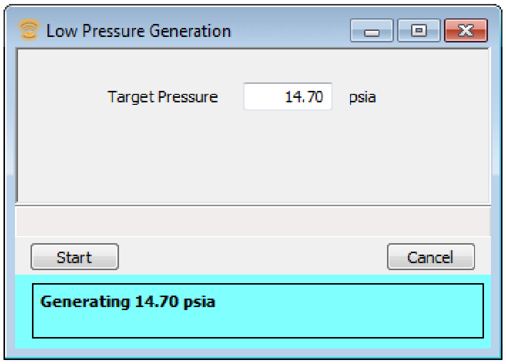
3. Insert fuse block and cover assembly into input power connector and snap it into place. After the fuse block and cover assembly are in place, the position of the indicator pin shows the input power selected.

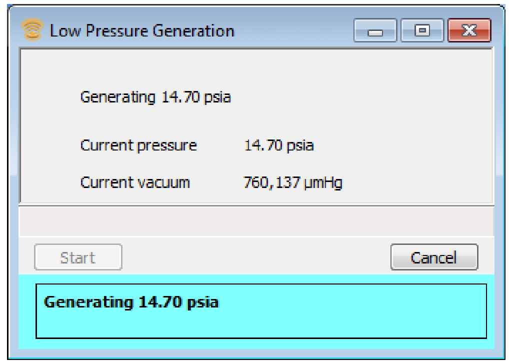


4. Connect the power cord to the analyzer and plug into an appropriate power source.

DRAIN THE LOW PRESSURE SYSTEM

Unit [n] > Generate Low Pressure



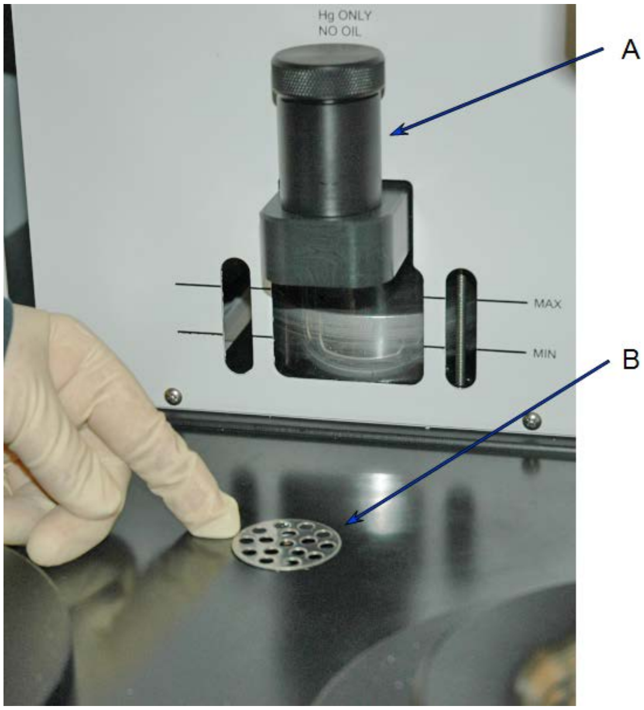


1. Specify a pressure of 10 psia. After the system reaches 10 psia and returns to *Idle* status, open the reservoir evacuation valve (#7) for 20 seconds.

   The *Hg reservoir evacuated* indicator should be displayed on the analyzer schematic. If it is not shown, do not proceed to the next step. Instead, verify that the mercury reservoir fill cap is properly sealed on top of the mercury filling funnel. 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 - 23***.

DISH FOR SPILLED MERCURY



A. Mercury fill
B. Mercury spill drain

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.

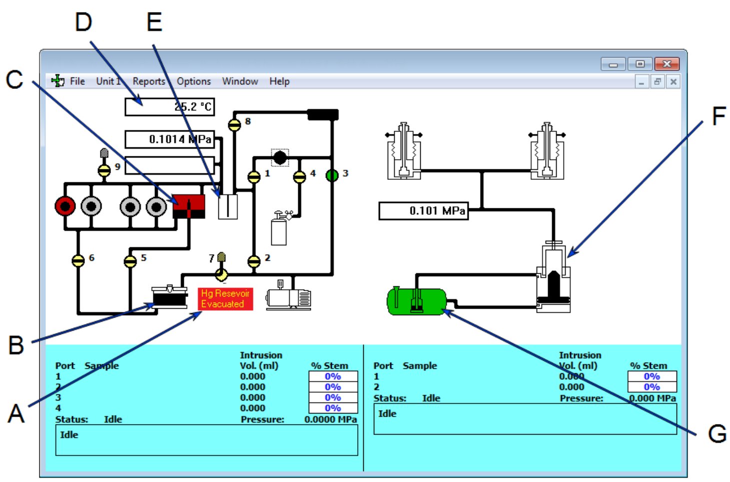
If mercury accumulates in the dish, remove it by removing the cover and extracting the mercury with the syringe accessory.

> Approximately 1/8 in.(3 mm) 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 pump components on the analyzer schematic. When this option is enabled, a checkmark appears to the left of ***Unit [n] > Enable Manual Control***. If the analyzer schematic is not immediately visible, go to ***Unit [n] > Show Instrument Schematic***.



Low Pressure

A. Hg reservoir indicator
B. Mercury reservoir
C. Mercury degasser
D. Mercury temperature sensor
E. Mercury trap

High Pressure

F. Intensifier
G. Hydraulic pump

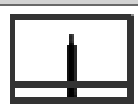
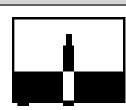
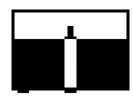
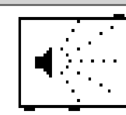
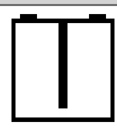
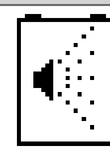
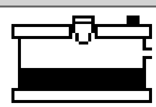
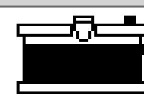
Schematic Components

Schematic Components	Description
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.

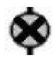
Analyzer Schematic Icons

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

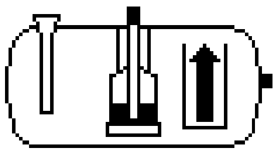
Low Pressure Schematic Icons

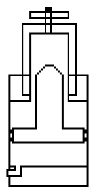
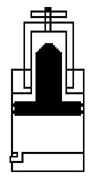
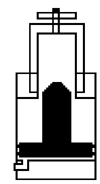
<table>
<tr><td colspan="4">Mercury Degasser. Displays the mercury level.</td></tr>
<tr><td>
Drained</td><td>
Partially Filled</td><td>
Filled</td><td>
Overfilled. This alarm displays
with a red background.
This is an alarm state.</td></tr>
<tr><td colspan="4">Mercury Trap. Displays the state of the mercury trap.</td></tr>
<tr><td colspan="2">
Empty</td><td colspan="2">
Contains more than 6 mm of mercury.
This is an alarm state.</td></tr>
<tr><td colspan="4">Mercury Reservoir. Displays the level of mercury in the reservoir.</td></tr>
<tr><td colspan="2">
If the mercury level is low, see Maintain Mercury Level on page 11 - 16.</td><td colspan="2">
Level is OK.</td></tr>
<tr><td colspan="4">Hg Reservoir Vacuum Switch</td></tr>
<tr><td colspan="2"></td><td colspan="2">When illuminated, indicates that mercury reservoir has been evacuated.</td></tr>
</table>

Low Pressure Schematic Icons (continued)

Low Pressure Ports		Indicates that the mercury fill sensor detects mercury.
		Indicates a rod and capacitance detector are present (detector reading is less than -5 pF).
		Indicates there is no mercury, but the capacitance detector is on with either a rod or a penetrometer.
		Indicates there is no capacitance detector and penetrometer or rod in place.

High Pressure Schematic Icons

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**

Low Pressure System Valve Locations

Valve	Description
Servo isolation valve [1]	Ensures no leaking occurs through the servo. It is open when the servo is in use and closed when the servo is off.
Fast evacuation valve [2]	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.
Vacuum valve [3]	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).
Gas inlet valve [4]	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.
Mercury fill valve [5]	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.
Drain valves [6]	See Mercury fill valve [5].
Mercury reservoir evacuation valve [7]	Connects the vacuum line to the mercury reservoir.
Evacuation reservoir valve [8]	See Vacuum valve [3].
Vent valve [9]	If a cap detector is removed when the pressure is above atmospheric pressure, the low pressure system will be vented to atmosphere with this valve.

PREVENTIVE MAINTENANCE

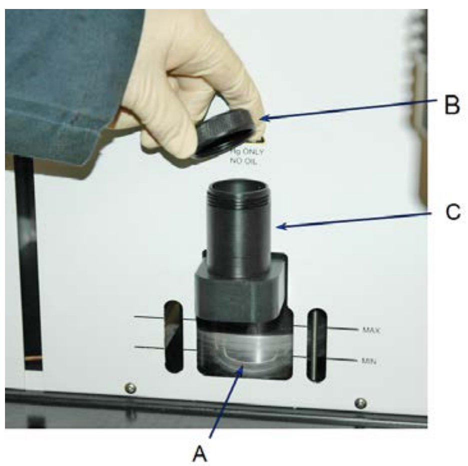
Perform the following preventive maintenance procedures to keep the analyzer operating at peak performance. Micromeritics also recommends that preventive maintenance procedures and calibration be performed by a Micromeritics Service Representative every 12 months.

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

MAINTAIN MERCURY LEVEL

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

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



A. Mercury level viewing window
B. Reservoir cap
C. Funnel for filling mercury

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

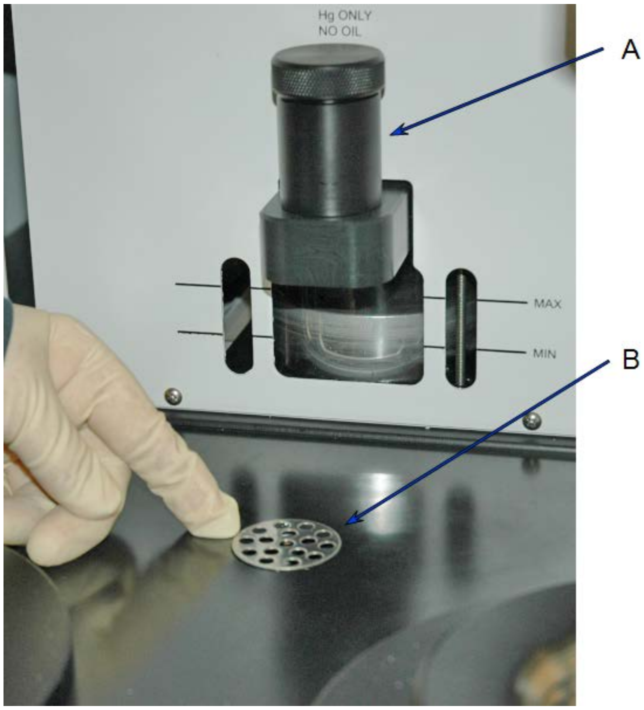
A spilled-mercury container, located in the center of the analyzer work surface behind the high pressure chambers, is provided so that any accidentally spilled mercury can be immediately brushed into it. Pour approximately 1.0 to 2.0 cc of oil into the container to prevent the mercury from vaporizing.

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

> 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.

DISH FOR SPILLED MERCURY



A. Mercury fill
B. Mercury spill drain

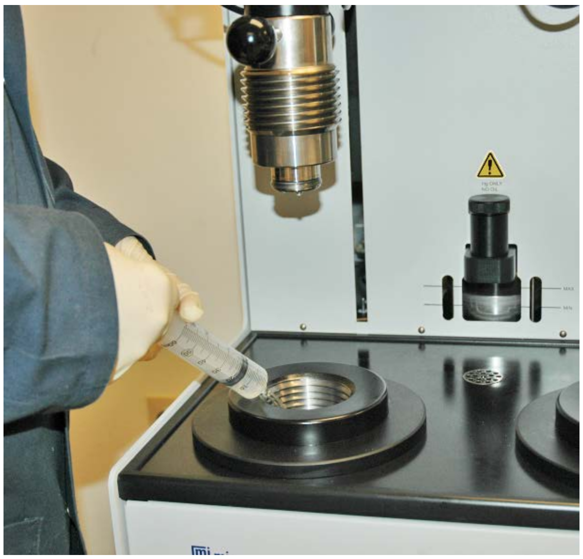
A dish for collecting mercury is located just behind the high pressure chambers. Pour approximately 1.0 to 2.0 cc of oil into the dish to prevent the mercury from vaporizing.

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

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

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.

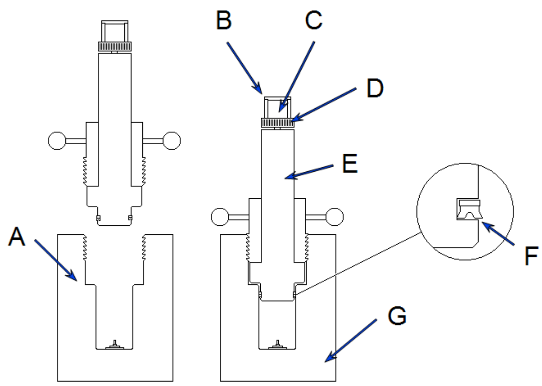


SEAL THE HIGH PRESSURE CHAMBER

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.



A. Ledge for oil level
B. Removable cap
C. Transparent cap
D. Vent valve
E. Chamber plug
F. Seal
G. High pressure chamber

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.

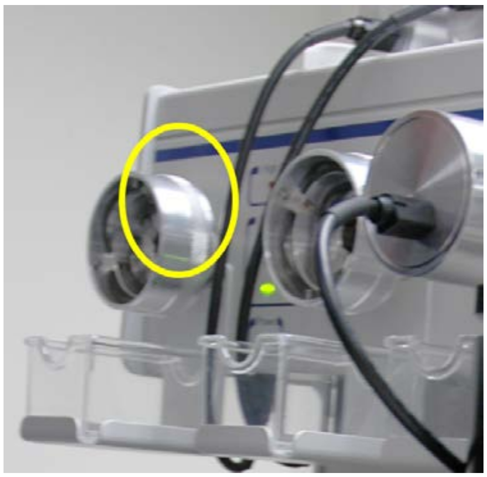
3. The cup should not be completely full. If the cup is too full, slowly open the chamber and recheck the fluid level. Fluid may need to be removed. Repeat the previous steps to release trapped air.
   - Large air bubbles may be caught in the chamber. Slowly loosen and tighten the plug approximately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

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

GREASE LOW PRESSURE PORTS

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

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



Turn retaining knob counter-clockwise to expose threads behind knob

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

HANDLING MERCURY OVERFILL

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

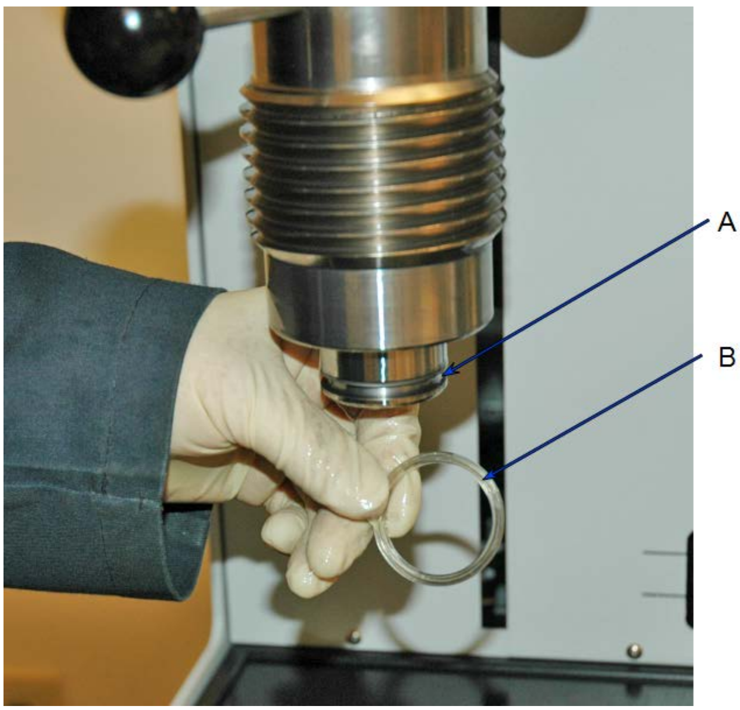
   The *Hg reservoir evacuated* indicator should be displayed on the analyzer schematic. If it is not shown, do not proceed to the next step. Instead, verify that the mercury reservoir fill cap is properly sealed on top of the mercury filling funnel. If the *Hg reservoir evacuated* indicator still is not displayed, contact a Micromeritics Service Representative.

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

REPLACE CHAMBER CUP SEALS

The chamber plug is sealed into the high pressure chamber by means of a cup seal and a backup ring. There is also an O-ring in the vent valve. 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

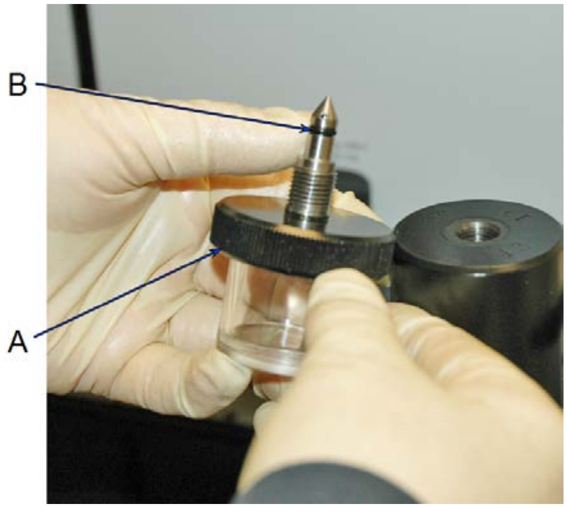
1. Remove the chamber cup seal and backup ring without scratching the surrounding surfaces.



A. Backup ring
B. Chamber cup seal

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



A. Vent valve
B. 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.

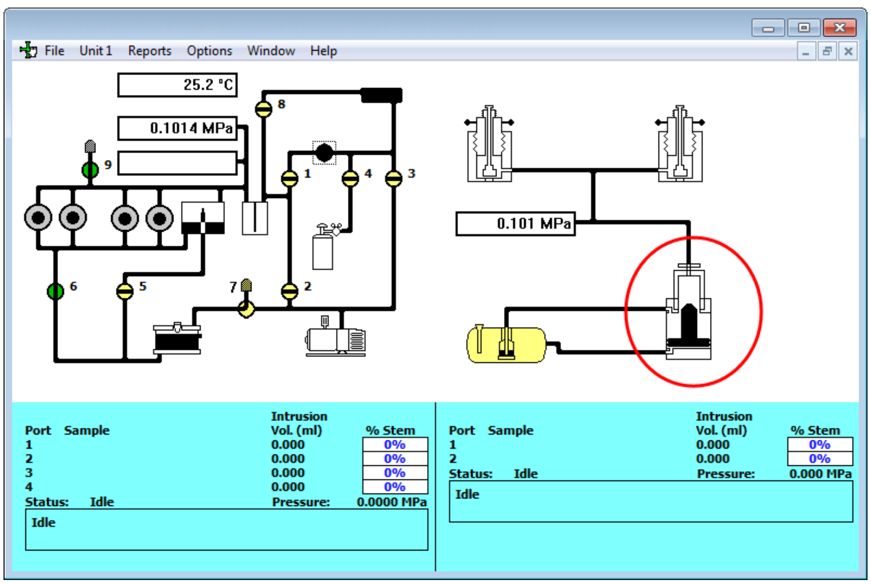
1. Remove the high pressure fluid from the high pressure chambers using the syringe provided.
2. Clean the high pressure chambers using a clean, lint-free cloth dampened with IPA.
3. When the chambers are completely dry, add new high pressure fluid.

MAINTAIN HYDRAULIC PUMP FLUID LEVEL

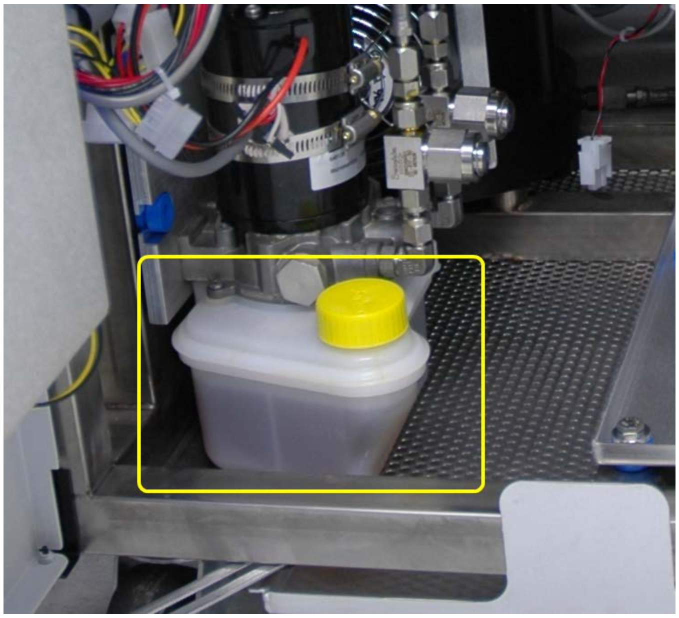
When changing the hydraulic pump fluid, use only Mobil-1 50W-50 Synthetic Motor Oil [*Micromeritics part number 004-16011-00*].

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

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



Intensifier in the DOWN position



Hydraulic fluid container

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.

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.

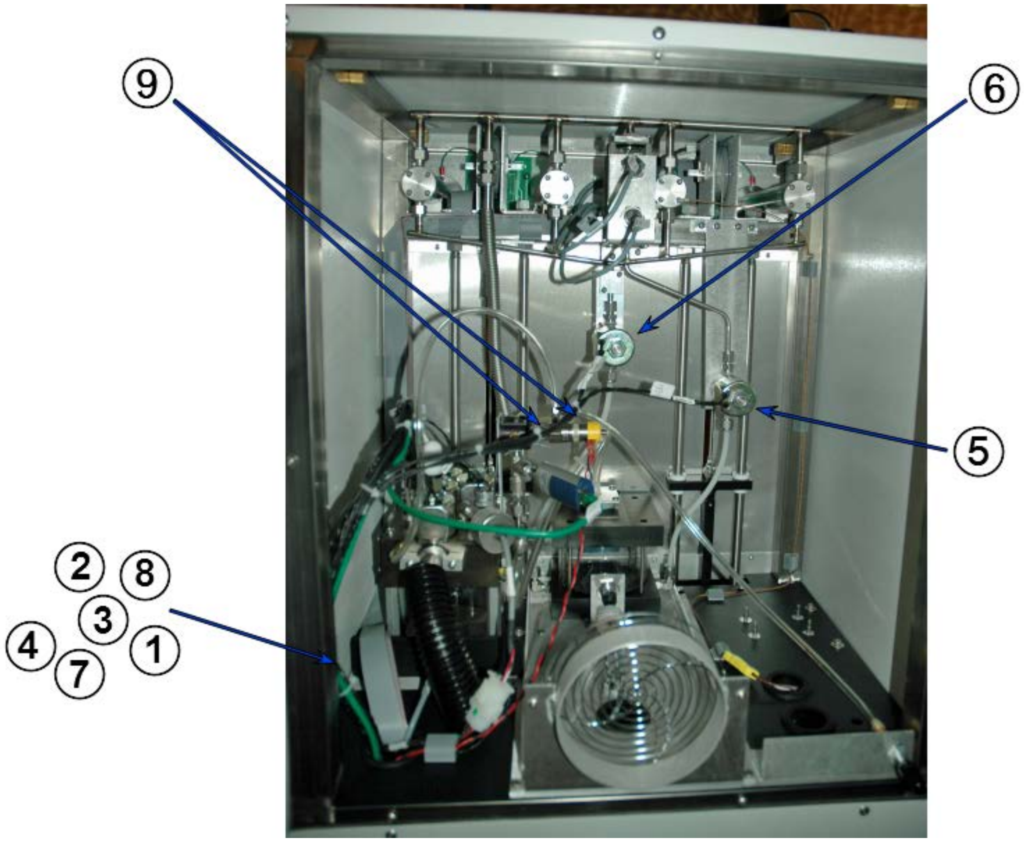
CLEAN VALVES

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

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



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.

Low Pressure System Valve Locations

Valve	Description
Servo isolation valve [1]	Ensures no leaking occurs through the servo. It is open when the servo is in use and closed when the servo is off.
Fast evacuation valve [2]	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.
Vacuum valve [3]	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).
Gas inlet valve [4]	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.
Mercury fill valve [5]	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.
Drain valves [6]	See Mercury fill valve [5].
Mercury reservoir evacuation valve [7]	Connects the vacuum line to the mercury reservoir.
Evacuation reservoir valve [8]	See Vacuum valve [3].
Vent valve [9]	If a cap detector is removed when the pressure is above atmospheric pressure, the low pressure system will be vented to atmosphere with this 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 INSTRUMENT

The exterior casing of the instrument may be cleaned using a clean, lint-free cloth dampened with isopropyl alcohol (IPA), a mild detergent, or a 3% hydrogen peroxide solution. Do not use any type of abrasive cleaner. It is not necessary to remove knobs, screws, etc. while cleaning.

Do not allow liquid to penetrate the casing of the instrument. Doing so could result in damage to the unit.

REMOVE MOISTURE FROM THE ANALYZER

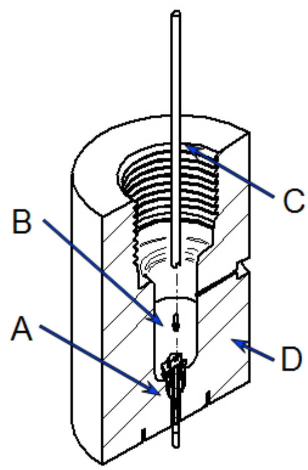
A difficult-to-detect vacuum problem arises if moisture is allowed to collect in the system. A probable accumulation point is the restricting frits that control flow rates. The best way to remove moisture is to evacuate the analyzer at full rate for several days. If the indicated vacuum continues to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

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

Replace the Banana Plug

High Pressure Chamber

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

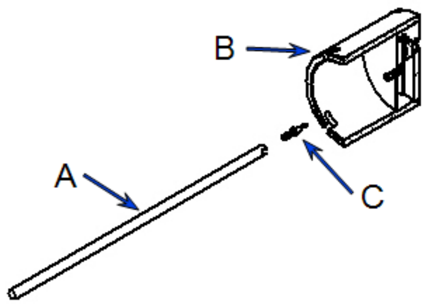


A. Feedthru assembly
B. Banana plug
C. Banana plug tool
D. High pressure chamber

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.



A. Banana plug tool
B. Low pressure capacitance detector
C. Banana plug

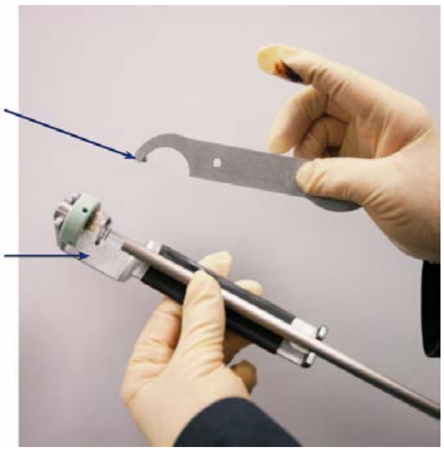
4. Screw the banana plug into the low pressure capacitance detector.

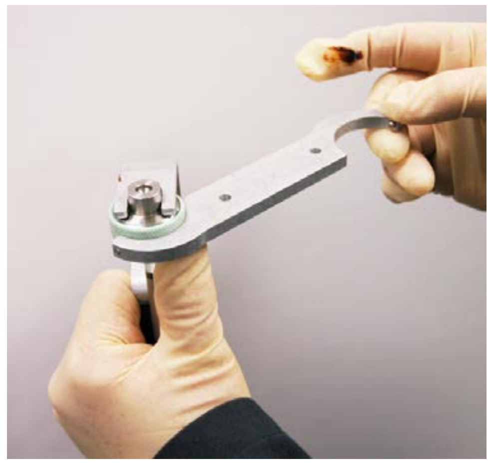
Penetrometer Nut

Remove the Penetrometer Nut

A

B





A. Wrench peg
B. Place penetrometer tool over the nut and cap

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.

To remove the penetrometer nut:

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.

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

POWER

The AutoPore V is designed to operate with a universal input power supply (100/120/220/240 VAC) at 50/60 Hz. Noise-free power of the correct voltage and frequency, with a safety earth ground, should be available through a standard wall receptacle. The power outlet should be able to supply 15 amps @ 100 or 120 VAC ±10% or 7.5 amps @ 220 or 240 VAC ±10%. These requirements can be checked by using a circuit analyzer (available at most hardware or electronic supply houses) or a multimeter. There should also be sufficient outlets for the analyzer, pump, computer, monitor, printer, and any other peripheral devices.

220V applications should use the 230V setting.

Correct power outlet wiring can be checked by using a circuit analyzer or a multimeter. The circuit analyzer is the preferred method. These devices plug directly into the wall receptacle and give a visual or audible indication of the status of the receptacle.

CAUTION

The analyzer and peripheral devices **must** be installed on their own dedicated power line. Other devices — such as motors, generators, or ovens — **should not** be placed on the same power line.

Replacement power supply cords must be rated for the specifications stated above.

POWER INSTRUMENT ON AND OFF

CAUTION

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

Power ON the equipment in the following order:

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 the vacuum pump manual.

1. Analyzer
2. Computer, monitor, and printer

Power OFF the equipment in the following order:

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

RECOVER FROM A POWER FAILURE

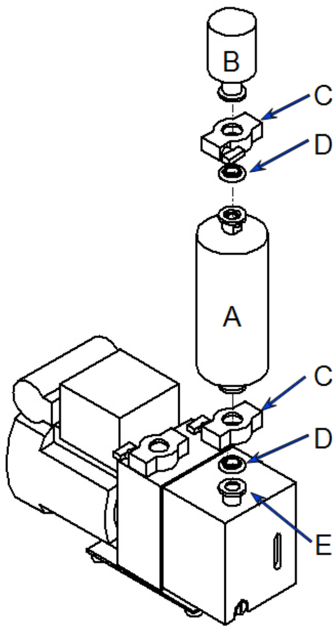
The analyzer saves entered and collected data in case of power failure. File parameters and any other data entered will still be present when power is restored. If an analysis was in progress when the power failure occurred, it will be canceled when the analyzer restarts. Any data collected during the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

VACUUM PUMP

The *Vacuum Pump Guide* can be found on the Micromeritics web page (www.Micromeritics.com).

VACUUM PUMP EXHAUST FILTER

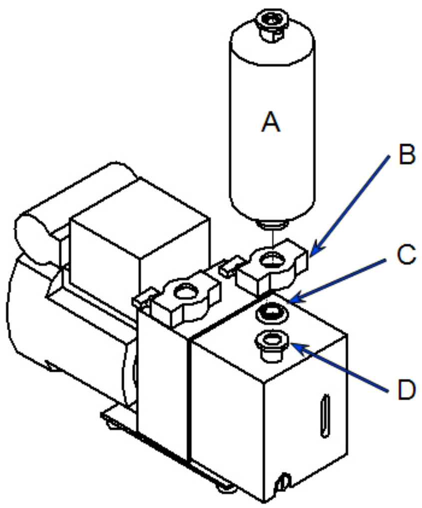
The exhaust filter is attached to the mercury vapor filter on the vacuum pump.



A. Mercury vapor filter
B. Exhaust filter
C. Wing nut clamp
D. Centering ring
E. Exhaust port

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.

VACUUM PUMP MERCURY VAPOR FILTER



A. Mercury vapor filter
B. Wing nut clamp
C. Centering ring
D. Exhaust port

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

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

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

CHANGE OR ADD VACUUM PUMP OIL

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

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

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

7. Remove the drain plug and drain the oil into the waste container.
8. Replace the drain plug.

9. Remove the drain plug from the oil-fill port on top of the pump.

10. 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.

11. Check the washer or O-ring used at the oil-filling port and replace if necessary.
12. Insert the oil-fill plug and turn clockwise to tighten.
13. Return the vacuum pump to the original position inside the analyzer.
14. Reconnect the vacuum pump hose and the exhaust filter to the top of the mercury vapor filter.
15. Reconnect the power cord to the power source and power on the vacuum pump on and recheck the oil level.
16. 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.

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A Advanced Reports - Python Module

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.

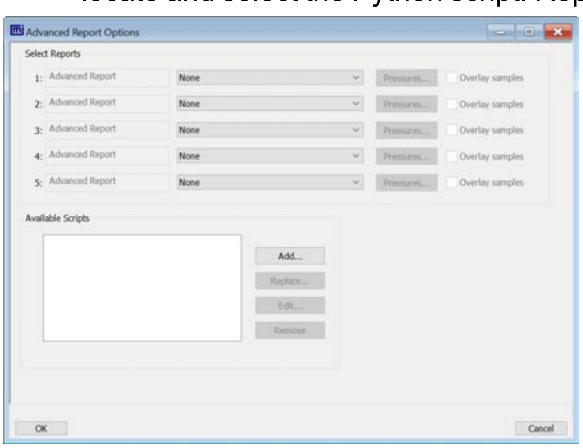
- **Summary reports.** Consist of summary sections, each containing a two-column table of label and value pairs. Summary reports are created with the *mic.summary* call.
- **Tabular reports.** Consist of one or more tables each containing one or more labeled columns of data. Tabular reports are created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

Advanced Report Options

Up to five Advanced reports, each with up to 10 summary reports, 10 tabular reports, and 10 graphical reports can be created. To use this feature, a file containing a Python script that imports a "mic" Python module must be created. See *MicModule Python Calls on page A - 13* for an example of a Python script and functions for the "mic" Python module.

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



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

Advanced Reports

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

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

SCRIPTS

Run a Script

1. Open a sample file with a *Complete* file status.
2. Select *Advanced* in the view selector drop-down list at the bottom of the window.
3. Select the *Report Options* tab.
4. Highlight *Advanced* in the *Selected Reports* list box, then click **Edit**.
5. On the *Advanced Report Options* window, click **Add**.
6. 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.
7. In the *Select Reports* section, select up to five *Advanced* reports in the drop-down lists.
8. Click **OK**.
9. Click **Preview** on the *Report Options* tab to view all reports selected in the previous window.

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.

Edit a Script

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

PYTHON REPORTS

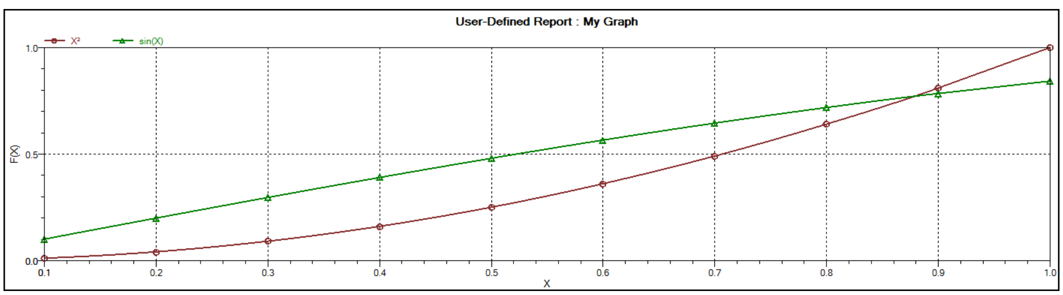
Graphic Report

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( 'X2', myx, myx*myx, marker='o' )
mic.graph.add( 'sin(X)', myx, np.sin(myx), marker='^' )
```

The results are:



Summary Report

This script produces a summary report with two summaries:

```
import mic
import numpy as np

mic.summary( "My Summaries" )
mic.summary.add( "Summary A",
                 ["Label 1:", "Label 2:", "Label 3:"],
                 ["val1", "val2", "val3"] )
mic.summary.add( "Summary B",
                 ["Label 4:", "Label 5:", "Label 6:"],
                 ["val4", "val5", "val6"] )
```

The result is:

Summary A

Label 1: val1
Label 2: val2
Label 3: val3

Summary B

Label 4: val4
Label 5: val5
Label 6: val6

Tabular Report

If more than one column is required, the call *mic.table* is employed. This script produces a tabular report consisting of two tables.

This script uses the Python package *numpy* and *c*-style formatting of the numerical values.

```
import mic
import numpy as np

mic.table( "My Tables" )
mic.table.addtable( "My Set A" )
mic.table.addcolumn( "X", ["1.0", "2.0", "3.0"] )
mic.table.addcolumn( "Y", ["0.5", "1.0", "1.5"] )
x1 = 0.2
x2 = 0.5
x3 = 3.14159/2
mic.table.addtable( "My Set B" )
mic.table.addcolumn( "X", ['{:8.3f}'.format(x1),
                           '{:8.3f}'.format(x2),
                           '{:8.3f}'.format(x3)] )
mic.table.addcolumn( "sin(X)", ['{:8.3f}'.format(np.sin(x1)),
                                '{:8.3f}'.format(np.sin(x2)),
                                '{:8.3f}'.format(np.sin(x3))] )
mic.table.addcolumn( "cos(X)", ['{:8.3f}'.format(np.cos(x1)),
                                '{:8.3f}'.format(np.cos(x2)),
                                '{:8.3f}'.format(np.cos(x3))] )
```

The result is:

My Set A

X	Y
1.0	0.5
2.0	1.0
3.0	1.5

My Set B

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

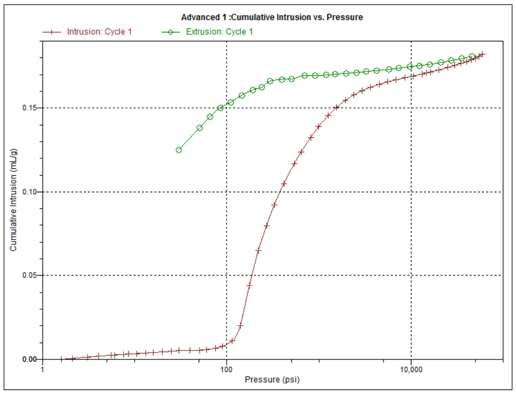
ACQUIRE BASIC INFORMATION

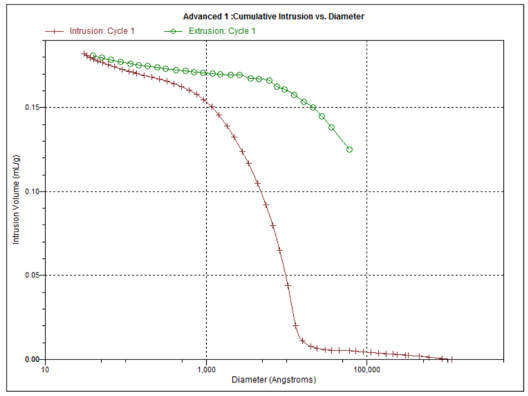
```
import mic

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

sampleinfo = [ ["Description:",          mic.sample_information("sample description")],
               ["Sample mass (g):",       "%8.3f" % mic.sample_information("sample mass")],
               ["Assembly mass (g):",     "%8.3f" % mic.sample_information("assembly mass"),],
               ["Penetrometer mass (g):", "%8.3f" % mic.sample_information("penetrometer mass")]  ]

materialinfo = [ [ "Material name:",                     mic.material_properties("material name") ],
                 [ "BET surface area (m^2/g):",          "%8.3f" % mic.material_properties("bet surface area") ],
                 [ "Mercury Density (g/ml):",            "%8.3f" % mic.mercury_properties("density") ],
                 [ "Mercury Surface Tension (dynes/cm):", "%8.3f" % mic.mercury_properties("surface tension") ],
```

```
                 [ "Advancing Contact Angle (degrees):",  "%8.3f" % mic.mercury_properties("advancing contact angle") ],
                 [ "Receding Contact Angle (degrees):",   "%8.3f" % mic.mercury_properties("receding contact angle")] ]

summaryinfo = [ [ "Total intrusion volume (mL/g):",                    "%8.3f" % mic.report("hgsum", "total intrusion volume") ],
                [ "Pore area (m^2/g):",                                "%8.3f" % mic.report("hgsum", "pore area")],
                [ "Bulk density (g/mL):",                              "%8.3f" % mic.report("hgsum", "bulk density")],
                [ "Apparent density (g/mL):",                          "%8.3f" % mic.report("hgsum", "apparent density")],
                [ "Median diameter by volume (Angstroms):",            "%8.3f" % mic.report("hgsum", "median diameter by volume")],
                [ "Median diameter by area (Angstroms):",              "%8.3f" % mic.report("hgsum", "median diameter by area")],
                [ "4 V/A average diameter (Angstroms):",               "%8.3f" % mic.report("hgsum", "4 V/A average diameter")],
                [ "Porosity (%):",                                     "%8.3f" % mic.report("hgsum", "porosity")],
                [ "Tortuosity:",                                       "%8.3f" % mic.report("hgsum", "tortuosity")],
                [ "Tortuosity factor:",                                "%8.3f" % mic.report("hgsum", "tortuosity factor")],
                [ "Permeability (mdarcy):",                            "%8.3f" % mic.report("hgsum", "permeability")],
                [ "Permeability constant:",                            "%8.3f" % mic.report("hgsum", "per-
```

```
meability constant")],
                  [ "Break-through pressure ratio:",            "%8.3f" % mic.report("hgsum", "break-through pressure ratio")],
                  [ "linear compressibility coefficient (1/psi):",      "%8.3f" % mic.report("hgsum", "linear compressibility coefficient")],
                  [ "quadratic compressibility coefficient (1/psi^2):",   "%8.3f" % mic.report("hgsum", "quadratic compressibility coefficient") ] ]

mic.summary( "Summaries" )

mic.summary.add( "Sample Information:",
                  [ a for a, b in sampleinfo],
                  [ b for a, b in sampleinfo] )

mic.summary.add( "Material & Mercury Properties",
                  [ a for a, b in materialinfo],
                  [ b for a, b in materialinfo] )

mic.summary.add( "Intrusion Summary Results",
                  [ a for a, b in summaryinfo],
                  [ b for a, b in summaryinfo] )
```

The results are:

Sample Information:

Description:	Clay
Sample mass:	2.110 g
Assembly mass:	140.390 g
Penetrometer mass:	62.379 g
Penetrometer volume:	6.760 mL
Stem volume used:	33.477%

Material Properties

Material name:	Garnet
BET surface area:	200.000 m²/g
Entered bulk density:	nan g/mL
Entered true density:	nan g/mL
Entered particle density:	nan g/mL
Entered conductivity formation factor:	nan
Entered threshold pressure:	nan psia
Linear compressibility coefficient:	-0.000 1/psia
Quadratic compressibility coefficient:	0.000 1/psia²

Mercury Properties

Advancing contact angle:	130.000°
Receding contact angle:	130.000°
Surface tension:	485.000 dynes/cm
Density:	13.533 g/mL
Linear compressibility coefficient:	-0.000 1/psia
Quadratic compressibility coefficient:	0.000 1/psia²

Intrusion Summary Results

Total intrusion volume:	0.182 mL/g
Pore area:	12.041 m²/g
Bulk density:	1.833 g/mL
Bulk density pressure:	1.594 psia
Apparent density:	2.751 g/mL
Apparent density pressure:	59853.699 psia
Median diameter by volume:	5595.058 Å
Median diameter by area:	53.917 Å
4 V/A average diameter:	605.072 Å
Porosity:	33.384%
Pressure range min:	0.000 psia
Pressure range max:	61000.000 psia
Pore range min diameter:	29.650 Å
Pore range max diameter:	0.000 Å
Tortuosity:	25.407
Tortuosity factor:	1.853
Permeability:	0.729 mdarcy
Permeability constant:	0.004
Break-through pressure ratio:	6.272
Linear compressibility coefficient:	-0.000 1/psia
Quadratic compressibility coefficient:	0.000 1/psia²

MICMODULE PYTHON CALLS

Tables

Available Mic Python calls for tables:

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

Add a Table

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

```
mic.table.addtable( name )

Keyword arguments:

  name --- the table name
```

Add a Column

This script adds a column to the last created table:

```
mic.table.addcolumn(header, values, align='r'):

Keyword arguments:

  header --- column header; must be a string (or convertible)
  values --- column values; must be a list of strings (or convertible)
  align  --- column alignment; 'r', 'l', 'c' for right, left, and center justified
```

Create a New Tabular Report

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

Keyword arguments:

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

Summary Reports

Add a Summary Section

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

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

Keyword arguments:

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

Create a New Summary Report

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

Keyword arguments:

  title --- the summary title
```

Graphic Reports

Add a Curve

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

```
mic.graph.add(name, x, y, yyaxis=False, color=None, linestyle='-',
              marker='a', graphtype='both', interpolation='akima'):

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
  interpolation -- linear or akima spline interpolation (default='akima')
                   'akima' use akima spline
                   'linear' use linear interpolation
```

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.
```

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,
          xinvert=False, yinvert=False, yyinvert=False,
          xrange=None, yrange=None, yyrange=None, xbars_id=''):

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)
  xinvert  --- Invert x-axis if true (default = False)
  yinvert  --- Invert y-axis if true (default = False)
  yyinvert --- Invert yy-axis if true (default = False)
  xrange   --- None, or two values giving the min and max
               range of the axis.
  yrange   --- None, or two values giving the min and max
               range of the axis.
  yyrange  --- None, or two values giving the min and max
               range of the axis.
  xbars_id --- None, or the id of an xbar control created
               via the mic.control() object
```

Get Intrusion-Extrusion Data

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

Get intrusion data for a specified cycle

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):

Get extrusion data for a specified cycle

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

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

Get Mercury Properties

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

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

Get Properties of Sample Material

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

Get material properties for a sample

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.
```

B Blank and Sample Compression Corrections

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

Baseline Errors

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

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

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

Another effect, and the one most difficult to predict, arises from the similarity of the penetrometer to a thermometer. This would not be troublesome if its temperature could be maintained constant, but compression of the surrounding oil causes a temperature rise of nearly 50 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.

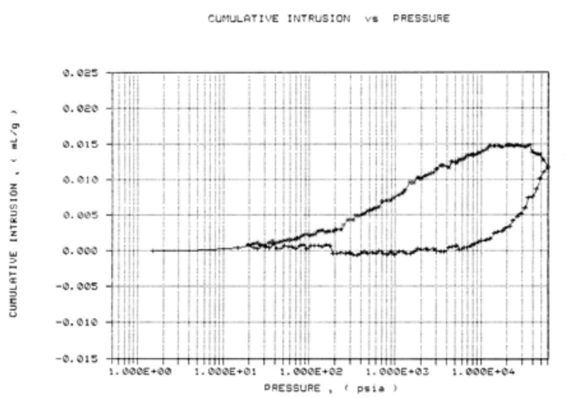


Figure 1

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.

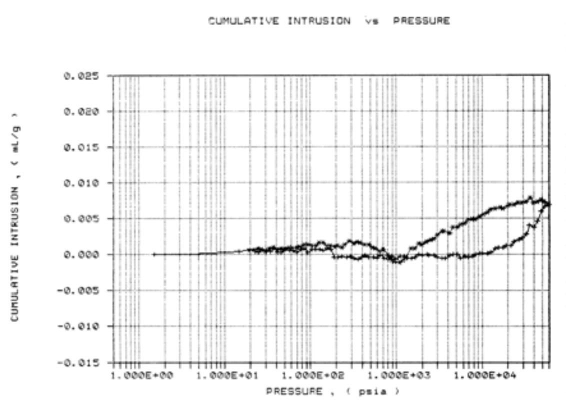


Figure 2

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.

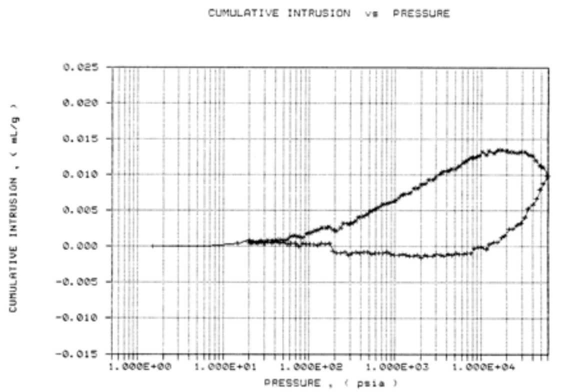


Figure 3

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.

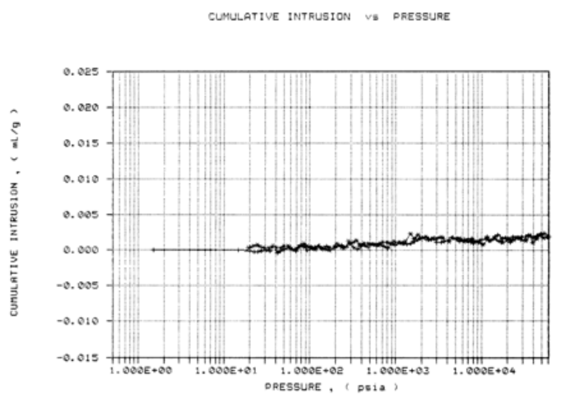


Figure 4

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.

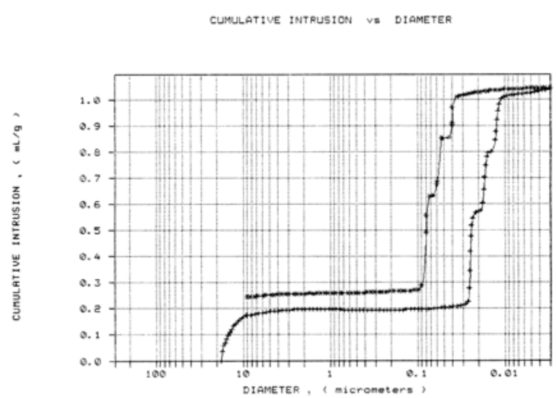


Figure 5

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.

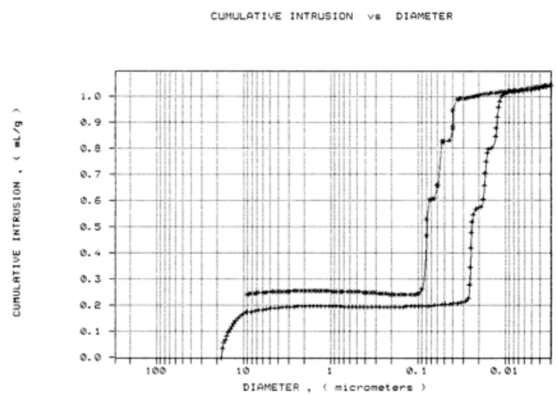


Figure 6

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.

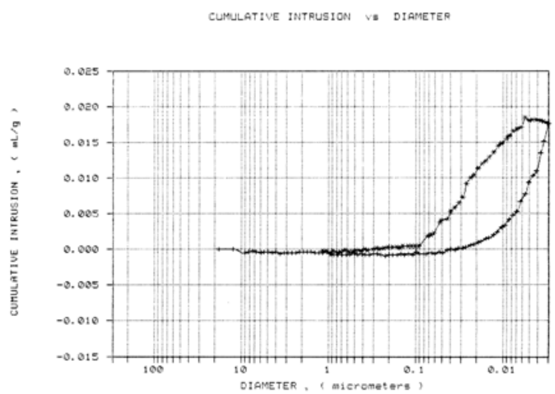


Figure 7

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.

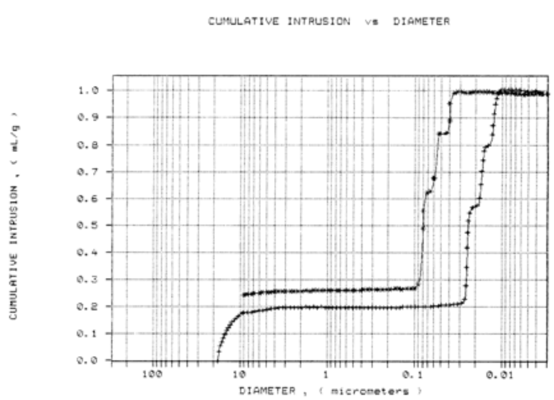


Figure 8

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

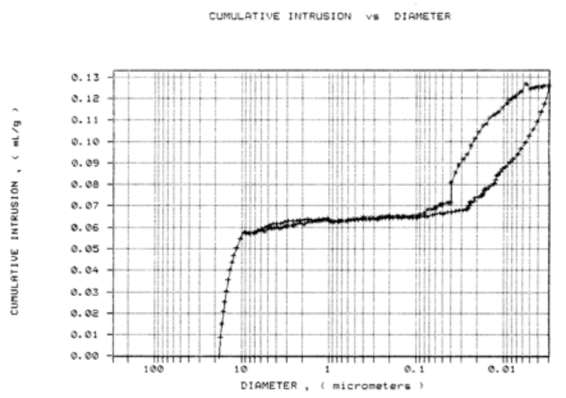


Figure 9

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

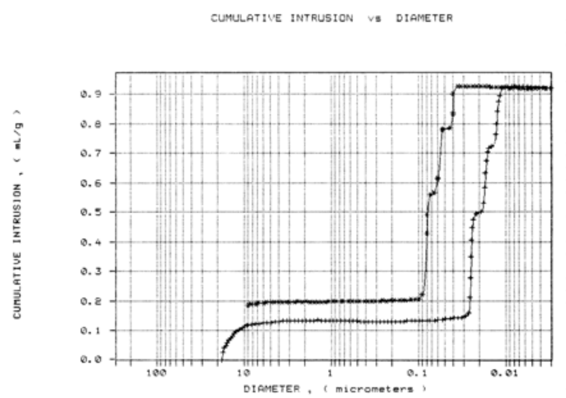


Figure 10

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

C 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.

D Proper Handling of Mercury

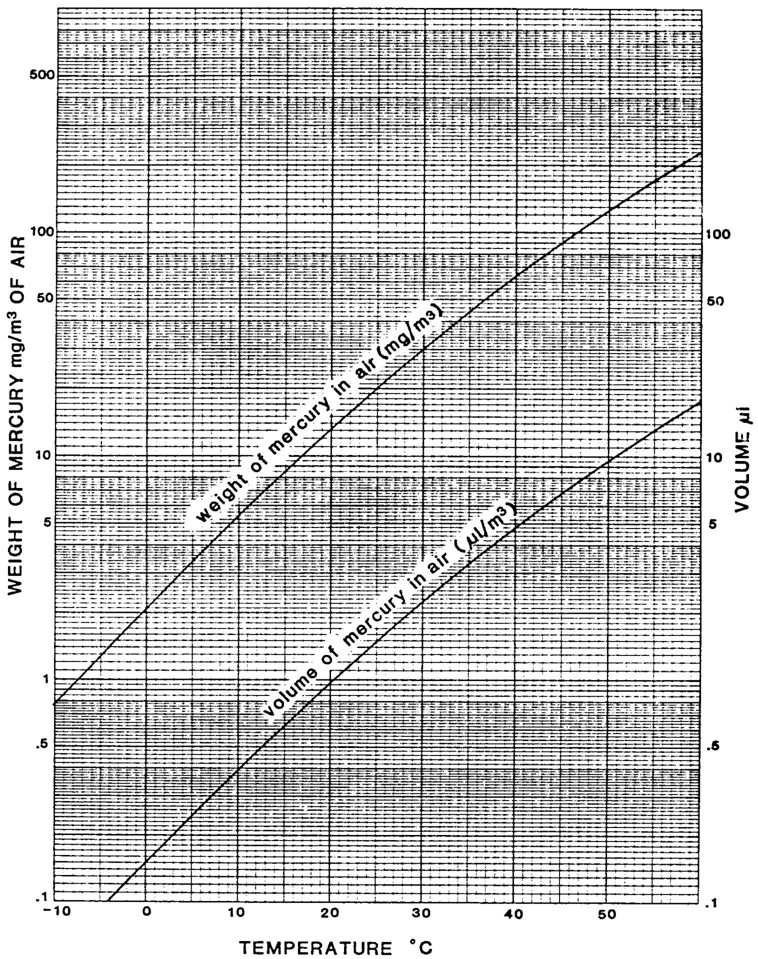
Because of its low melting point (-38.87 ºC), mercury (Hg) is slightly volatile at ordinary room temperatures and its vapor may pose a health hazard if allowed to accumulate in the work space. Although mercury can enter the body through the skin, lungs or digestive system, breathing air laden with high 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. Hand washing facilities, including hot and cold running water, soap, hand cream, and towels should be made available adjacent to mercury work areas. Clothing contaminated with mercury should be stored in vapor-proof containers pending removal for laundering.

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 back. 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.

[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.

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E THEORY FOR MERCURY POROSIMETRY

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 the *Calculations* document on the Micromeritics web page (www.Micromeritics.com) for a discussion on Pore Surface Area Computation.

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F MAXIMUM INTRUSION VOLUME OPTION

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

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

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

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G WORKSHEETS

Worksheets in this section may be copied as needed.

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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:

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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 penetrometer filled with mercury ______ g
2. Mass of sealed, empty penetrometer ______ g
3. Mass of mercury (line 1 minus line 2) ______ g

   Room Temp = ______ °C Density of mercury * = ______ g/mL

4. Volume of penetrometer (line 3 ÷ density of mercury) ______ mL

Second Calibration of Penetrometer Volume:

1. Mass of penetrometer filled with mercury ______ g
2. Mass of sealed, empty penetrometer ______ g
3. Mass of mercury (line 1 minus line 2) ______ g

   Room Temp = ______ °C Density of mercury * = ______ g/mL

4. Volume of penetrometer (line 3 ÷ density of mercury) ______ mL

Third Calibration of Penetrometer Volume:

1. Mass of penetrometer filled with mercury ______ g
2. Mass of sealed, empty penetrometer ______ g
3. Mass of mercury (line 1 minus line 2) ______ g

   Room Temp = ______ °C Density of mercury * = ______ g/mL

4. Volume of penetrometer (line 3 ÷ density of mercury) ______ mL

Average Reference Capsule Volume: ______ mL

* Refer to the table ***Density of Mercury on the next page*** for values.

DENSITY OF MERCURY

Density of Mercury

°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**

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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.				

Volumetric Reference Capsule Worksheet

Worksheet Instructions

Either a Volumetric Capsule test or a Reference Material test is required for verification.

A *Volumetric Reference Capsule* test is performed to verify the ability of the low pressure and high pressure system to measure mercury intrusion volume accurately. Perform the following tests and record the data in this worksheet.

- Perform the Low Pressure ports test using 5 cc solid penetrometers with a 1.131 mL stem volume and an 8 µm membrane.
- Perform the High Pressure ports test using 5 cc solid penetrometers with a 1.131 mL stem volume and a 3 µm membrane.

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

Penetrometer Number ______________________ Date: ______________________

By: ______________________

First Calibration of Reference Capsule Volume:

1. Mass of capsule filled with mercury ______________ g
2. Mass of sealed, empty capsule ______________ g
3. Mass of mercury (line 1 minus line 2) ______________ g

Room Temp = ____________ °C Density of mercury * = ______________ g/mL

4. Volume of capsule (line 3 ÷ density of mercury) ______________ mL

Second Calibration of Reference Capsule Volume:

1. Mass of capsule filled with mercury ______________ g
2. Mass of sealed, empty capsule ______________ g
3. Mass of mercury (line 1 minus line 2) ______________ g

Room Temp = ____________ °C Density of mercury * = ______________ g/mL

4. Volume of capsule (line 3 ÷ density of mercury) ______________ mL

Third Calibration of Reference Capsule Volume:

1. Mass of capsule filled with mercury ______________ g
2. Mass of sealed, empty capsule ______________ g
3. Mass of mercury (line 1 minus line 2) ______________ g

Room Temp = ____________ °C Density of mercury * = ______________ g/mL

4. Volume of capsule (line 3 ÷ density of mercury) ______________ mL

Average Reference Capsule Volume: ______________ mL

* See ***Density of Mercury Table on the next page*** for values.

Copy and use this worksheet as needed

DENSITY OF MERCURY TABLE

Density of Mercury

°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

H Exported Data Example

This exported data has been truncated for this manual.

Sample Information

Sample Information

Method:	Default
Sample:	garnet ref mat 60k eqil (rate) 568
Operator:	jch
Submitter:	micromeritics performance test
Type of data:	Automatically collected
Instrument type:	9600
Original instrument type:	9600
Comments:	
Penetrometer mass:	63.9542 g
Sample mass:	0.2890 g
Assembly mass:	104.2008 g

Material Properties

Material:	Garnet
BET surface area:	3.0000 m^2/g
Use user entered density:	Yes
Bulk Density:	1.2287 g/mL
True Density:	3.8800 g/mL
Effective Particle Density:	3.8800 g/mL
Use user entered conductivity formation factor:	Yes
Factor:	1.000
Use user entered pressure threshold:	No
Linear compressibility:	-2.7400e-07 1/psia

```
                       Quadratic compressibility:    2.8500e-13 1/psia²

                        Penetrometer Properties

         Penetrometer:    #568 - (14-0765)  3 Bulb, 0.412 Stem, Powder
    Penetrometer mass:    63.9542 g
               Volume:    3.2091 mL
             Constant:    11.117 µL/pF
          Stem volume:    0.4125 mL
   Max. head pressure:    4.680 psia
    Correction method:    None


Analysis Conditions



  Analysis conditions:    Run Conditions

                 Mercury Properties

     Advancing contact angle:    130.000 °
      Receding contact angle:    130.000 °
             Surface tension:    485.000 dynes/cm
                Density type:    Entered
  Mercury density (entered):    13.5335 g/mL
    Linear compressibility:    -2.7400e-07 1/psia
  Quadratic compressibility:    2.8500e-13 1/psia²

                  Evacuation Options

              Sample type:    Other
```

```
    Initially evacuate at:    5.0 psia/min
      Switch to medium at:    Use pressure transducer
                              0.25 psia
        Switch to fast at:    200 µmHg
        Evacuation target:    50 µmHg
  Continue evacuating for:    5 min

           Low Pressure

    Filling pressure:    1.000 psia
  Equilibration time:    1 s

          High Pressure

        Equilibration time:    1 s
  Hold at maximum pressure:    No

            Reverberi Options

  Autocalculate Reverberi pressures:    No

                                   Pressure Table

                                                                              Intru-
sion
          Pressure      Points     Ending        Maximum       Pressure         Sca-
n
          Increment      per    Pressure      Intrusion     Scan Rate         Rate
     (psia)       Decade     (psia)        (mL/g)       (min/decade)    (s·mL/g)
-------
     1         1.00                    30.00                              5.0       0.0-
0100
     2                                 30.46                              5.0       0.0-
0100
```

```
     3                              30.93                          5.0      0.0-
0100
     4                              31.40                          5.0      0.0-
0100
     5                              31.88                          5.0      0.0-
0100
     6                              32.37                          5.0      0.0-
0100
     7                              32.87                          5.0      0.0-
0100
     8                              33.38                          5.0      0.0-
0100
     9                              33.89                          5.0      0.0-
0100
    10                              34.41                          5.0      0.0-
0100
    11                              34.94                          5.0      0.0-
0100
    12                              35.47                          5.0      0.0-
0100
    13                              36.02                          5.0      0.0-
0100
    14                              36.57                          5.0      0.0-
0100
    15                              37.13                          5.0      0.0-
0100
    16                              37.70                          5.0      0.0-
0100
    17                              38.28                          5.0      0.0-
0100
    18                              38.87                          5.0      0.0-
0100
    19                              39.46                          5.0      0.0-
0100
    20                              40.07                          5.0      0.0-
0100
    21                              40.68                          5.0      0.0-
0100
```

```
    22                          41.31                        5.0      0.0-
0100
    23                          41.94                        5.0      0.0-
0100
    24                          42.59                        5.0      0.0-
0100
    25                          43.24                        5.0      0.0-
0100
    26                          43.90                        5.0      0.0-
0100
    27                          44.58                        5.0      0.0-
0100
    28                          45.26                        5.0      0.0-
0100
    29                          45.96                        5.0      0.0-
0100
    30                          46.66                        5.0      0.0-
0100
    31                          47.38                        5.0      0.0-
0100
    32                          48.11                        5.0      0.0-
0100
    33                          48.84                        5.0      0.0-
0100
  *
34                          49.59                        5.0      0.00100
    35                          50.35                        5.0      0.0-
0100
    36                          51.13                        5.0      0.0-
0100
    37                          51.91                        5.0      0.0-
0100
    38                          52.71                        5.0      0.0-
0100
    39                          53.52                        5.0      0.0-
0100
    40                          54.34                        5.0      0.0-
0100
```

```
    41                                    55.17                         5.0      0.0-
0100
    42                                    56.02                         5.0      0.0-
0100
    43                                    56.88                         5.0      0.0-
0100
    44                                    57.75                         5.0      0.0-
0100
    45                                    58.64                         5.0      0.0-
0100
    46                                    59.54                         5.0      0.0-
0100
    47                                    60.45                         5.0      0.0-
0100
    48                                    61.38                         5.0      0.0-
0100
    49                                    62.32                         5.0      0.0-
0100
    50                                    63.28                         5.0      0.0-
0100
    51                                    64.25                         5.0      0.0-
0100
    52                                    65.24                         5.0      0.0-
0100
    53                                    66.24                         5.0      0.0-
0100
    54                                    67.26                         5.0      0.0-
0100
    55                                    68.29                         5.0      0.0-
0100
    56                                    69.34                         5.0      0.0-
0100
    57                                    70.40                         5.0      0.0-
0100
    58                                    71.48                         5.0      0.0-
0100
    59                                    72.58                         5.0      0.0-
0100
```

```
    60                                  73.69                              5.0       0.0-
0100
    61                                  74.82                              5.0       0.0-
0100
    62                                  75.97                              5.0       0.0-
0100
    63                                  77.14                              5.0       0.0-
0100
    64                                  78.32                              5.0       0.0-
0100
    65                                  79.52                              5.0       0.0-
0100
    66                                  80.74                              5.0       0.0-
0100
    67                                  81.98                              5.0       0.0-
0100
    68                                  83.24                              5.0       0.0-
0100
    69                                  84.52                              5.0       0.0-
0100
    70                                  85.82                              5.0       0.0-
0100
    71                                  87.13                              5.0       0.0-
0100
    72                                  88.47                              5.0       0.0-
0100
    73                                  89.83                              5.0       0.0-
0100
    74                                  91.21                              5.0       0.0-
0100
    75                                  92.61                              5.0       0.0-
0100
    76                                  94.03                              5.0       0.0-
0100
    77                                  95.47                              5.0       0.0-
0100
    78                                  96.94                              5.0       0.0-
0100
```

```
   79                            98.43                            5.0      0.0-
0100
   80                            99.94                            5.0      0.0-
0100
   81                           101.47                            5.0      0.0-
0100
   82                           103.03                            5.0      0.0-
0100
   83                           104.61                            5.0      0.0-
0100
   84                           106.22                            5.0      0.0-
0100
   85                           107.85                            5.0      0.0-
0100
   86                           109.50                            5.0      0.0-
0100
   87                           111.18                            5.0      0.0-
0100
   88                           112.89                            5.0      0.0-
0100
   89                           114.62                            5.0      0.0-
0100
   90                           116.38                            5.0      0.0-
0100
   91                           118.17                            5.0      0.0-
0100
   92                           119.98                            5.0      0.0-
0100
   93                           121.82                            5.0      0.0-
0100
   94                           123.69                            5.0      0.0-
0100
   95                           125.59                            5.0      0.0-
0100
   96                           127.52                            5.0      0.0-
0100
```

I Compute Volumetric Compressibility of a Sample Material

Ideally, choose a sample material that is completely non-porous; if this is not the case, then 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, 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, 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%.

micromeritics®

EU DECLARATION OF CONFORMITY

This declaration of conformity is issued under the sole responsibility of the manufacturer:

Micromeritics Instrument Corporation
4356 Communications Drive
Norcross, GA 30093, USA

Hereby declares that the product:

AutoPore V (9600, 9605, 9610, 9620) Automated Mercury Porosimeter

is in conformity with the following **EU harmonization legislation**:

2014/35/EU - LVD Directive
2014/30/EU - EMC Directive
2011/65/EU - RoHS Directive

and that the equipment is in conformity with the following harmonized and other appropriate standards;

2014/35/EU (LVD)
IEC 61010-1:2010/A1:2016 - *Safety requirements for electrical equipment for measurement, control, and laboratory use — Part 1: General requirements.*
IEC 61010-2-081:2019 - *Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes*

2014/30/EU (EMC)
IEC 61326-1:2020 - *Electrical equipment for measurement, control and laboratory use — EMC requirements — Part 1: General requirements*
IEC 61000-3-2:2014 - *Part 3-2: Limits — Limits for harmonic current emissions (equipment input current ≤ 16 A per phase)*
IEC N 61000-3-3:2013 - *Part 3-3: Limits — Limitation of voltage changes, voltage fluctuations and flicker in public low-voltage supply systems, for equipment with rated current <= 16 A per phase and not subject to conditional connection*

2011/65/EU (RoHS)
EN 63000:2018 - *Technical documentation for the assessment of electrical and electronic products with respect to the restriction of hazardous substances*

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Title: Vice President, R & D

Signature:

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